



THÈSE DE DOCTORAT DE L'UNIVERSITÉ
BOURGOGNE FRANCHE-COMTÉ PRÉPARÉE À
L'UNIVERSITÉ DE FRANCHE-COMTÉ

ÉCOLE DOCTORALE N°37

SCIENCES POUR L'INGÉNIEUR ET MICROTECHNIQUES
SPIM

Doctorat de Ingénierie mécanique

PAR

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**Etude des effets du cuivrage et de
l'irradiation par proton à 440 GeV sur
les propriétés mécaniques de fils en
nanotubes de carbone pour
l'instrumentation des faisceaux de
particules au CERN**

Thèse présentée et soutenue à Besançon, le 3 mai 2023

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Remerciements

Je tiens à remercier chaleureusement Ray et Thibaut pour leur soutien et leur confiance au cours de ces 6 années passées au CERN qui m'ont permis d'évoluer dans un environnement de travail sain et motivant.

Merci également à Michel, pour son implication dans ce projet, sa précise aide tout au long de ce travail, et la rigueur avec laquelle il a prêté attention aux différents rendus écrits, et particulièrement ce manuscrit.

Un immense merci à Tania, ma merveilleuse femme, pour son soutien moral (et logistique), à Leïla et Anaïs, mes deux amours de filles qui m'ont donné une motivation supplémentaire à vite et bien terminer ce travail.

Je souhaiterais remercier l'ensemble de l'équipe ML: William, Dmitri, Gerhard, Paulo, Arnaud, Morad, Patrick, Christophe, Claude, Thierry que ce soit d'un point de vue professionnel comme personnel.

Je souhaiterais sincèrement remercier l'équipe directe avec qui j'ai pu mener à bien le projet HiRadMat: Ben, Michel, Stéphane, Anité, Thomas et Clémence, qui se sont impliqués personnellement sur un projet qui n'était pas le leur. Un grand remerciement également à Anastasiia et Juan Jose pour leur collaboration, temps et expertise ainsi que la fourniture des échantillons qui ont servi dans l'expérience.

Une collaboration qui, j'espère, perdurera.

Merci à Abou, Pascal and Nickos pour le soutien logistique sur l'expérience dans HiRadMat. Les autres personnes du CERN m'ayant permis d'avancer: Stéphane, Federico, Stefano et Araceli leur aide précieuse dans des domaines qui ne sont pas les miens. Merci à la section MME section, et spécifiquement Anité pour sa disponibilité et son aide sur l'analyse des échantillons.

Un vrai, grand merci à Jean-Emmanuel pour son aide pleine, entière et désintéressée, une très belle rencontre.

Merci à Xavier du DMA de FEMTO-ST pour son aide et son expérience pour les tests en traction d'échantillons micrométriques.

Merci à ma famille (parents, frères, beaux-parents, beaux-frères et belles-soeurs) pour leurs questions basiques, mais pas que, qui permettent une remise en question et une prise de recul nécessaire, pour expliquer sur quoi on travail, ainsi que leur soutien moral et logistique.

Un merci tout particulier à Hans (presque un troisième co-encadrant, mais on ne le dit pas pour des raisons de conflits d'intérêts...) pour ces longues discussions plus qu'instructives du week-end qui m'ont énormément aidées. Et enfin, merci à toutes les personnes que j'ai pu cotoyer, de près ou de loin, et qui m'ont permis d'être là aujourd'hui.

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Parameters

Table 1: Parameters used in this work

A_n	Fourier coefficients		
c	Speed of light	299792458	m.s ⁻¹
c_p	Specific heat capacity		J.kg ⁻¹ .K ⁻¹
d_{CNT}	CNT outer diameter		nm
$\langle dE/dx \rangle$	Specific stopping power		MeV.cm ² .g ⁻¹
d_{eq}	Equivalent diameter		μm
d_{iw}	CNT inter wall distance	0.34	nm
d_p	Distance between two points		m
d_w	Wire diameter		μm
E	Young's modulus		MPa
E_{beam}	Beam energy		GeV
E_c	Young's modulus in c-axis direction		MPa
E_{dep}	Energy deposited in matter		MeV
$E_{dep-tot}$	Energy total deposited		MeV
F_p	Preload		N
G	Shear modulus		MPa
G_c	Shear modulus in c-axis direction		MPa
$\hbar$	Reduced Planck constant	$1.055.10^{-34}$	J.s
h_{eff}	Effective part of the beam		mm
H_{sub}	Enthalpy of sublimation		J.mol ⁻¹
I_b	Beam intensity		
J_{th}	Thermionic current surface density		A.m ⁻²
k	Slip factor		
k_B	Boltzman constant	$1.38.10^{-23}$	J.K ⁻¹
k_{th}	Thermal conductivity		W.m ⁻¹ .K ⁻¹
K	Kinematic factor		
l	Wire length		mm
l_b	Bundle length		μm
l_{CNT}	CNT length		nm
$\mathcal{L}$	Luminosity		cm ⁻² .s ⁻¹
m	mass		kg
m_c	Carbon atom mass		
m_e	Electron mass	0.511	MeV.c ⁻²
m_{heat}	Heated mass		kg
m_{ip}	Incident particle mass		MeV.c ⁻²

m_p	Proton mass	938	MeV.c ⁻²
m_W	Weibull shape parameter		
M	Molar mass		g.mol ⁻¹
N_b	Number of bunches		
N_p	Number of particles in the beam		
N_{pb}	Number of particles per bunch		
n_{pps}	Number of points per sigma		
n_v	Number of fibers per volume unit		
n_w	Number of walls		
$\dot{n}$	Event rate		s ⁻¹
$\mathcal{N}_A$	Avogadro number	6.02.10 ²³	mol ⁻¹
N_C	Number of contact points between two fibers		
P	Pressure		Pa
p_{beam}	Beam momentum		MeV.c ⁻²
s	Slip length of the CNTs		nm
S	Cross section area		m ²
q_e	Electron charge	-1.602.10 ⁻¹⁹	C
q_p	Proton charge	1.602.10 ⁻¹⁹	C
Q	CNT migration length		m
r_e	Classical electron radius	2.817940	fm
R	Forks length		m
$\mathcal{R}$	Ideal constant gas	8.314	J.mol ⁻¹ .K ⁻¹
t_{rev}	Beam time of revolution		s
T	Temperature		K
T_{amb}	Ambiant temperature		K
T_d	Displacement threshold energy		eV
v_p	Speed of the particles		m.s ⁻¹
v_{scan}	Linear speed of scan		m.s ⁻¹
V	Volume		cm ³
w_b	Bundle width		μm
Z	Atomic number		

α	Wire twist angle		deg
α_{cs}	Courant-Snyder parameter		
α_s	Fine structure constant	1/137.035	
β	Relativistic factor		
β_{cs}	Courant-Snyder parameter		
β_f	Beta function		mm.rad ⁻¹
β_W			
γ	Lorentz factor		
γ_{cs}	Courant-Snyder parameter		
γ_g	Surface energy of graphene sheet		
δ	Wire deflection		μm
ε	Engineering strain		%
$\varepsilon_{h/v}$	Emitance (horizontal / vertical)		mm.rad ⁻¹
θ	Rotation angle of the phase space diagram		rad
λ	Linear density		kg.m ⁻¹
μ	CNT friction coefficient		
ν	Poisson's coefficient		
ρ	Density		kg.m ⁻³
ρ_D	Areal density of defects		cm ⁻²
σ	Stress of the wire		
σ_b	Beam size (1σ)		mm
σ_B	Stefan-Boltzmann constant	$5.67.10^{-8}$	W.m ⁻² .K ⁻⁴
σ_{eff}	Physical cross-section		barn
σ_{mb}	Maximal axial stress before shear failure		MPa
σ_0	Weibull scale parameter		
τ	Shear strength		MPa
τ_c	Critical shear strength		MPa
Φ	Collision angle		μrad
Φ_P	Packing fraction		
Φ_V	Volume fraction		
Φ_w	Work function		V
ω	Angular velocity		rad.s ⁻¹

Introduction

CERN, European Council for Nuclear Research, founded in 1954, located on the Franco-Swiss border near Geneva, is the largest and most powerful particle accelerator complex in the world. The energies during the collision of the beams within the experiments in the LHC (Large Hadron Collider) reach today 13 TeV, and could increase to 100 TeV within the framework of the FCC project (Future Circular Collider). The LHC, the final circular accelerator, is powered by a succession of linear (LINAC2, LINAC3 and LINAC4) and circular (Low Energy Ion Ring (LEIR), Proton Synchrotron Booster (PSB), Proton Synchrotron (PS) and Super Proton Synchrotron (SPS)) accelerators.

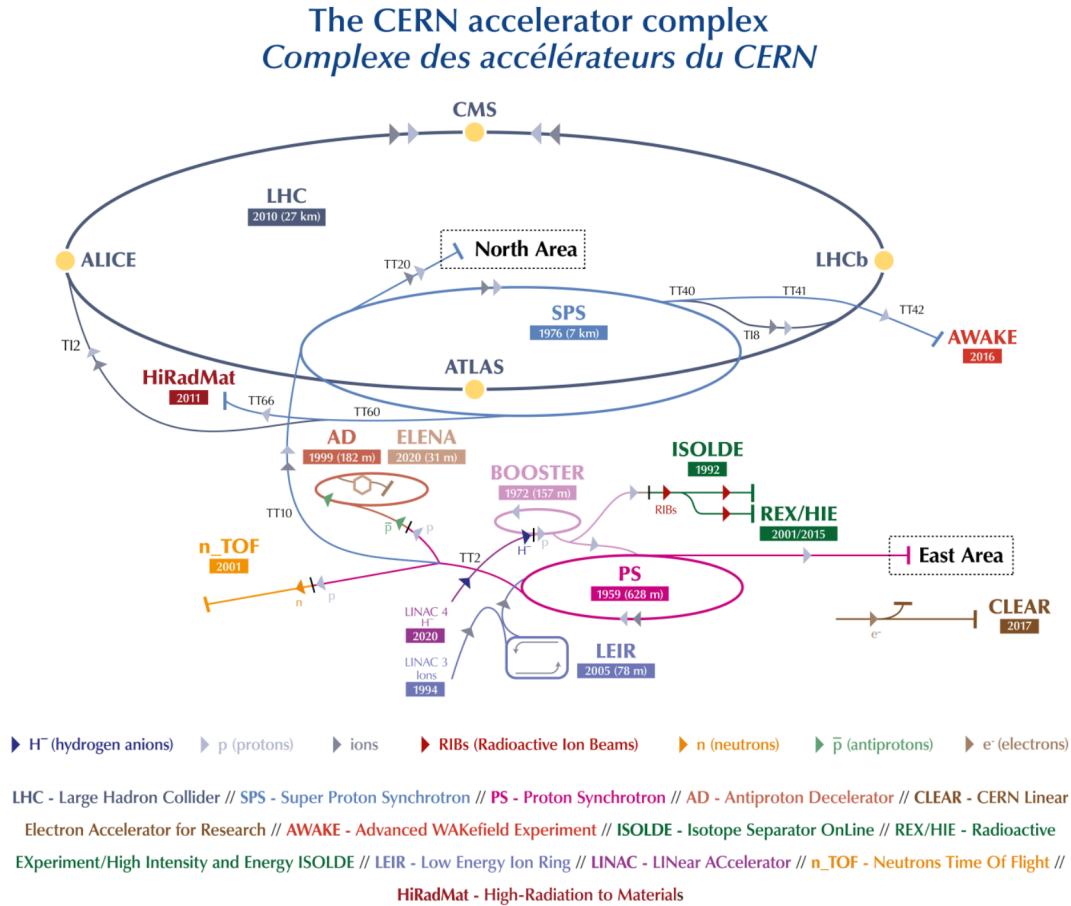


Figure 1: The LHC is the last ring (dark blue line) in a complex chain of particle accelerators. The smaller machines are used in a chain to help boost the particles to their final energies and provide beams to a whole set of smaller experiments, which also aim to uncover the mysteries of the Universe [1].

The injection chain also makes it possible to provide beams of various particles with particular specificities for the experimental areas, including:

- Research on antimatter with Antiproton Decelerator (AD) and Extra Low Energy Antiproton (ELENA)
- Research on ion beams with Isotope Separator On-Line DEvice (ISOLDE)
- Research on neutron-nucleus interactions for a wide range of neutron energies with Neutron Time-of-Flight (n_TOF)
- Study on the use of proton-driven plasma wakefields for charged particles acceleration in Advanced WAKefield Experiment (AWAKE)
- Research on the induced damage on materials caused by the irradiation in High Radiation to Materials (HiRadMat) (described more precisely later)

The accelerator chain is composed of a linear accelerator followed by three circular accelerators before the injection in the LHC for the main major experiments:

- ***Linac4***

The production of accelerated proton beams starts with compressed hydrogen gas injected in the Linac4 (Linear Accelerator). At the beginning of the accelerator, the electrons are stripped off to leave protons that are accelerated to an energy of 160 MeV before extraction to the PSB (Proton Synchrotron Booster). In the Linac4, the hydrogen ions are pulsed every 400 μ s along the accelerator with the help of radio-frequency cavities to accelerate them and quadrupole magnet to focus the beam. [2].

- ***Proton Synchrotron Booster***

The booster is the first and smallest circular accelerator (50 m in diameter), composed of four superimposed rings that accelerate the beam from 160 MeV to 2 GeV, in approximatively 530 ms. The presence of this intermediate circular accelerator allows an injection of a number of protons into the next accelerator which is much larger. The PSB can also feed the ISOLDE experiment [3].

- ***Proton Synchrotron***

The Proton Synchrotron (PS) is a key element of the LHC accelerator chain at CERN. It picks up protons from the PSB at 2 GeV to bring them at an energy of 26 GeV. During the energy ramp in the PS, the particles undergo the so-called " γ -transition", after which the added energy on the protons by the accelerating electrical field is not translated into an increase of velocity but into an increase of mass. The PS also provide heavy ions coming from the Low Energy Ion Ring (LEIR). If the PS does not feed the SPS, it can provide beams for the EAST area, AD and n_TOF. Since its construction, the different modifications increased the intensity of proton beams by a thousand [4].

- ***Super Proton Synchrotron***

The Super Proton Synchrotron (SPS) is the largest circular accelerator after the LHC, with a circumference of 7 km. The protons coming from the PS

are accelerated up to 450 GeV during 4.3 s before extraction to the LHC or experimental lines such as HiRadMat (High Radiation Material). Beams are delivered to the LHC through two different transfer lines allowing to fill each LHC ring in the clockwise and anti-clockwise direction. This accelerator allowed a deep understanding of the particle physics and the discovery in 1983 of the W and Z bosons, leading to the Nobel prize [5].

- ***Large Hadron Collider***

The Large Hadron Collider (LHC) is the largest and the most powerful accelerator worldwide with a circumference of 27 km and an energy of 6.5 TeV per ring and 13 TeV at the collision. It succeeded to the Large Electron-Positron collider (LEP) in 2008. A nominal LHC fill consists of 39 batches of 72 bunches coming from the SPS, corresponding to 2808 bunches per ring in total. The filling of the LHC can take more than 30 minutes. The particles circulate at speeds close to the speed of light thanks to the combination of a ultra high vacuum environment (10^{-12} mbar) and temperature of 2 K. Four main experiments: ATLAS, CMS, ALICE and LHCb are located at the four collision points [6].

In high-energy physics, physicists are pushing for an increase in intensity and beam energy and as well as a decrease of the beam sizes. New technically challenging projects are planned. The LHC Injector Upgrade (LIU) [7], the High Luminosity LHC Project (HL-LHC) [8] and the Future Circular Collider (FCC) [9] will improve the beam brightness in the next years (Fig. 3).

Wire-scanners (WS) are devices used since the 1980s to measure the transverse beam density profile, by moving a thin wire across the particle beam and detecting the emitted particles. Under some conditions, a degradation of the carbon fibers currently used in the SPS has been observed. To overcome this issue, a new generation of rotary wire-scanners has been designed to meet higher specifications. The whole engineering concept has been redesigned with new machining and manufacturing concepts. However, the solution of accelerating the scan to minimize the time of interaction between the beam and the wire leads to another problem. The size of the beam being small, the number of points to reconstruct the signal is not sufficient. For now, in the LHC, only 8 bunches can be scanned with the wire-scanners on 2808 possible, and even in the SPS it is not possible to make a measurement at flat top (maximal energy in the ring). Everything in this new WS was changed, except the centerpiece of the instrument: the wire. The same material with the same mounting system has been used from the beginning, i.e. partially copper coated carbon fiber. It was therefore studied whether another material could be used that would allow a slower scan without notable degradations. The problem is that first, most common wires (carbon fiber, gold coated tungsten,...) will not provide the required accuracy or will be degraded or even destroyed and second, the accuracy of the wire installation is currently mainly dependent on the operators and showed large variations in the calibration results.

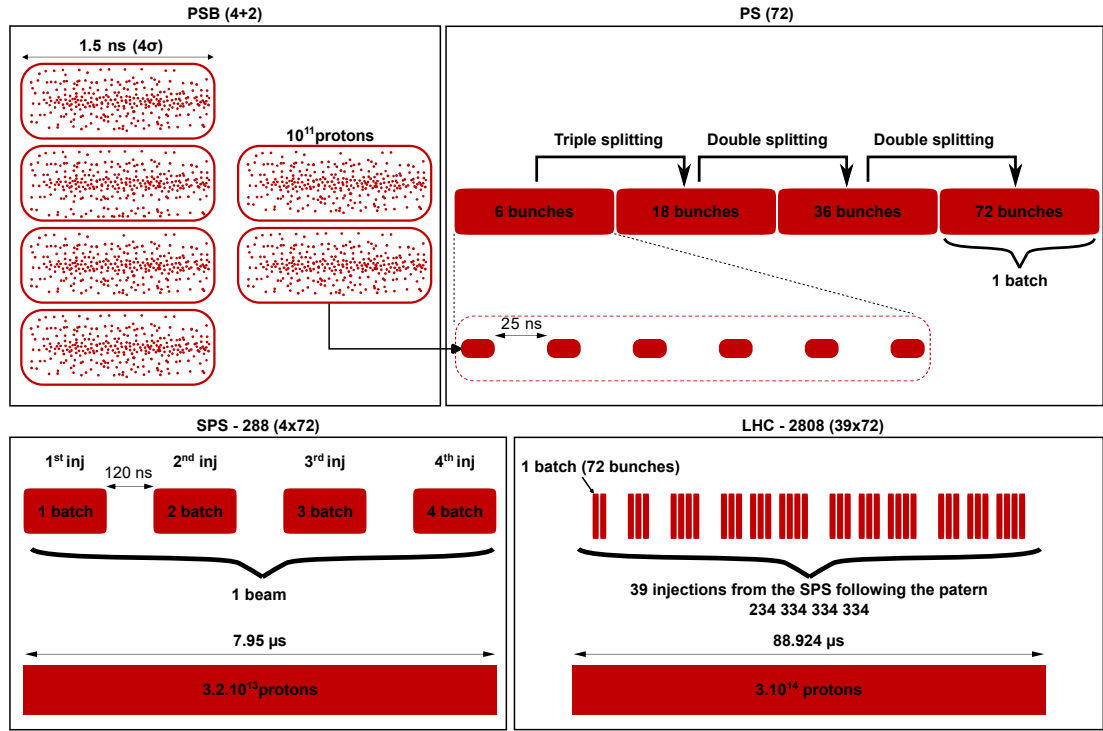


Figure 2: Beam structure along the LHC accelerator chain for a 25 ns beam. Bunches are created in 4 parallel lines in PSB and injected in PS in two consecutive cycles as 4+2. The bunches are triple split first and then two successive double splitted to get 72 bunches forming a batch. 4 batches are injected at 120 ns space in SPS creating the beam of 288 bunches. The LHC beam is formed by 39 injections of 72 bunches spaced by 25 ns to get 2808 bunches (from Ref [10, 11]).

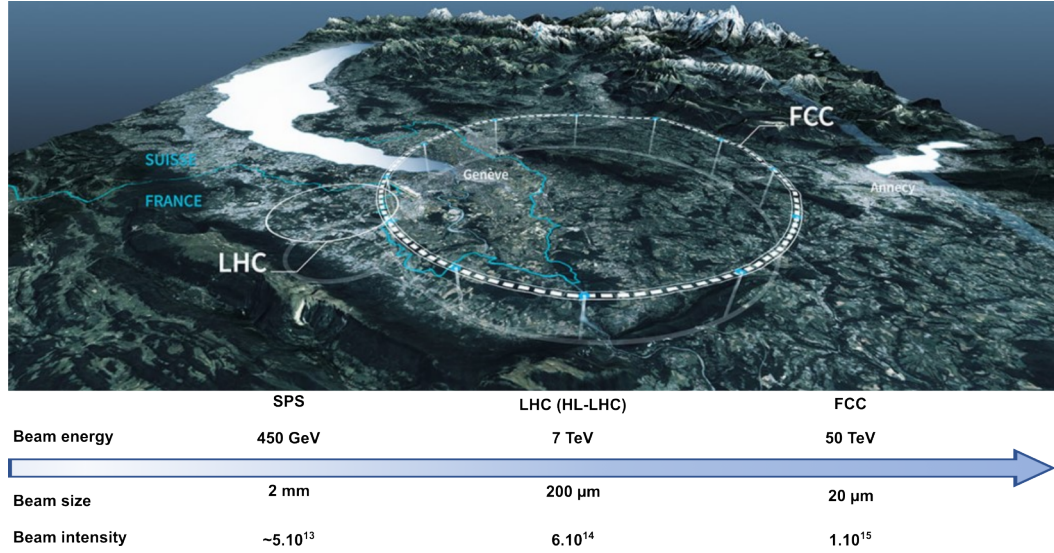


Figure 3: Increase of the energy and intensity parameters and decrease of the beam size for the HL-LHC and the future FCC project.

The use of a large number of various instruments at CERN shows a wide gap in the number of interactions with the beam for the interceptive and non-interceptive instruments (Fig. 4).

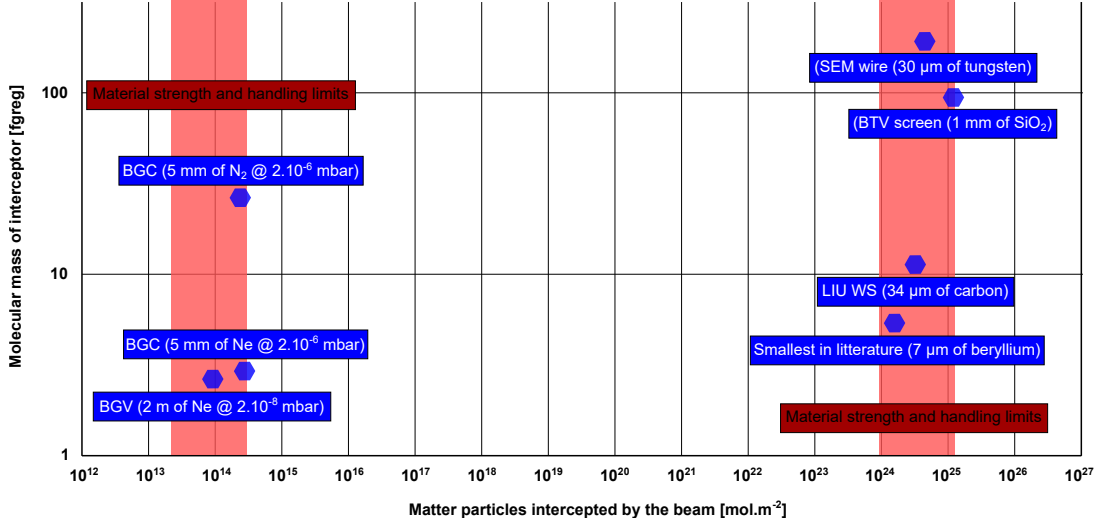


Figure 4: Material criteria to characterize the number of interactions (from Ref [12]).

The idea underlying this study was to consider this free space as potentially interesting in terms of research into a new material. If we find a material with sufficiently large mechanical and thermal properties, and a low density, this would make it possible to gain a few orders of magnitude in the number of interactions with the beam, and therefore to reduce the risks of overheating. These properties can be found in Carbon NanoTubes (CNT). Individual carbon nanotubes therefore seem to be a very promising material, showing impressive mechanical and thermal properties, much higher than any other material available on the market. However, due to their nanoscale, their handling and installation on our instrument would need a complete change in the current technology. Fortunately, recent development of technologies to assemble these tubes together to make long stranded yarns has opened up a new world of potential applications. A first step in the material transition would be to find out a solution with the microscopic assembly in yarns or wires of these CNT. Many academic institutes and industrial companies are interested in investing and developing these new Carbon NanoTube wires further. Indeed, even if several studies showed a large difference between the properties of a single nanotube and the properties of the wire assembly, methods to improve the mechanical properties of pristine yarns exist such as chemical treatment or densification by twisting but also by irradiation. The question at the origin of this study was therefore to assess the possibility of using CNT wires for the beam instrumentation.

Chapter 1

State of the art

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1.1 SUMMARY OF THE CHAPTER

This thesis work being located at the interface of various fields, namely physics of accelerators, interaction particles / matter and materials, it seems to be judicious to establish a state of the art on all these aspects. We start with the physics studied at CERN, in particular with the issues and research on which all technical and technological progress in terms of engineering is based. The structure of the beams evolving in the LHC acceleration chain as well as the parameters used throughout the study are detailed. We will also focus on particle / matter interactions, a major aspect for our problem. A detailed part concerning the wire-scanners will then be developed. We will end with a review of the studies made on the deterioration of the wires during the scans.

In a second step we focus on the individual carbon nanotubes, in order to understand their structure and their properties. The manufacturing process by Chemical Vapor Deposition as well as the various parameters influencing the final product will be detailed. The other two main methods are mentioned more briefly. The different mechanical properties of these CNTs are also discussed.

Finally, we will discuss carbon nanotube wires, microscopic assemblies of nanomaterial elements. The methods of obtaining the final product are detailed as well as the post-processing improvement treatments. A part is dedicated to the structure of these yarns and to the theory of fibers which make it possible to explain microscopic mechanical properties according to the properties of the constituent elements. Finally, we look at the few studies that concern the irradiation of CNT wires and the influence on the mechanical properties.

1.2 PHYSICS AT CERN

The LHC accelerator chain after the last upgrade of 2021 is composed of a linear accelerator (Linac4), three intermediate circular accelerators (PSB, PS and SPS) and the final circular accelerator (LHC) where the collisions occur. To ensure the good quality of the beam in term of size, emittance, intensity, astigmatism and centering, and increase the probability of collisions, a wide range of instruments control the beams. These instruments are located all around the chain, at strategic positions depending on the parameter to observe. They can be classified in two categories : the non-interceptive instruments and the interceptive instruments. The non-interceptive instruments can use electro-magnetic or electro-static methods (pickup [13], Beam Current Transformers (BCT) [14]), the radiation (synchrotron light monitor (BSRT) [15]) or make a direct particle measurement with the beam loss ionization chambers (BLM) [16]. The purpose of this work does not concern these instruments. The second category of instruments is the one with an interceptive solution. These instruments are also divided in two sub-categories: the first one with a beam-gas interaction and the second one with a beam-solid interaction. The beam-gas interaction instruments may measure the fluorescence

induced by the beam such as Beam-Gas Curtain (BGC) [17], the ionisation of the gas by the beam with the Beam-Gas Ionisation (BGI) [18], and the particle scattered by the beam with the Beam-Gas Vertex (BGV) [19]. These instruments are also not within the scope of our study. We focused on the instruments which interact directly with the beam. Among these instruments, we find the Beam TV monitor (BTV), the Faraday cup, the Secondary Electron Monitor (SEM) and Wire-Scanners (WS), detailed and discussed further.

1.2.1 Basics of physic of accelerators

1.2.1.1 Luminosity

1.2.1.1.1 General case

At CERN, there is a permanent aim to increase the energy and intensity of the beams in order to: 1- break matter into more energetic smaller pieces to observe new particles, and 2- increase the number of collisions in the various experiments to observe rare events. In particles colliders, two beams run in opposite directions, and collide at specific points. At CERN, there are four collision points in the LHC (ATLAS, CMS, ALICE and LHC-b). This method of collision allows to get an energy during the collision much larger than collisions with traditional fixed targets. At the collision points, various types of reactions are possible. Each of these reactions are characterized by a physical cross-section (σ_{eff}). The interaction event rate $\dot{n}$ is proportional to the physical cross-section by a factor called the luminosity ($\mathcal{L}$), so that

$$\dot{n} = \mathcal{L}\sigma_{eff} \quad (1.1)$$

The luminosity can be computed as [20]

$$\mathcal{L} = \frac{2N_{p1}N_{p2}}{t_{rev}} \iiint\limits_{-\infty}^{+\infty} B_1(x, y, z, z_0)B_2(x, y, z, z_0)dx dy dz dz_0 \quad (1.2)$$

with N_{p1} and N_{p2} the intensities (total number of protons) of beams 1 and 2 respectively, equal to $N_p = N_{pb}N_b$ where N_{pb} and N_b the number of particles per bunch and the number of bunch in the beam respectively, t_{rev} is the revolution time of the beam in the accelerator and B_1 and B_2 the characteristics for the beam 1 and the beam 2 respectively, equal to $B_1(x, y, z, z_0) = \rho_{b1x}(x)\rho_{b1y}(y)\rho_{b1z}(z - z_0)$ and $B_2(x, y, z, z_0) = \rho_{b2x}(x)\rho_{b2y}(y)\rho_{b2z}(z + z_0)$, with ρ_b the beam density distribution functions.

To solve this equation, an analytical calculation is not always possible and a numerical integration is required.

1.2.1.1.2 Luminosity of gaussian beams colliding head on

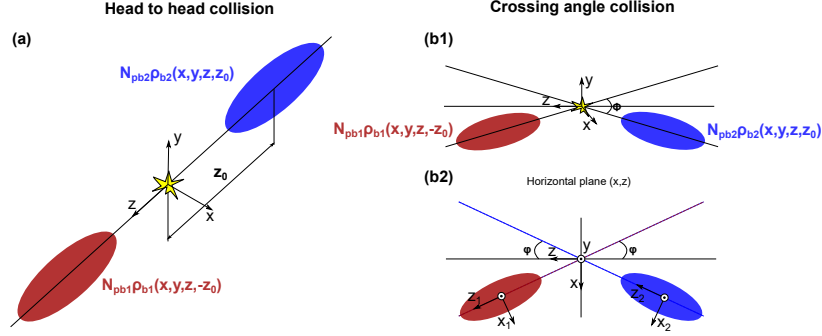


Figure 1.1: Schematic view: (a) A head to head colliding beam interaction; (b1) 3D view; (b2) Projection in the horizontal plane with the change of base (from Ref [20]).

The gaussian distribution is a model frequently used. The luminosity is determined by the overlap of the core of the distributions. The transverse profile in both horizontal and vertical planes can be expressed as

$$\rho_b(x, y) = \frac{1}{2\pi\sigma_{bx}\sigma_{by}} \exp\left(-\frac{1}{2}\left(\frac{x^2}{\sigma_{bx}^2} + \frac{y^2}{\sigma_{by}^2}\right)\right) \quad (1.3)$$

with σ_{bx} and σ_{by} the horizontal and vertical beam size respectively.

The beams also frequently have a gaussian distribution along the propagation axis. This profile is expressed as

$$\rho_b(z \pm z_0) = \frac{1}{\sqrt{2\pi}\sigma_{bz}} \exp\left(-\frac{1}{2}\frac{(z \pm z_0)^2}{\sigma_{bz}^2}\right) \quad (1.4)$$

with σ_{bz} the size of the beam on the longitudinal direction.

Considering that the two beams, circulating in opposite ways, have the same geometrical and intensity properties, we get $\sigma_{1x} = \sigma_{2x}$, $\sigma_{1y} = \sigma_{2y}$ and $\sigma_{1z} = \sigma_{2z}$. In the ideal case of exactly head to head collisions of beams at a speed very close to the speed of light, the kinematic factor K becomes 2. The luminosity can be then explicated from

$$\mathcal{L} = \frac{2N_{p1}N_{p2}}{(\sqrt{2\pi})^6 \sigma_{bx}^2 \sigma_{by}^2 \sigma_{bz}^2 t_{rev}} \iiint \exp\left(-\frac{1}{2}\left[\left(\frac{x}{\sigma_{bx}}\right)^2 + \left(\frac{y}{\sigma_{by}}\right)^2 + \left(\frac{z}{\sigma_{bz}}\right)^2 + \left(\frac{z_0}{\sigma_{z0}}\right)^2\right]\right) dx dy dz dz_0 \quad (1.5)$$

Using

$$\int_{-\infty}^{+\infty} e^{-at^2} dt = \sqrt{\frac{\pi}{a}} \quad (1.6)$$

we obtain the expression of the luminosity for a head to head collision of similar Gaussian beams

$$\mathcal{L} = \frac{N_{p1}N_{p2}}{4\pi\sigma_{bx}\sigma_{by}t_{rev}} \quad (1.7)$$

For the general case where the beam sizes are different, the luminosity is expressed as

$$\mathcal{L} = \frac{I_1 I_2}{2\pi\sqrt{\sigma_{1x}^2 + \sigma_{2x}^2}\sqrt{\sigma_{1y}^2 + \sigma_{2y}^2}t_{rev}} \quad (1.8)$$

1.2.1.1.3 Crossing angle collision

In reality, the beams do not collide perfectly head to head. A small angle (Φ) is introduced between the two beam directions (Fig. 1.1.b1). For example, in the LHC, the two beams collide with an angle of approximatively 300 μrad ($\sim 0.02^\circ$). This complicates the equation with a change of coordinate system. We define $\varphi = \Phi/2$ the angle between the direction of the beams and the theoretical axis z . The new coordinate system is explicitated as

$$\begin{cases} x_1 = x \cos \varphi - z \sin \varphi, & x_2 = x \cos \varphi + z \sin \varphi \\ z_1 = x \sin \varphi + z \cos \varphi, & z_2 = -x \sin \varphi + z \cos \varphi \end{cases} \quad (1.9)$$

The kinematic factor K becomes in a case where the two beams are not colinear, $K = 2 \cos^2 \varphi$. Replacing the new values of x_1, z_1, x_2 and z_2 in equation. (1.2), the luminosity can be expressed as

$$\mathcal{L} = \frac{N_{p1}N_{p2}}{4\pi\sigma_{bx}\sigma_{by}t_{rev}} S_r \quad (1.10)$$

with S_r an additional factor called luminosity reduction factor. Under the approximation of x and $\sin \varphi$ small, the terms of the type $\sigma_{bx}^k \sin^l(\varphi)$ and $x^k \sin^l(\varphi)$ are neglected for $k+1 \geq 4$, and $\sin \varphi \approx \varphi$. These considerations and for $\sigma_z \gg \sigma_{xy}$ allows to explicit S_r as:

$$S_r = \frac{1}{\sqrt{1 + \left(\frac{\sigma_{bz}}{\sigma_{bx}}\varphi\right)^2}} \quad (1.11)$$

An interpretation of this is to introduce the idea of an effective beam size σ_{eff} defined by

$$\sigma_{eff} = \sigma \sqrt{1 + \left(\frac{\sigma_{bz}}{\sigma_{bx}}\varphi\right)^2} \quad (1.12)$$

In the LHC¹, the luminosity is $\mathcal{L} = 1.2 \cdot 10^{34} \text{ cm}^{-2} \cdot \text{s}^{-1}$ for a head to head interaction and $\mathcal{L} = 1.0 \cdot 10^{34} \text{ cm}^{-2} \cdot \text{s}^{-1}$ for a crossing angle interaction. The luminosity reduction factor is then $S_r = 0.835$.

¹ Considering the following parameters: $N_{pb1} = N_{pb2} = 1.5 \cdot 10^{11}$, $N_{b1} = N_{b2} = 2808$, $\sigma_x = \sigma_y = 0.170 \text{ mm}$, $\sigma_z = 7.7 \text{ cm}$ and a crossing angle $\Phi = 285 \mu\text{rad}$

This clearly illustrates the interest and the importance of the precise measurements by beam instruments of the size, density profile and the position of the beams.

A large number of parameters characterizes a beam, such as emittance, size, intensity, energy, particles, revolution frequency, wakefields, synchrotron tune, beta function, energy loss per turn, tuning angle, twiss parameters and more [21]...

1.2.1.2 Beam properties

1.2.1.2.1 Beam guiding

As we saw, the luminosity is the ultimate factor of beams that physicists want to maximize. This factor depends on numerous other beam parameters that are controlled all along the LHC accelerator chain. In the LHC accelerator chain at CERN, the radio-frequency (RF) cavities accelerate, the magnetic dipoles guide and the magnetic quadrupoles focus the beam following a reference trajectory.

The RF cavities are large hollow and metallic structures with an electromagnetic field inside. They are enclosed in cryo-modules which allow to work at a temperature close to the absolute zero (2-4.5 K [22]) so that the cavities are in a supra-conductor state. Each cavity is driven by a vacuum tube called klystron which allows medium and high power amplifications at narrow band in microwaves providing electron beams. Beams are modulated in intensity at a frequency depending on the accelerator (PSB, PS, SPS or LHC). A waveguide transmit the energy to the cavity. The electromagnetic field of the cavity oscillates sinusoidally and the modulation is synchronized with the revolution frequency. Particles arriving in the cavity with the appropriate energy are not changed whereas particles with different energies are slowed down or accelerate to get the good energy [23].

The trajectory is guaranteed along the acceleration cycle with magnetic dipoles in which a magnetic field curves the trajectory of the charged particles through the Lorentz force. These dipoles are designed to have an action on the horizontal plane only.

The quadrupole magnets are the key parts to focus the beam in both horizontal and vertical planes. They compensate the angle difference at the exit of the dipole. The focus is allowed by the difference of load applied on the particle depending on its position. The force is stronger for particles further away from the center of the quadrupole center. The focusing effect of a quadrupole apply on a single transverse plane only. If it increases in the horizontal plane, it decreases for the vertical plane. A net focusing in both transverse planes can be obtained using consecutive quadrupoles with opposite polarities [24].

1.2.1.2.2 Momentum (p) and energy (E)

In the accelerators, the speed of the particles approaches the speed of light. The parameter which quantifies the ratio between the speed of the particle (v_p) and the speed of light (c) is the relativistic factor β expressed as

$$\beta = \frac{v_p}{c} \quad (1.13)$$

The energy of the beam is defined from the average momentum of each particle expressed in $[\text{MeV} \cdot \text{c}^{-1}]$ and the mass of the incident particle expressed in $[\text{MeV} \cdot \text{c}^{-2}]$. For the rest of this study, we will focus on proton beams only. The mass of a proton is $1.672 \cdot 10^{-27} \text{ kg}$ or $938 \text{ MeV} \cdot \text{c}^{-2}$, which is the unit commonly used for the different calculations. The charge of the proton is $1.602 \cdot 10^{-19} \text{ C}$.

Using relativistic mechanics, the energy of the beam is defined on the average energy of all protons in the beam and can be computed by

$$E_{beam} = \sqrt{p_{beam}^2 c^2 + m_p^2 c^4} \quad (1.14)$$

A Lorentz factor γ is then introduced such that $E_{beam} = \gamma m_p c^2$, so that, using [equation. \(1.14\)](#), γ can be expressed in terms of

$$\gamma = \sqrt{1 + \left(\frac{p_{beam} c}{m_p c^2} \right)^2} \quad (1.15)$$

Since it can be demonstrated that we also have $\vec{p} = \gamma m_p \vec{v}$, Lorentz factors β and γ can then be related by

$$\gamma = \frac{1}{\sqrt{1 - \beta^2}} \quad (1.16)$$

$$\beta = \sqrt{1 - \frac{1}{\gamma^2}} \quad (1.17)$$

with $\gamma \in [0, +\infty[$ and $\beta \in [0, 1[$.

Finally, the kinetic energy $E_k = E_{beam} - m_p c^2$ can be written as a function of γ with

$$E_k = (\gamma - 1) m_p c^2 \quad (1.18)$$

At CERN, for low energies ($E \leq 2 \text{ GeV}$), beams which circulate in the Linac, the PSB and the PS, are characterized by their energy, whereas beams at high energies ($E_k > 2 \text{ GeV}$), are characterized by their momentum. For high momenta, the energy (total), the kinetic energy and the momentum tend to the same value. We can speak indifferently of one or the other.

For the rest of the study, I will therefore use the kinetic energy and consider that kinetic energy for the SPS to be 450 GeV (instead of 449 GeV) and 7000 GeV (instead of 6999 GeV) for the LHC.

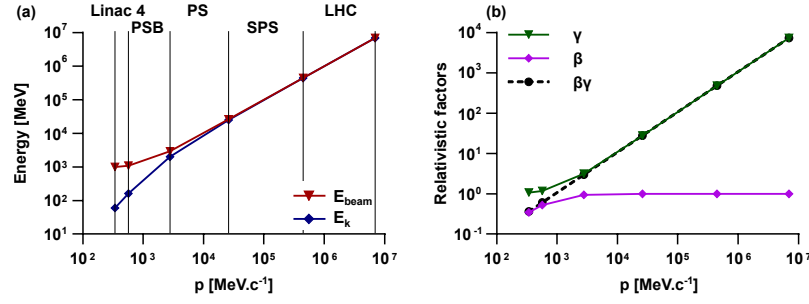


Figure 1.2: (a) Beam energy; (b) Lorentz factor vs momentum for the LHC accelerator chain.

1.2.1.2.3 Beam emittance (ϵ) and beam size (σ_b)

The position of every particle of a beam can be plotted on a phase space diagram (Fig. 1.3.a). If we consider the particles centered around the reference orbit, we can define an area in the phase space diagram occupied by the particles (Fig. 1.3.b).

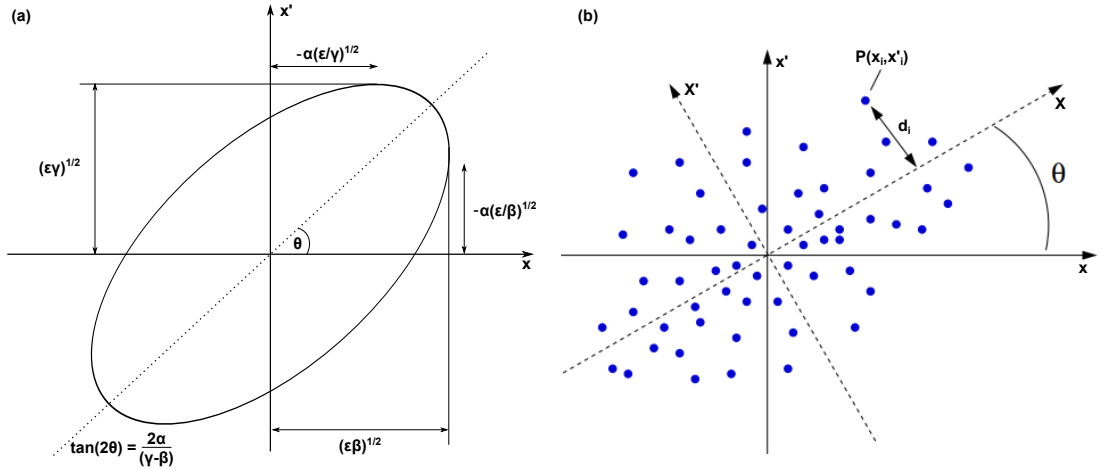


Figure 1.3: (a) Motion of a single particle, at a longitudinal location, maps an ellipse in the phase space relative to one transverse degree of freedom. The ellipse is characterized by the Courant-Snyder parameters α_{cs} , β_{cs} and γ_{cs} ; (b) Particles distribution in phase space. The cartesian X, X' axis are chosen in order to minimize the sum of the square distances between the points and the X axis.

For convenient reasons, the coordinate system to two axis X, X' centered on the distribution barycenter and orientated in order to minimize the sum of the square distances of each point from each axis. The distance d_i of the point P(x_i, x'_i) from the axis X is

$$d_i = |x'_i \cos \theta - x_i \sin \theta| \quad (1.19)$$

with θ the rotation angle of X with respect to x.

The rotation angle θ can be defined by minimizing the distance using

$$\frac{d}{d\theta} \left(\frac{1}{N} \sum_{i=1}^N d_i^2 \right) = \frac{d}{d\theta} \sigma_X^2 = 0 \quad (1.20)$$

This results in

$$\tan(2\theta) = \frac{2\overline{xx'}}{\overline{x^2} - \overline{x'^2}} \quad (1.21)$$

where

$$\overline{x^2} = \frac{1}{N} \sum_{i=1}^N x_i^2 \quad (1.22)$$

$$\overline{x'^2} = \frac{1}{N} \sum_{i=1}^N x_i'^2 \quad (1.23)$$

$$\overline{xx'} = \frac{1}{N} \sum_{i=1}^N x_i x_i' \quad (1.24)$$

are the central second-order moments of the distribution being $\overline{x} = \overline{x'} = 0$. The value of θ also defines the orientation of X' to $\theta + \pi/2$. referring to the new cartesian system, the variances of the particle distances from X to X' result

$$\sigma_X^2 = \frac{1}{2} \left(\overline{x^2} + \overline{x'^2} + \frac{2\overline{xx'}}{\sin(2\theta)} \right) \quad (1.25)$$

$$\sigma_{X'}^2 = \frac{1}{2} \left(\overline{x^2} + \overline{x'^2} - \frac{2\overline{xx'}}{\sin(2\theta)} \right) \quad (1.26)$$

The variances of the particle distribution in the phase space diagram can be assigned as semi-axes of the beam envelope ellipse of equation

$$\frac{X^2}{\sigma_X^2} + \frac{X'^2}{\sigma_{X'}^2} = 1 \quad (1.27)$$

The area of the ellipse is then $A_e = \pi \sigma_X \sigma_{X'}$ and the beam emittance (ε) is

$$\varepsilon = \frac{A_e}{\pi} = \sigma_X \sigma_{X'} \quad (1.28)$$

By a rotation of $-\theta$, the ellipse can be expressed with respect to the axis x,x' [25]

$$x^2 \sigma_{x'}^2 - 2xx' r \sigma_x \sigma_{x'} + x'^2 \sigma_x^2 = \sigma_x^2 \sigma_{x'}^2 \quad (1.29)$$

with σ_x and $\sigma_{x'}$ are the standard deviations and $r = \overline{xx'}/\sqrt{\overline{x^2} \overline{x'^2}}$ is the correlation coefficient. Using the parametrization

$$\sigma_x = \sqrt{\beta_{cs} \varepsilon} \quad (1.30)$$

$$\sigma_{x'} = \sqrt{\gamma_{cs}\varepsilon} \quad (1.31)$$

$$4r\sigma_x\sigma_{x'} = -\alpha_{cs}\varepsilon \quad (1.32)$$

Considering [equation. \(1.28\)](#), [equation. \(1.29\)](#) becomes

$$\varepsilon = \gamma_{cs}x^2 + 2\alpha_{cs}xx' + \beta_{cs}x'^2 \quad (1.33)$$

which reproduces the Courant-Snyder invariant. This result explain the concept of beam emittance as the area of the ellipse with contour defined by particles' dispersion in phase space [\[21\]](#).

For a beam with a Gaussian shape in the transverse plane, the standard deviation of the distribution coincides with the Gaussian width σ_b . Since by definition, the emittance is constant during the travel of the beam, the beam size varies with the variation of the beta function (β_f), which represents the amplitude of the modulation due to changing focus strength. The beam size decreases with the increase of the energy. Considering a particle at one standard deviation of the transverse distribution and neglecting the dispersion of the beam, the horizontal and vertical size of the complete beam size at a specific position (z) can be then calculated by

$$\sigma_{b(h,v)}(z) = \sqrt{\beta_{f(h,v)}(z)\varepsilon_{(h,v)}(z)} \quad (1.34)$$

One parameter also frequently used is the full width at half maximum (FWHM). For a normal distribution, it is defined as

$$\text{FWHM} = 2\sigma_b\sqrt{2\ln 2} \quad (1.35)$$

1.2.1.3 Interaction with matter

1.2.1.3.1 Mean free path (λ)

The mean free path is the mean distance traveled by a particle in matter between two successive collisions which modify its direction and energy. This distance depends on the physical cross-section and the number of targeted particles per volume unit:

$$\lambda = \frac{1}{\Sigma} \quad (1.36)$$

where Σ is the macroscopic cross-section per unit volume. Σ can be expressed in terms of microscopic cross-section (σ_{eff}) and density of atom per unit volume $\rho_{at} = \mathcal{N}_A \frac{\rho}{M}$ with

$$\Sigma = \rho_{at}\sigma_{eff} \quad (1.37)$$

1.2.1.3.2 Cross-section (σ_{eff})

In physics, the cross-section is a measure of the probability that a specific process will happen over a given surface area unit. In our particular case, we will focus on the inelastic scattering with an electron or the nucleus of the material crossed by the beam. The inelastic scattering is a scattering process in which the kinetic energy of the incident proton is not conserved. Some of the energy is lost or increased [26]. For the elastic scattering, the kinetic energy is conserved in the center of mass frame but the trajectory is modified from its original one [27]. The physical cross-section represents the area, in the plane perpendicular to the particle's trajectory, around the particle, in which an event has a certain probability of occurring. Experiments were performed in HERA (Hambourg, Germany) leading to a phenomenological equation (Eq. (1.38)) which gives at worst 7 % of error between 150 and 920 GeV

$$\sigma_{eff} = 52.86A^{0.7694} \quad (1.38)$$

Thus leads to a cross-section for carbon of $\sigma_{eff} = 209.8 \text{ mb}^2$.

1.2.1.3.3 Radiation length (X_0)

The radiation length, expressed in g.cm^{-2} is a characteristic of a material related to the energy lost during interaction with particle at high energy.

$$\frac{1}{X_0} = 4 \left(\frac{\hbar}{m_e c} \right)^2 Z(Z+1) \alpha^3 n_a \log \left(\frac{183}{Z^{1/3}} \right) \quad (1.39)$$

with n_a the number density of nuclei, $\hbar$ the reduced Planck constant ($\sim 1.055 \cdot 10^{-34} \text{ J.s}$), m_e the electron mass (in MeV.c^{-2}), c the speed of light and α the fine structure constant ($\simeq 1/137.035$)

1.2.1.3.4 Stopping power dE/dx

The stopping power defines the linear mean loss of energy when an incident particle (in our case protons) goes through a material. The particle has a certain probability of interaction with the material, nuclei or electrons. The interaction induces a loss of a part of its initial energy and a deviation from the original trajectory. The stopping power includes the collision and radiation contributions. At high energy, the electronic contribution is predominant, while the nuclei contribution is the largest for low energy. The processes of this loss of energy depend on the irradiated material and the nature of the incident particle (electron, positron, heavy charged particles, neutrons...). Processes causing this effect include inelastic collisions with the electrons and elastic scattering on the nuclei also called Coulomb scattering. Other processes are also involved such as Bremsstrahlung radiation effect and Cherenkov radiation.

We will only focus on the protons, considered as an heavy particle. Protons lose their energy mainly by ionizing or exciting the atoms of the material. The Bethe

² $1 \text{ mb} = 1 \text{ mbarn} = 10^{-27} \text{ cm}^2$

equation describes the stopping power (Eq. (1.40)), expressed in $[\text{MeV.cm}^2.\text{g}^{-1}]$, of a material for a specific particle which is also the mean rate of energy loss during the travel of a particle through a medium [28]

$$-\frac{1}{\rho} \left\langle \frac{dE}{dx} \right\rangle = \frac{KZ}{A} \frac{1}{\beta^2} q_p^2 \left[\frac{1}{2} \ln \left(\frac{2m_e c^2 \beta^2 \gamma^2 T_{max}}{I^2} \right) - \beta^2 - \frac{\delta(\beta\gamma)}{2} \right] \quad (1.40)$$

with ρ , Z , A and I are the density, the atomic and mass number and the characteristic ionization constant of the material respectively, K is a factor depending on the classical electron radius (r_e), the electron mass (m_e) and the Avogadro number ($\mathcal{N}_A$) defined as $K = 4\pi\mathcal{N}_A r_e^2 m_e c^2$, q_p is the charge of the incident particle, β and γ are the relativistic parameters, T_{max} is the maximum energy transfer possible in a single collision (Eq. (1.41)) and $\delta(\beta\gamma)$ is a correction factor due to the density effect.

$$T_{max} = \frac{2m_e c^2 (\beta\gamma)^2}{1 + 2\gamma \frac{m_e}{m_p} + \left(\frac{m_e}{m_p} \right)^2} \quad (1.41)$$

The structure of the equation. (1.40) induces a high energy deposition in the material for particles with a high charge q_p and a low velocity β [29].

The total stopping-power comes mainly from the electronic interactions and varies with the energy. It increases from 1 keV to 150 keV then decreases up to 1.5 GeV before increasing again. Values vary from $1.76 \text{ MeV.cm}^2.\text{g}^{-1}$ to $786 \text{ MeV.cm}^2.\text{g}^{-1}$ and from $1.14 \text{ MeV.cm}^2.\text{g}^{-1}$ to $121.4 \text{ MeV.cm}^2.\text{g}^{-1}$ for carbon and tungsten respectively (Fig. 1.4).

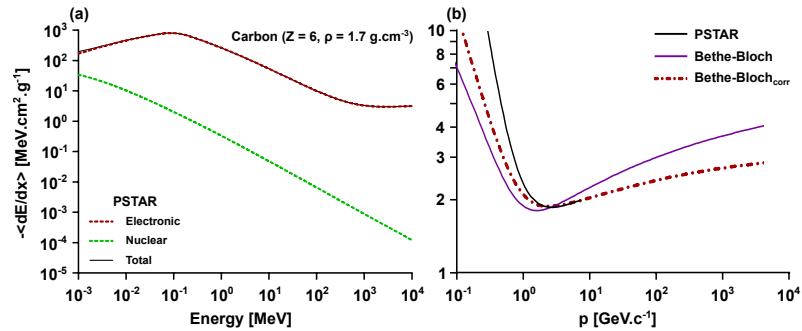


Figure 1.4: (a) Electronic (red dashed line), nuclear (green dashed line) and total (black continuous line) stopping power in carbon calculated for protons according to methods described in ICRU Reports 37 and 49 (from Ref [28]); (b) Stopping power for intermediate energy (100 MeV - 10 TeV) according to the PSTAR data up to 10 GeV (black line), simplified Bethe-Bloch equation (purple line) and Bethe-Bloch equation considering the density effect correction (red dashed line).

The stopping power for energy in the range of 1 keV to 10 GeV can be obtained for various material from the PSTAR database [30]. For energy above 10 GeV, the Bethe-Bloch equation has to be considered.

1.2.1.4 Secondary electron theory

During the passage of the beam through a material, it will transfer some energy to the electrons of the material. Depending on the energy these electrons receive, they can be excited to a higher energy level or acquire enough energy to be extracted from the material. The latter process is known as Secondary Electron Emission (SEE) and is the method used in SEM grid instruments (Secondary Electron Monitor) for the characterization of beam profiles at CERN. This effect is a surface effect that can therefore be quantified at the entry and exit of the material. The first step is the creation of secondary electrons in the volume of the material. The minimum energy to create a secondary electron is the energy required to excite an electron above the Fermi level of the conduction band. The second step of the SEE process is the diffusion inside the material. The electrons which cross the material lose part of their initial energy. The distance an electron travels in the material decreases with increasing energy loss. The last step of SEE is the emission of electrons. To extract itself from the material, the electron must have enough energy to free itself from the surface potential, characterized by the work function $W = -(E_F + \phi e)$, with E_F the Fermi level in the material, ϕ the electrostatic potential in the vacuum nearby the surface and e the charge of the electron [31, 32]. Furthermore, the emission process must be directed inside an escape cone of maximal angle (θ_{max}) with respect to the normal of the surface

$$\theta_{max} = \arccos\left(\sqrt{\frac{W}{E_i}}\right) \quad E_i \geq W \quad (1.42)$$

with E_i the energy of the incident particle.

The Secondary Electron Yield (SEY) is the parameter which allows the quantification of the SEE. It expresses an average number of electrons emitted from the material per incident particle. It is given by [33]:

$$\text{SEY} = 0.01 L_s \left. \frac{dE}{dx} \right|_{el} \left[1 + \frac{1}{1 + 5.4 \cdot 10^{-6} \left(\frac{E_k}{m_p c^2} \right)} \right] \quad (1.43)$$

with dE/dx the electronic energy loss expressed in $[\text{eV} \cdot \text{cm}^{-1}]$, E_k and $m_p c^2$ the kinetic energy and the mass energy of the incident particle expressed in $[\text{eV}]$, L_s the thickness of the material from which the low energy electrons can escape, expressed in $[\text{cm}]$ as

$$L_s = \frac{1}{3.68 \cdot 10^{-17} \left(\frac{\rho N_A}{M} \right) Z^{\frac{1}{3}}} \quad (1.44)$$

with ρ the density, M the molar mass and Z the atomic mass of the target. N_A is the Avogadro number.

From the SEY, given the input parameters, the number of secondary electrons emitted from the material can be calculated by multiplying the SEY by the number of incident particles, which will be useful in our simulations of irradiation of a carbon nanotube wire acting as the sensitive element of our wire-scanners described in the next section.

1.2.2 Wire-scanners

1.2.2.1 Principle and development

As it has been previously shown, the knowledge of the position and the distribution of the particles in the transverse plane is necessary to improve the luminosity. Wire-scanners are devices used to measure the transverse beam density profile by moving a thin wire across the particle beam. The interaction between the beam and the wire creates a secondary particle shower with an intensity proportional to the number of particles crossing the wire. These secondary particles are intercepted by a scintillator positioned downstream of the wire and coupled with a photo-multiplier which amplifies the intensity of the light produced. The acquisition of the wire position and the signal intensity are combined to reconstruct the transverse beam density profile [34, 35].

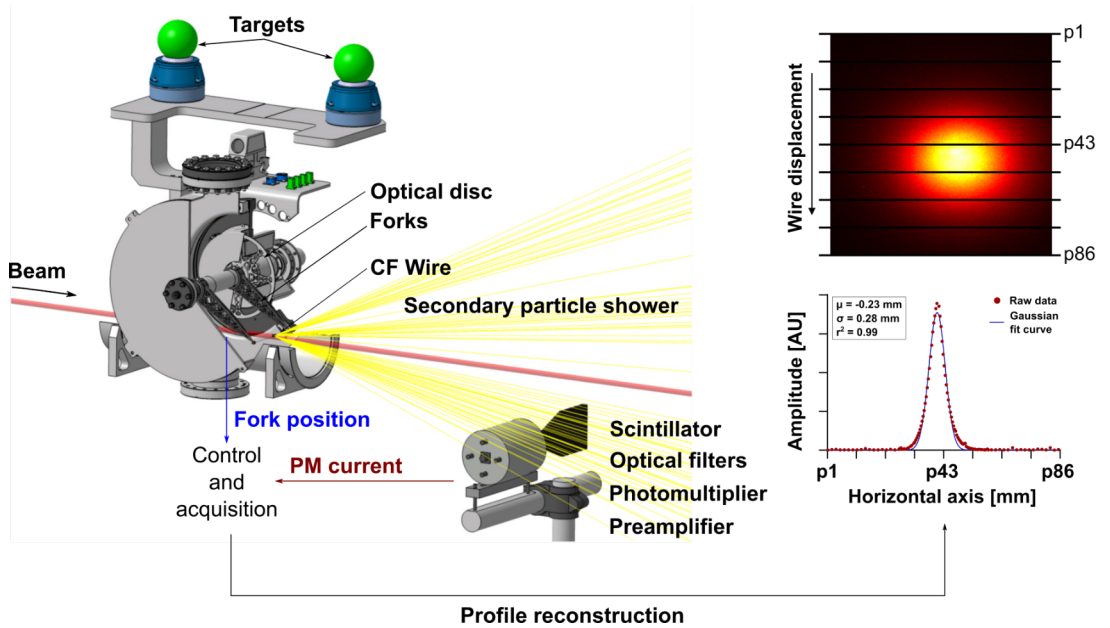


Figure 1.5: (a) Sketch of the new generation of fast wire-scanners (WS) for beam profile measurement in the LHC accelerator chain with creation of secondary particle shower (yellow) during the interaction between the beam and the wire; (b) Illustration of the intersection points in the transverse plan between the wire and the beam; (c) Profile reconstruction of the vertical axis with the raw data (blue dots) and gaussian fit (red line).

First studies on the WS occurred in the 50's [36, 37]. Even if the use was different from our need, it laid the foundations of a system of measurement. First mentions of such concept at CERN appears in the end of the 60's beginning of the 70's [38]. Ten years later appeared the first real studies, considering all the aspects of the topic : beam size, interaction with matter, scan speed, mechanical constraints and thermal issues [39]. It really developed as a reliable system in the 80's [40, 41]. The first WS moved linearly with a stepper motor [42, 43] which required very low scan speeds, of $\approx 1 \text{ m.s}^{-1}$ [44, 45]. Various studies on this instrument were carried, from the influence of the diameter of the wire [46] to its thermal behavior [43, 47, 48]. Another generation of rotary WS was then designed to meet higher specifications [49] in the Proton Synchrotron Booster (PSB) and the Proton Synchrotron (PS). However, in the near future, the beam will progressively be smaller and more intense for HL-LHC and FCC. This will lead to two main issues with the current generation of wire-scanners: an overheating of the wire [50, 51, 52] and an insufficient number of interaction points to reconstruct the profile of the beam [53]. Due to this higher brightness, the principal wire failure mode is expected to be due to fast increase of temperature leading to melting or surface sublimation. The loss of matter weakens the wire until the failure [50]. In order to address the overheating issue, a new generation of fast wire-scanners [35, 54] was developed at CERN (Fig. 1.5), for the whole accelerator chain, from PSB to the Super Proton Synchrotron (SPS). The whole engineering concept has been redesigned with new machining and manufacturing concepts [55, 56]. For example, the forks were redesigned to be lighter and stronger and reduce the impedance, source of heat loads [54]. There were obtained in a single part by 3D printing from titanium [55]. The major improvement is the increase of the scan velocity up to 20 m.s^{-1} , which allows to reduce the beam/wire interaction time and consequently the increase of temperature. Everything in this new WS was changed, except the centerpiece of the instrument: the wire. The same material with the same mounting system has been used from the beginning, i.e. partially copper coated carbon fiber. The problem is that first, most common wires (carbon fiber, gold coated tungsten and copper coated carbon fibers) will be destroyed in the HL-LHC conditions [50] and second, the precision of the wire installation is currently mainly dependent on the technicians who install the wire manually and is prone to large variations in the calibration results.

1.2.2.1.1 Material

The material of the wire plays a dominating role in the interaction with the beam. The number of interactions, and then, the deposited energy, between the wire and the beam depends on the atomic number Z and the density. Beryllium and carbon have both a low Z (4 and 6 respectively) with low density (1.85 and $1.4\text{-}2.3 \text{ g.cm}^{-3}$ respectively) . Beryllium has been suggested for the SPS [39, 57] and the LEP [40]. If its mechanical and thermal properties are very close to the properties of carbon [58], its high toxicity is however problematic. Its use as a wire for the instruments has therefore been stopped. Tungsten, which has a high Z (74),

is frequently used for its large strength and high melting temperature [42]. It also allows to get a good signal by increasing the number of interactions. Anecdotaly, silicon carbide used in J-Parc has been tested for electron beams [59].

1.2.2.1.2 Diameter

The wires, depending on the specifications (measurement of proton or electron beams, size of the beam, materials), have a large variety of diameters. Diameters of the wires may vary from 4 [42] to 36 μm for carbon fiber [41]. For beryllium, the diameter is slightly larger with 50 to 100 μm [39, 57]. Wires in tungsten have a large range of diameter, from 10 to 40 μm [42]. The diameter has an impact on the number of interactions with the beam. There is a balance between the signal produced, the energy deposited in the wire and the interference effect on the beam structure. The most effective solution is to reduce the diameter of the wire since the number of interactions as well as the scattering per hit will be reduced.

1.2.2.1.3 Scan speed

The scan speed is determined by many factors. First, the type of the instrument, linear or rotational, implies different design and drive the maximum acceptable speed (Fig. 1.6). Usually, the linear wire-scanners are the slowest scanners with speeds from 500 $\mu\text{m.s}^{-1}$ [60] to 1 m.s^{-1} [61, 62, 44, 59]. Some rotary wire-scanners have been designed to reach 30 m.s^{-1} [35, 54, 63] however the resolution increases with the decrease of the speed. An idea is then to slow down the scan to increase the signal and the resolution. However, doing that, depending on the conditions, the wire can be submitted to high thermal load due to a too high energy deposited by the beam, leading to a failure [50, 42], which favors high speeds. A compromise had to be found. This is the reason why scan speeds never exceed 20 m.s^{-1} .

1.2.2.2 Installation at CERN

Wire-scanners are installed all along the LHC accelerator chain, at specific and strategic locations. The new fast wire-scanners are so far installed in the PSB, PS and SPS. Studies are in progress to assess the possibility of installing them in the LHC, but the environmental conditions in the LHC are more constraining than for the other rings.

In the PSB, rotary wire-scanners are installed on the four lines of the ring, at different strategic points (Table. 1.1).

So far, only carbon fiber of 34 μm are used for the new instrument in the PSB, PS and the SPS. The speed of the scan for the PSB can be set up to the maximal speed (20 m.s^{-1}) because the beam size is large enough. Tests have been carried out in the SPS for speeds of 1, 6, 10, 15 and 20 m.s^{-1} . For now, the nominal speed is 1 m.s^{-1} .

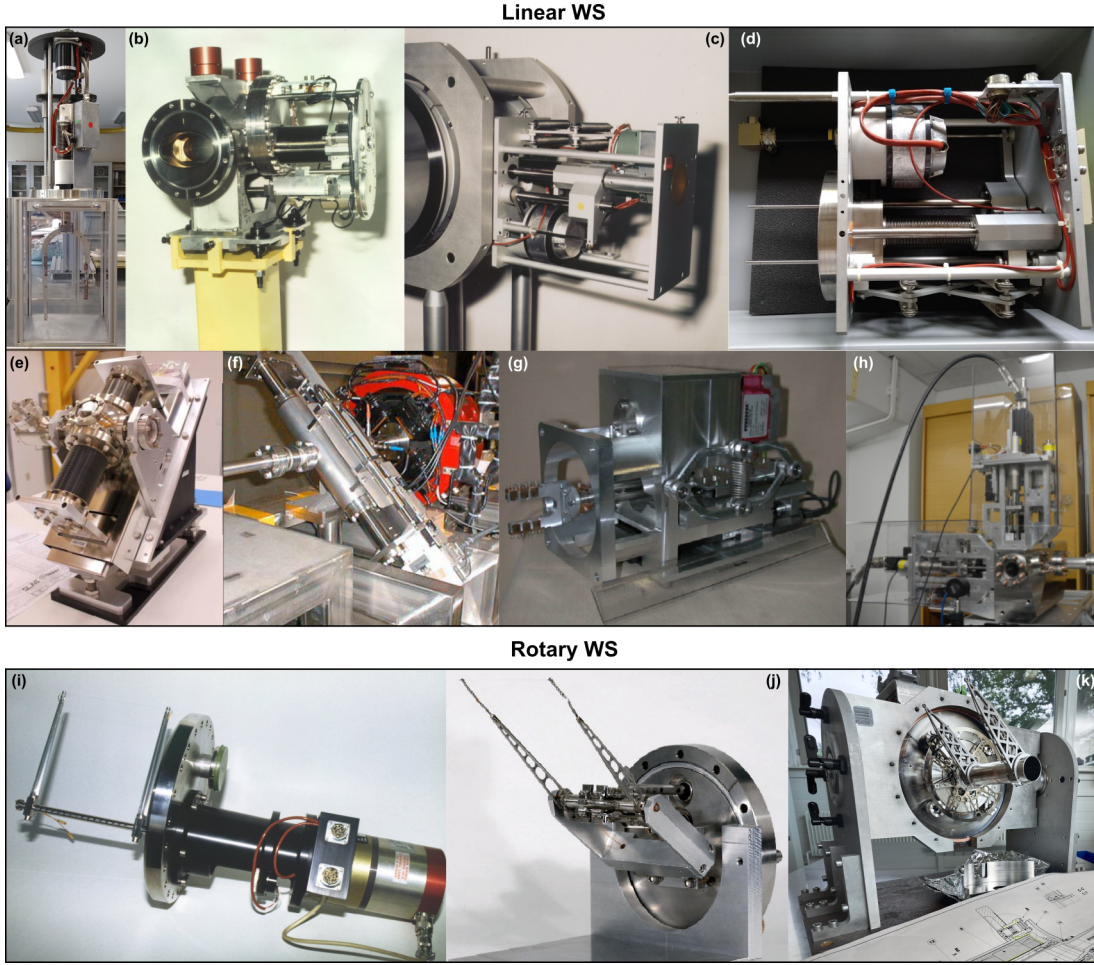


Figure 1.6: Linear wire-scanners: (a) WS for Linac4 at CERN; (b) WS for SPS first generation [64]; (c) WS for SPS second generation; [64]; (d) WS currently used in the SPS; (e) WS for TTF II in DESY [61]; (f) WS for the MicroTCA Environment for the European XFEL [62]; (g) WS at the LCLS [44]; (h) WS for ATF [60]. Rotary wire-scanners: (i) for SPS at CERN; (j) for PS at CERN; (k) for PSB, PS and SPS new generation of fast instrument [65].

Table 1.1: Rotary WS information for the PSB.

Scanner name	Type	Location	Orientation	Quantity
BR.BWS.2L1.V_ROT	Old	2L1	Vertical	4
BR.BWS.2L1.H_ROT	Old	2L1	Horizontal	4
BR.BWS.4L1.H_ROT	New	4L1	Horizontal	4
BR.BWS.11L1.V_ROT	New	11L1	Vertical	4

The wire-scanners allow to get the beam size at a specific location. Values of the relevant parameters for the PSB, PS and the SPS and the minimum and maximum beam sizes measured for a LHC 25 ns beam are given in the [table. 1.4](#).

Table 1.2: Rotary WS information for the PS.

Scanner name	Type	Location	Orientation	Quantity
PR.BWSH54	New	PR.SD54	Horizontal	1
PR.BWSV64	New	PR.SD64	Vertical	1
PR.BWSH65	New	PR.SD65	Horizontal	1
PR.BWSH68	New	PR.SD68	Horizontal	1
PR.BWSV85	New	PR.SD85	Vertical	1

Table 1.3: Rotary WS information for the SPS.

Scanner name	Type	Location	Orientation	Quantity
BWSSRC.41677	New	41677	Vertical	1
BWSSRC.41678	New	41678	Vertical	1
BWSSRC.51737	New	51737	Horizontal	1
BWSSRC.51738	New	51738	Horizontal	1

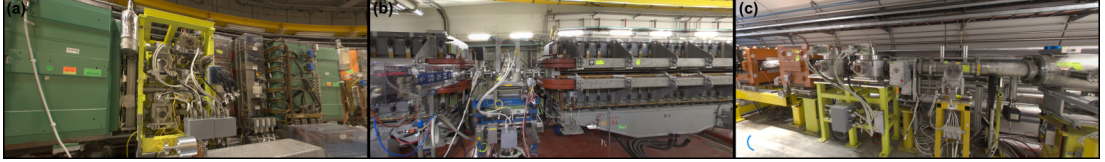


Figure 1.7: (a) New WS installed on the four lines of the PSB ring; (b) Horizontal WS installed in the PS; (c) Vertical WS installed in the SPS, near old linear WS.

This table shows the injection / extraction data for the LHC accelerator chain. The data with the star (*) are the ones provided by OP (Operation) and the other ones are calculated using the [equation. \(1.14\)](#) and the [equation. \(1.18\)](#).

1.2.2.3 Wire-scanners over the world

Wire-scanners are not only used at some places other than CERN. Various laboratories developed their own prototypes with specifications responding to their own needs. In 1964, a rotating WS using a single wire was produced, able to scan a beam in two dimensions [66]. Oxford also developed a device on the base of the rotation of a frame [67]. The SLAC team designed a linear wire-scanner used for monitoring electron beam profiles [68]. At CERN, the monitoring is made by collecting the secondary particles on a scintillator, it can also be made by measuring the emission of electrons from the wire after the passage of the beam. In this case, a conductive material is required. Linear WS with a tungsten wire [69] mounted on steel studs are also used at SLAC. This instrument was used for medium energy (1 - 50 GeV) and pulsed beams of electrons. Wire-scanners are also used in the KEKB linac for electrons in Japan [70] or with protons in SNS (Oak Ridge National Laboratory) [71] and at DESY [72].

Table 1.4: Machine and beam parameters at injection for the accelerator chain complex.

Machine parameters			PSB	PS	SPS	LHC
Ring circumference	C_r	km	0.157	0.628	6.9	26.6
Momentum	p	GeV.c ⁻¹	0.571	2.8	26*	450*
Energy (total)	E_b	GeV	1.098	2.9	26	450
Energy (kinetic)	E_k	GeV	0.160*	2.0*	25	449
Lorentz factor	γ	-	1.171	3.1	28	480
Relativistic factor	β	-	0.520	0.9	1	1
	$\beta\gamma$	-	0.608	3.0	28	480
Beam parameters						
Revolution freq	f_{rev}	Hz	991968	436647	43347	11245
Revolution period	t_{rev}	μ s	1.01	2.29	23.1	88.9
Beam size (horizontal)	σ_h	mm	1.6 - 13.5	0.8 - 13.0	0.2 - 8.7	0.16 - 1.19
Beam size (vertical)	σ_v	mm	1.2 - 9	0.5 - 8.8	0.3 - 6.7	0.22 - 1.79
Bunch length (4 σ)	l_b	ns	180	180	3.9	-
Bunches per ring	N_b	-	1	6	288	2808
Bunch spacing	s_b	ns	25	25	25	25
Particles per bunch	N_{pb}	-	1.65.10 ¹²	1.60.10 ¹²	1.32.10 ¹¹	1.15.10 ¹¹
Longitudinal emittance	ε_l	eVs	1.1	1.1	0.35	1
Transverse emittance	ε_t	μ m.rad	2	2.5	2.86	3.5

All these wire-scanners correspond to a large amount of designs, parameters and characteristics (Table. 1.5). Each instrument has its own specifications due to the environment requirements. In addition to the mechanism itself, the wire is the central piece of this instrument. There are as much studies and simulations as specific situations. We introduce here a new one for carbon nanotube material.

1.2.3 Fiber damage studies

During the passage of the beam through the wire, the protons deposit a certain energy inside the wire.

The common model for the beam-wire interaction is described for a complete scan by the energy deposited on average in the wire by an incident proton [40, 47, 73]

$$E_{dep} = \rho d_{eq} \left\langle \frac{dE}{dx} \right\rangle \quad (1.45)$$

so that the total energy deposited by the beam in the wire is

$$E_{dep-tot} = N_p E_{dep} \quad (1.46)$$

with N_p the total number of protons in the beam, ρ the density of the material, $\langle dE/dx \rangle$ the average collision loss, expressed in MeV.g⁻¹.cm⁻². d_{eq} which can be considered as the average thickness of the wire seen by each incident particle

Table 1.5: Use and parameters of wire-scanners over the world.

	Date	Speed [m.s ⁻¹]	Material	Wire diameter [μm]	Signal
Oxford	1964	0.2	Fe (?)	Big (?)	SEM
Heidelberg	1964	1.3	Ta	Big (?)	SEM
AGS	1969	6	Be	10 μm	SEM (?)
DORIS	1973	6	C	7-30 μm	Scintillator
Manitoba	1976	?	?	?	?
CERN	1984	up to 20	C / Be	7-50	SEM+ scintillator
SLC	1988		C / W	4-40 μm	Bremsstrahlung
Tevatron	1989	2-5	C	25 μm	Scintillator
CERN-DESY	1990	1	C / Quartz	7 μm	Shower
HERA	1991	1	C	7 μm	SEM+ scintillator

during a full scan is expressed as

$$d_{eq} = \frac{\pi}{4} \frac{d^2}{v_{scan} t_{rev}} \quad (1.47)$$

where v_{scan} is the speed of the scan and t_{rev} the revolution period of the beam.

The increase of the temperature is then due to the energy deposited by the beam in the wire. The final temperature can be first approximated under adiabatic assumptions by only considering the wire mass heated [40]

$$m_{heat} = \frac{\pi d^2}{4} h_{eff} \rho \quad (1.48)$$

with h_{eff} the effective part of the beam in interaction with the wire and equal to $\sqrt{2\pi}\sigma_{h/v}$ for a Gaussian distribution.

First studies describe the increase of the temperature after a scan as the ratio between the total energy deposited by a proton during the whole scan and the heat capacity of the heated mass [57, 40]. It is expressed as

$$\Delta T = \frac{E_{dep-tot}}{m_{heat} c_p} \quad (1.49)$$

Those early models were progressively updated with the introduction of a factor 3 for a better comparison with experimental results in the SPS [43] and in the LEP [74]. This probably comes from the fact that δ electrons carrying away energy are not considered. The new increase of temperature is then expressed as [47]

$$\Delta T = \frac{N_p}{3\sqrt{2\pi}v_{scan}t_{rev}\sigma_b c_p} \left\langle \frac{dE}{dx} \right\rangle \quad (1.50)$$

By introducing the real Gaussian shape of the beam, the increase of the temperature is given by [75]

$$\Delta T(x, y) = \frac{N_{part}}{c_p} \left\langle \frac{dE}{dx} \right\rangle \frac{1}{2\pi\sigma_h\sigma_v} \exp\left(-\left(\frac{x^2}{\sigma_h^2} + \frac{y^2}{\sigma_v^2}\right)\right) \quad (1.51)$$

The most complete and accurate model was built and coded numerically by M. Sapinski in 2008 [51]. It precisely took in consideration the energy deposited in the wire with Geant 4 simulations and considered all direct mechanisms:

- Radiative cooling
- Conduction
- Thermionic emission
- Sublimation

It is expressed as a power budget and described by the equation

$$\begin{aligned} E_{dep} \frac{\Delta N_{hits}}{\Delta t} &= \rho V c_p(T) \frac{\Delta T}{\Delta t} \\ &\quad - A_{rad} \varepsilon_{th} \sigma_B (T^4 - T_{amb}^4) - k_{th}(T) S \frac{\Delta T}{\Delta y} \\ &\quad - A_{rad} \left(\Phi_w + \frac{2k_B T}{q_e} \right) J_{th}(y) + C(y) \Delta R J_{th-tot}^2 \\ &\quad - H_{sub} \frac{\Delta n}{\Delta t} \end{aligned} \quad (1.52)$$

with E_{dep} the deposited energy by a single proton, obtained with a simulation software such as Geant4 or Fluka, ρ the density of the wire [g.cm⁻³], V the volume of a wire bin where the energy balance is calculated, c_p the specific heat capacity [J.g⁻¹.K⁻¹], A_{rad} the surface of the wire which irradiates [cm²], ε_{th} the thermal emissivity of the wire, σ_B the Stefan-Boltzmann constant [5.67.10⁻¹² J.s⁻¹.cm⁻².K⁻⁴], T_{amb} the temperature of the environment, k_{th} the thermal conductivity [W.cm⁻¹.K⁻¹], S the cross-section of the wire [cm²], k_B the Boltzmann constant [J.K⁻¹], Φ_w the work function per electron for the wire material [V], J_{th} the thermionic current surface density, q_e the elementary charge [C], $C(y)$ a coefficient (between 0 and 1) for current compensation, ΔR the electrical resistance of the wire, J_{th-tot} the total thermionic current emitted from the wire, H_{sub} the enthalpy of sublimation and Δn the amount of material sublimated.

This model allows to have a description step by step during the travel of the wire through the beam. One of the major improvement in this model is the consideration of the thermal properties as a function of the temperature for the heat capacity and the thermal conductivity (The emissivity appears to be weakly dependent on temperature), which is not negligible with the very high temperature reached.

It has been shown that the cooling processes have different contribution with the increase of the temperature. Up to 1300 K, the conductive cooling is the main process to evacuate the heat. The radiative cooling is then dominant with more

than 50 % of the all contribution between 1300 and 3200 K with a maximum (near 100 %) at 2400 K. Above 2400 K, the thermionic emission becomes the main factor.

This model is the only one to consider the loss of matter due to the heating, which is very important to estimate the lifetime of the wires under operating conditions.

The results of these models were corroborated by the observation in 2009 of a significant degradation of the 34 μm carbon wire for low scanning speeds ($\leq 1 \text{ m.s}^{-1}$). For a beam of 400 GeV.c^{-1} , beam sizes around 0.50 mm and a total intensity of $2.4.10^{13}$ protons, the wire sublimated by 95 % and the center diameter of the remaining part was only 7.5 μm (Fig. 1.8).

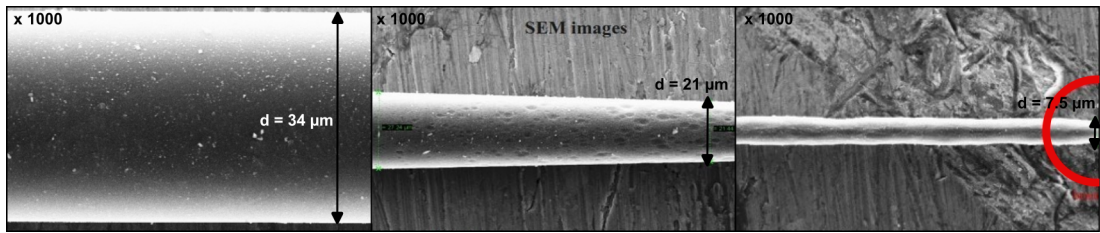


Figure 1.8: Carbon fiber sublimated at three different locations (1 mm from the beam impact, 0.5 mm from the beam impact and at the center) (from Ref [50]).

In another experiment, carbon fibers of 8 μm and 36 μm broke at currents measured in the wire of about 1.5 mA and 4.3 mA respectively, well above previous limits. The average maximum temperature during the scan was measured at about 2020 K [48]. Despite a lower melting temperature of quartz, the wire survived to these same conditions. The measurement of the wire resistance and the observation of the light emission during the scan showed that the main mechanism responsible of the wire destruction in lepton storage rings is by radio-frequency fields. The induced temperature increase is more than twice compared with the estimation made from the energy deposition. This shows that conductive material like carbon and beryllium are more impacted if the forks, the "home" position (geometry) and the tank are not well designed concerning the RF spectrum.

Various other studies have finally shown a weak dependence due to the variation of the wire diameter on the maximum temperature reached [51]. However, thin carbon fiber wires, due to their internal structure graphitized in the last stage of the manufacturing process, have much better mechanical properties than thick wires. In addition, they reduce the number of secondary particles emitted which could quench the magnet located after the instrument [76].

The damage caused by radiation has also been studied for the PS, using Stopping and Range of Ions in Matter (SRIM) software, with the following carbon material parameters: lattice binding energy: 3 eV, surface binding energy: 7.41 eV and displacement energy: 28 eV. It has been shown that a single scan causes $3.51.10^{-7}$ displacement per atom (dpa). The full simulation for a large number of scans

(~ 10000) shows the importance of the wire damage due to recoils already produced by previous recoils, however, the obtained values for structural damages are very small [77].

The diversity of thermal models allows, depending on the needs, to have quick access to conservative preliminary results for pre-studies with the first models or much more precise results with the complex Sapinski model.

1.3 THE CARBON NANOTUBES (CNTs)

1.3.1 General introduction

1.3.1.1 Discovery

Carbon nanotubes, referred to throughout this manuscript as CNTs, are an allotropic form of carbon belonging to the fullerene family. Fullerenes are carbon molecules, taking specific geometric shapes, such as spheres, ellipsoids or tubes, in which carbon atoms are in the so-called sp^3 hybridization state. Fullerenes were discovered in 1985 which granted their discoverers the Nobel Prize in Chemistry in 1996 [78]. The observation of identified as such CNTs is widely attributed to Iijima in 1991[79] (Fig. 1.9.a). However, in a 1952 article not translated from Russian, LV Radushkevich and VM Lukyanovich already show the existence of such structures thanks to the first electronic microscopy images [80]. In 1976, a study on the growth of carbon by decomposition of benzene clearly showed hollow carbon fibers, later considered to be a single-walled CNT [81]. In 1979, Abrahamson presents at the 14th conference dedicated to Carbon in Pennsylvania, proofs of the existence of CNTs, gives an explanation of their manufacturing process, as well as their characteristics. Fourteen years later SWCNTs were produced independently by Iijima with gaseous phase method and Bethune et al., who worked at IBM, using a technique of covaporisation of carbon and cobalt [82, 83].

1.3.1.2 Structure

CNTs exist in two main forms: single-walled CNTs (SWCNTs), *ie* a single sheet of graphene rolled up in a specific direction, and multi-walled CNTs (MWCNTs). The walls are composed of carbon atoms forming hexagons. Each hexagon shares two atoms with an adjacent hexagon in a honeycomb structure. Bonds between carbon atoms are strong covalent bonds. Another important parameter defining a CNT is its chirality. We can define the rolling up direction ($\vec{C}_h$) and therefore the axis of the CNT ($\vec{T}$) (vector orthogonal to $\vec{C}_h$ in the plane of the graphene sheet), thanks to two vectors $\vec{a}_1$ and $\vec{a}_2$ and their integer coordinates m and n (Fig. 1.9.b).

$$\vec{C}_h = m\vec{a}_1 + n\vec{a}_2 \quad (1.53)$$

Depending on the relative angle of the bond directions with $\vec{T}$, three different kinds of nanotubes can be described:

- $m = 0$, the structure of the CNT is called *zigzag*
- $m = n$, the structure is called *armchairs*
- for all other cases, the CNT is *chiral*

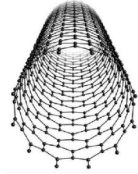
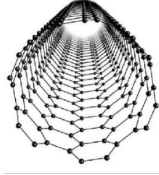
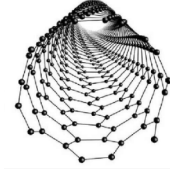
The chiral angle θ_χ is defined as the angle between $\vec{C}_h$ and the direction (n,0), corresponding to the zigzag structure. The chiral angle for the zigzag axis is then $\theta_\chi = 0^\circ$ and for the armchair axis $\theta_\chi = 30^\circ$ which is also non chiral due to the plane symmetries orthogonal to the unrolled graphene sheet. The diameter of the nanotube (d_{CNT}) for a SWCNT can be expressed in geometric terms with the integers parameters n and m as the circumference (length of $\vec{C}_h$) divided by π .

$$d_{CNT} = \frac{a_{C-C}\sqrt{3}\sqrt{n^2 + nm + m^2}}{\pi} \quad (1.54)$$

with a_{C-C} the distance between two atoms of carbon equal to 0.1421 nm in graphite. The chiral angle is given by

$$\theta_\chi = \arctan\left(\frac{m\sqrt{3}}{2n + m}\right) \quad (1.55)$$

Table 1.6: Summary of the CNT characteristic parameters depending on their structure(from Ref [84]).

Symmetry Indices		Chirality angle	Diameter	Example
Zigzag	$(m,0)$	$\theta_\chi = 0^\circ$	$d = a_{c-c} \frac{\sqrt{3}}{\pi}$	 $(12,0)$ $d = 9.4 \text{ \AA}$
	$m = 0$			
	or $n = 0$			
Armchair	(m,n) $m = n$	$\theta_\chi = 30^\circ$	$d = a_{c-c} \frac{3n}{\pi}$	 $(6,6)$ $d = 8.1 \text{ \AA}$
Chiral	(m,n) $m \neq n$	$0^\circ < \theta_\chi < 30^\circ$ $\theta_\chi = \arctan\left(\frac{m\sqrt{3}}{2n+m}\right)$	$d = a_{c-c} \frac{\sqrt{3(n^2 + nm + m^2)}}{\pi}$	 $(6,4)$ $d = 6.8 \text{ \AA}$ $\theta_\chi = 19^\circ$

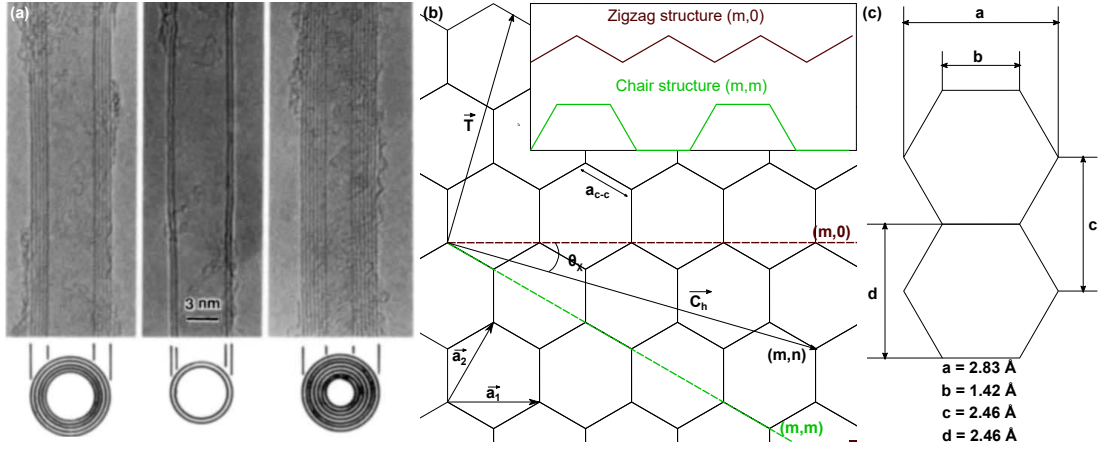


Figure 1.9: (a) Electron micrographs of CNT. Parallel dark lines correspond to the (002) lattice images of graphite. A cross-section of each tubule is illustrated: (1) tube consisting of five graphitic sheets with a diameter of 6.7 nm; (2) DWCNT of 5.5 nm in diameter; (3) MWCNT of 7 walls with a diameter of 6.5 nm (from Ref [79]); (b) Unrolled honeycomb structure of a CNT with the unit vectors ($\vec{a}_1$ and $\vec{a}_2$), the lattice parameters (m and n), the chiral and translational vectors ($\vec{C}_h$ and $\vec{T}$ respectively), the chiral angle (θ_χ) and the distance between two neighbor carbon atoms (a_{c-c}); (c) inter-atomic distances of interest for a graphene sheet.

1.3.1.3 Physical properties

The physical properties of carbon atoms, useful for simulations and characterizations of CNTs are summarized in the table. 1.7.

The mean excitation energy $\bar{I}$, used in the Bethe-Bloch equation, is precisely known for carbon and is 81 eV. The minimum ionization, $\bar{I}$ for a material is defined as being the logarithmic average over the excitation energies weighted by the forces of the corresponding oscillators and is for carbon $1.742 \text{ MeV.cm}^{-2}.\text{g}^{-1}$.

The nuclear interaction length is the mean distance traveled by a hadronic particle before undergoing an inelastic nuclear interaction whereas the nuclear collision length is the mean free path of a particle before undergoing a nuclear reaction. The collision length is smaller than the nuclear interaction length because the latter excludes the elastic and quasi-elastic (diffractive) reactions.

For the rest of the study, the simulations will be performed using these data, except if it is precised.

1.3.1.4 Type of defects

During the manufacturing process or damaging process (mechanical stress, irradiation), different types of defects may occur:

- Atomic lacuna:
 - one-atom vacancy or single-atom vacancy, symmetric reconstruction
 - two-atom vacancy or divacancy, symmetric reconstruction or asymmetric reconstruction

Table 1.7: Physical data of carbon in graphite [85].

	Value	Units
Atomic number	6	
Atomic mass	12.0107	g.mol ⁻¹
Density	2.21	g.cm ⁻³
Mean excitation energy	81	eV
Minimum ionization	1.742	MeV.cm ² .g ⁻¹
Nuclear interaction length	85.8	g.cm ⁻²
Nuclear collision length	59.2	g.cm ⁻²
Radiation length	42.7	g.cm ⁻²
Critical energy	81.74	MeV (for e^-)
Plasma energy $\hbar\omega_p$	30.28	eV
Sublimation temperature (1 atm)	4098	K

- greater than two atom vacancy
- Stone–Wales
 - Pentagon-heptagon (5775) defects
 - Pentagon-hexagon (5665) defects

For the atomic lacuna, one or several atoms of carbon are lacking in the structure, which leads to an incomplete hexagon. In the case of a single atom missing (Fig. 1.10.a), the structure reorganize symmetrically and a hole of 10 atoms is created. For a two atom vacancy defect, the reorganization can either be symmetric (circular hole of 8 atoms) (Fig. 1.10.b) or asymmetric (elliptic hole) (Fig. 1.10.c). For more atoms missing, the structure is deeply disorganized (Fig. 1.10.d). The Stone-Wales defect is a crystallographic defect which involves a change in the relationship of two carbon atoms linked by π bonds and which leads to a 90° rotation of the bond between these two atoms [86]. The reorganizations can be of type (5775) (Fig. 1.10.e) with the creation of two heptagons and two pentagons or of type (5665) (Fig. 1.10.f) with the creation of two pentagons connected to two hexagons [87].

The excellent mechanical characteristics *cf.* (section 1.3.3) of the CNTs come from the honeycomb structure of the graphene. This structure is optimized to get the maximum energy in each of bonds. The original defects obtained during the manufacturing process are the entry points to weak areas. A CNT subject to a mechanical stress will fail in these areas.

1.3.1.5 Records

In 2007, scientists from the university of Cincinnati in US announced the synthesis of 2 cm long CNTs. It corresponds to a ratio with the diameter of 900 000. They used the method of chemical vapor deposition (CVD) of thin layers of materials in a furnace called *EasyTube 3000*.

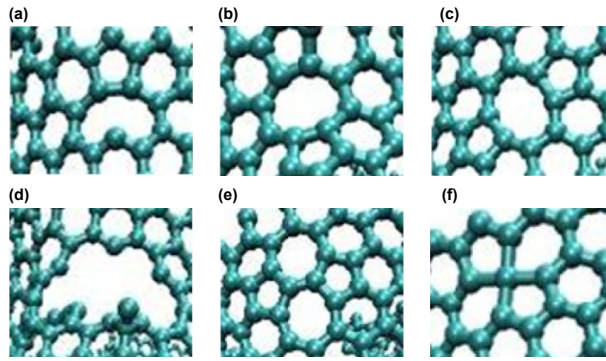


Figure 1.10: Defects which can appear during the manufacturing process: (a) One atom vacancy (symmetric reconstruction); (b) Two atom vacancy (symmetric reconstruction); (c) Two atom vacancy (asymmetric reconstruction); (d) Greater than two atom vacancy; (e) Stone–Wales (5775) defects; (f) Pentagon–hexagon (5665) defects (from Ref [87]).

In 2013, researcher from the Tsinghua University in China claimed to have produced CNTs of 1 to 3 walls and 55 cm in length and reported a mechanical strength of 120 GPa. The method used was also CVD [88].

However, these results, from an industrial point of view, are not yet convincing due to the low yield during production ($\sim 10\text{--}100\text{ CNTs.mm}^{-1}$). This could soon change thanks to a combination of several manufacturing process showing results of the order of $10\,000\text{ CNTs.mm}^{-1}$ [89].

1.3.2 Manufacturing process

The work for new methods of synthesis of individual carbon nanotubes (CNTs) has considerably developed with the discovery of the extraordinary good properties of the CNTs. The current and future possible applications are numerous and varied. To answer the needs worldwide in terms of quantities and costs, different production methods emerged with advantages and limitations for each of them. The most used are the Chemical Vapor Deposition (CVD), the Laser Ablation (LA), the Arc Discharge (AD) and the High-Pressure Carbon Monoxide Disproportionation (HiPco).

Since in all the wires we got (from HitachiZosen, Japan, IMDEA, Spain and Dex-Mat, US), the CNTs are obtained by CVD, this method will be fully detailed. The other methods will be succinctly described.

1.3.2.1 Chemical Vapor Deposition

Individual CNTs can be obtained in a vertically aligned forest, which is one of the most used method. It allows a large panel of possibilities for further applications. The CVD method for Chemical Vapor Deposition consists in growing up carbon structure on a wafer. The preparation of the wafers is a critical step and needs high material skills. A thick layer of a hundred nano-meter is photolithographically patterned with a thin film, between 1 and 10 nm thick, of a catalyst

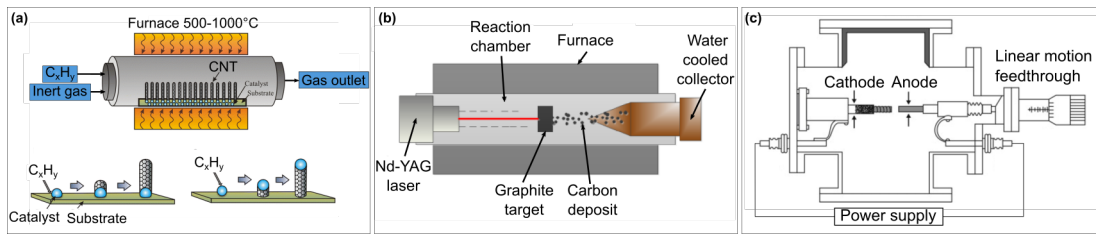


Figure 1.11: Schematic representation of CNT manufacturing processes: (a) Chemical vapor deposition (CVD) with simplified scheme of a CVD reactor for CNTs synthesis, base-growth model of CNT growth mechanism and tip-growth model of CNT growth mechanism (from Ref [90]); (b) Laser ablation experimental setup (from Ref [91]); (c) arc discharge experimental setup (from Ref [92]).

material. This pattern method allow to focus the CNT growth in specific region by forcing the carbon structure in a specific organization.

The production of vertical CNT forests is possible with a large range of initial conditions, but the final target which is getting an assembly of these individual CNT into a microscopic wire implies specific conditions. That is particularly true for the solid-state processing.

1.3.2.1.1 Mechanism

The nucleation of the MWCNTs takes place via a catalytic dehydrogenation of the molecules of the initial hydrocarbon gas by the preformed catalytic particles. The gas vapor strikes the atomic structures on the surface of the substrate which breaks the carbon-hydrogen bonds. The free carbon atoms diffuse on the surface or through the mass, on the open structures and participate in the growth of the CNT. It would seem that during the formation of the MWCNTs, all the walls grow at the same time. This is explained in particular by the surface energies involved, which are particularly high for nanometric particles. A metallic nanoparticle contains a large percentage of atoms on the surface, which creates too much surface energy. To overcome this problem, a surplus of carbon can be injected. The carbon atoms assemble into a sheet of graphene and encompass the metal particles. Since graphite planes have a much lower surface energy (10 to 20 times lower [93] than for metals [94]), the total surface energy decreases. The nucleation of SWNCT and MWCNT from this hemispherical layer is called the *yarmulke* mechanism [95]. A crucial aspect of this mechanism is the minimization at all times of the CNT growth of openings in the graphene sheets. The new carbon atoms continue to assemble on the surface of the catalyst. These additional atoms can be positioned at three distinct positions:

- 1- on the original surface envelope, which, in the case of a total coating of the catalytic particle, is deactivated
- 2- under the primary surface envelope to form a second hemisphere, spaced by 0.34 nm, the distance between the sheets of graphite. If additional hemispheres form below, the old ones rise up and form a cylindrical tube, the

end of which remains attached to the catalytic particle. The creation of additional internal layers stops when the constraint of the curvature of the new layers becomes too strong. These energy considerations explain why the inner walls of MWCNTs are rarely smaller than 3 nm [96]

3 to add to the surface of a growing layer (Fig. 1.11.a)

This mechanism highlights the bonds between the diameter of the CNT and the size of the catalyst particle. Various parameters play a keyrole in the building of the CNT. The final structure of the product depends on:

- The substrate
- The growth time
- The temperature
- The pressure
- The carbon source
- The precursor gas
- The gas flow velocity

Currently, there are two widely accepted mechanisms [97], and they can be summarized as follows:

- 1- When a hydrocarbon vapor is contacted with heated metal nanoparticles, it is first decomposed into carbon and hydrogen. Hydrogen leaves with the passing carrier gas or reducing gas, and carbon dissolves in the metal catalyst.
- 2- When the temperature reaches the carbon solubility limit of the metal, the decomposed carbon particles precipitate and crystallize to form CNTs. The decomposition of hydrocarbons is an exothermic process, carbon crystallization is an endothermic process, and the thermal gradient continues this process. The reason for the two growth mechanisms is that the interaction between the catalyst and the substrate is different:
 - 1- When the catalyst interacts weakly with the substrate, carbon decomposed from the hydrocarbon diffuses from the metal catalyst to the bottom of the metal catalyst and precipitates between the substrate and the metal catalyst, thereby promoting the rise of the entire metal catalyst nanoparticles. When the metal particle is entirely covered by excess carbon, growth stops, which is called tip-growth.
 - 2- When the catalyst interacts strongly with the substrate, the carbon precipitates without pushing up the metal particles, so it is forced to precipitate from the top of the metal, which is called the "basic growth model", also called root growth.

1.3.2.1.2 Substrate

The substrate is prepared with a layer, basically silicon [98] or alumina [99] and coated with metal catalyst particles, most commonly nickel, cobalt [100] iron [101], molybdenum [99] or a combination [102]. It exists an optimal thickness for

the catalyst to get the longest array which can vary from 2 to 20 nm [99, 101]. Considering the *yarmulke* mechanism, the nucleation and the growth of the CNT occur if the size of the metal-catalyst particle does not exceed a maximum diameter and height, to get a sufficient large curvature. A too large catalyst particle leads to a basic carbon overcoating of the substrate hence an optimal thickness of the substrate [103, 104, 105].

The pattern of the substrate allow to control the length of the tubes. The CNTs have a larger growth when the spacing between each tube is small, ie a tiny pattern [101]. This is probably due to the production of an initial gas reaction which occurs in the region of high catalyst density. The ethylene molecules serving as the basis for the formation of solid carbon, the CNTs, by chemical reaction with the catalyst also give highly volatile molecular by-products. In a process such as CVD with flow of a carbonaceous gas through a reactor, the steady state gas phase concentration is higher in areas of high iron catalyst density. The presence of these by-products in the medium increases the growth rate of the tubes by reacting more strongly with the iron catalyst than the original gas.

1.3.2.1.3 Growth time

The length of the CNT at the end of the growth depends on the duration of three steps: a fast linear increase, a decrease of the growth rate to finally arriving at 0 [101]. The partial or complete interruption of the CNT growth using the thermal CVD method with metal-catalysts substrate have been shown [99, 106, 107]. One possibility is that the metal particles from which CNT nucleate remain catalytically active for only a short time after the carbon-containing feedstock is introduced. Possible mechanisms for the inactivation of these metal particles include overcoating with carbon or conversion of the metal into a metal carbide or other non-catalytic form (e.g., by absorption of too much carbon into the interior of the particle).

1.3.2.1.4 Temperature

The iron atoms composing the substrate need an energy, and therefore a temperature, high enough to be sufficiently mobile and allow the aggregation, nucleation and growth of the CNTs. A too low temperature slows down the catalytic decomposition of the initial gas and therefore prevents the formation of CNTs. However, if the temperature is too high, the iron atoms have too much mobility and the particles become too large for nucleation [103, 104, 105, 107, 108, 109, 110, 111]. During the process, the CNTs show a maximal length and optimal growth rate for a specific temperature corresponding to specific initial conditions [112, 113, 114, 115, 116, 117, 118]. It was observed for a growth time of 15 min, a Fe catalyst thickness of 2.5 nm, C₂H₄ and H₂ flows of 380 and 190 sccm (standard cubic centimeters per minute) respectively and a pressure of 0.3 bar, a linear increase of the CNT length becoming significant from 870 K and maximum between 970 and 1020 K. At the maximum temperature of 1070 K, the growth is very small except for the highest density bundles.

1.3.2.1.5 Pressure

The pressure inside the enclosure plays a major role. The concentration of the molecules of the initial gas limits the growth rate of the CNTs as well as their maximum length. Indeed, at low pressure, the gas molecules are less available to react with the catalyst. At high pressure, other mechanisms come into play and limit growth. The length of the CNTs increases linearly with an increase in pressure between 0 and 0.1 bar. The growth rate increases up to 0.2 bar. Beyond 0.2 bar, the growth rate is almost nil, except for very dense networks [101].

1.3.2.1.6 Carbon source

The carbon source, *ie*: the precursor, can be in solid, liquid or gas state. In the case of a liquid source, it just needs to be heated to transform as a steam and inserted in the reactor with the carrier gas. If the source is solid, the hydrocarbon can be stored directly in the quartz tube with the catalyst and placed in the reactor tube. The acetylene (C_2H_2) [98] and ethylene (C_2H_4) [101, 119] are commonly used as precursor gas because models and experiments were well studied. Carbon atoms get free after different time scale corresponding to different steps of the decomposition. The analysis of gas exiting out of the tube gives high molecular weight species (1,3-butadiene, cyclopentene, 3-penten-1-yne, 1-pentene, 1,4-pentadiene, 2-hexane...). This confirms molecular growth by radical recombination [119].

1.3.2.1.7 Gas flow velocity

Among the large number of parameters manageable during the production of CNT, there is the flow velocity of the initial and inert gas. The CNT length increases up to a maximum, depending on other initial parameters, and decreases with the increase in flow velocity [101]. As the CNT length depends on the catalyst particle parameters, this highlights the importance of some intermediate molecular species for the CNT growth. The variation of the flow velocity affects the local concentration of these specific intermediate molecules during the growth of the tubes. The increase in flow velocity implies a lower concentration of these necessary molecules which leads to a lower growth of CNT. On the other hand, a zero flow velocity allows to the initial gas to polymerize and therefore to give solid carbon or heavy carbonaceous tar and molecular hydrogen.

1.3.2.1.8 Advantages and limitations

CVD can be performed at low temperatures, which means that it is energy efficient and cost-effective. This parameter is also a key point for the mass-production, with a yield which can reach 60 to 90 %. Moreover, this method can produce CNTs with extremely high purity, which is important for many applications. Moreover, the manufacturer has a greater control over the CNTs' structure and properties. This allows for the creation of MWCNTs with specific properties, such as specific lengths, diameters, and chirality. This method induces little impurities compared

to other processes which requires further purification. Finally, CVD-produced CNTs can be used in a variety of applications, including electronics and energy storage.

The production of MWCNT faces technical difficulty which is the impossibility to operate with temperature higher than 1080-1200 K. Indeed, above these temperatures, the rate of pyrolysis of the initial hydrocarbon gas overtakes the rate of CNT growth. The result is an unwanted carbon amorphous overcoating [95]. Another issue is the production of CNTs with higher relative defectiveness. The process is complex to control and produces toxic and corrosive gases.

1.3.2.2 Laser Ablation

High power laser vaporization (YAG type) is a method for synthesizing CNTs which involves heating a quartz tube containing a block of pure graphite inside a furnace to 1200 °C in an Ar atmosphere. To generate SWNTs, the addition of metal particles as catalysts to the graphite targets is necessary. The laser pulse power affects the diameter of the nanotubes, with higher pulse power leading to thinner nanotubes. Sub-picosecond laser pulses are capable of creating large amounts of SWNTs, with up to 1.5 g.h⁻¹ of nanotube material potentially produced. Numerous parameters, such as the structural and chemical composition of the target material, the properties of the laser, buffer gas flow and pressure, and pressure gradient, can affect the properties of the CNTs. This method has the potential to produce high purity and high quality SWNTs. The principles and mechanisms of the laser ablation technique are similar to the arc discharge technique, except the energy is provided by a laser striking a pellet of pure graphite containing catalytic materials (frequently cobalt or nickel) (Fig. 1.11.b).

The laser used in this method adds radiation energy to the synthesis reaction which results in very high quality products. In addition, it is possible to produce them in large quantities.

Unfortunately, the laser ablation method is not economically advantageous because it requires a high-energy consumption and the procedure encompasses high purity graphite rods, the required laser powers are large (in some cases two laser beams are required) and the amount of nanotubes that can be synthesized per day is not as high as arc discharge technique. Moreover, impurities adheres to the tubes which requires further purification.

1.3.2.3 Arc discharge

The arc discharge technique is a method for synthesizing CNTs in which two different approaches are used: synthesis with catalyst precursors and without catalyst precursors. Commonly, MWNTs can be synthesized without catalyst precursors, while SWNTs require the use of different catalyst precursors, such as Gd, Co, Ni, Fe, Ag, Pt, Pd, etc., or mixtures of Co, Ni and Fe with other elements like Co-Pt, Co-Ru, Ni-O, etc. Ni-Y-graphite mixtures have been reported to yield high amounts of SWNTs with an average diameter of 1.4 nm. This method has

the capacity to produce large amounts of nanotubes, but has limited control over their alignment and chirality. In addition, the products require purification due to the metal catalyst used. This technique uses temperatures above 2000 K, which allows for less structural defects compared to other methods. It is done by forming an arc between two high purity graphite electrodes of 6-12 mm in diameter, separated by 1-2 mm, within a helium chamber at 0.7 bar. The cathode deposits half of the evaporated carbon which forms a "cylindrical hard deposit" or "shaped structure cigar". The anode is consumed, while carbon condenses into "chamber soot" near the walls and "cathode soot" on the cathode, which produces single-walled or multi-walled carbon nanotubes and interlocking polyhedral graphene particles. Scanning electron microscopy shows two different textures and morphologies in the cathode deposition: a dark, soft inner core with randomly arranged nanotubes and a gray outer shell with layers of curved, solid graphenes (Fig. 1.11.c).

The process of obtaining CNT by arc discharge makes it possible to obtain products of much better quality than by thermal CVD methods. The main advantage of this technique is both a high yield and a small quantity of residual metallic catalytic particles which evaporate at the end of the tube once closed.

The main disadvantage of this technique from a structural point of view is that the CNTs obtained are not necessarily uniformly straight and may contain ramifications. In addition, if there are only a few metallic particles, there is a presence of soot and amorphous carbon which must be got rid of. The CNTs must therefore be purified in post-processing. From a production point of view, this method can only supply a small quantity of materials.

1.3.3 Intrinsic properties

CNTs are an extremely promising material because of their intrinsic properties. In particular, CNTs present remarkable density and strength. The intrinsic properties depend on the structure of the CNT (the number of walls, the length and sometimes the chirality or diameter). In the following section, we will report ranges of observed values.

1.3.3.1 Density

One of the most important and interesting parameter for applications is the density. The density of a ideal perfect CNT can be calculated geometrically [120]. To make the calculation, four simplifying hypotheses can be made: 1- the double carbon-carbon bond is the same in the curved graphene sheet than in the plan, *ie*: 0.1421 nm (Fig. 1.9.c). 2- MWCNTs are composed of concentric walls with a regular space of 0.3400 nm. 3- the contribution of the electron density is neglected. 4- the aspect ratio (length over diameter) is high enough (>1000) to neglect the area of the "tip" surfaces in comparison to the cylindrical surfaces. It has been shown that the specific surface area of a SWCNT, expressed in $\text{m}^2 \cdot \text{g}^{-1}$, is independent of the diameter and is the same than a graphene sheet ($S_{s-SW} = 1315 \text{ m}^2 \cdot \text{g}^{-1}$) [121]. The specific mass is equal to $1/S_{s-SW}$ and is $m_{s-SW} = 7.602 \cdot 10^{-4}$

g.m^{-2} . The mass, in g, of a SWCNT can then be calculated depending on its diameter by

$$m_{SW} = \frac{\pi l_{CNT} d_{SW}}{1315} \quad (1.56)$$

with l_{CNT} and d_{SW} the length and the diameter of the SWCNT respectively.

The volume of the CNT is expressed as $V_{SW} = \pi l_{CNT} d_{SW}^2 / 4$ and the density is then

$$\rho_{SW} = \frac{4}{1315 d_{SW}} \quad (1.57)$$

For a MWCNT, the external surface is $S_{ext} = \pi l_{CNT} d_{CNT}$. The specific surface area of the MWCNT is defined as

$$S_{s-MW} = \frac{S_{ext-MW}}{m_{MW}} \quad (1.58)$$

$$S_{s-MW} = \frac{S_{s-SW} d_{CNT}}{n_w (d_{CNT} - d_{iw} (n_w - 1))} \quad (1.59)$$

The total surface of the MWCNT is the sum of the successive surfaces of the walls. It is expressed as a function of the external diameter of the CNT and the number of walls by

$$S_{tot-MW} = \pi l \left(n_w d_{CNT} - 2 d_{iw} \sum_{i=1}^{n_w-1} i \right) \quad (1.60)$$

with n_w the number of walls, d_{CNT} the external diameter and d_{iw} the inter-wall distance.

The mass (g) of the MWCNT is then expressed as the specific mass (g.m^{-2}) of a SWCNT time the total surface of the walls (m^2).

$$m_{MW} = m_{s-SW} S_{tot-MW} \quad (1.61)$$

Finally, using the volume of the CNT equal to $\pi l_{CNT} d_{CNT}^2 / 4$, the density of the MWCNT is expressed as

$$\rho_{MW} = \frac{4 \left(n_w d_{CNT} - 2 d_{iw} \sum_{i=1}^{n_w-1} i \right)}{1315 d_{CNT}^2} \quad (1.62)$$

$$\rho_{MW} = \frac{4 n_w}{1315 d_{CNT}} \left(1 - \frac{d_{iw}}{d_{CNT}} (n_w - 1) \right) \quad (1.63)$$

Using this model, we obtain the specific surface area and the density as functions of the external diameter and the number of walls. The specific surface area of a CNT is constant with the increase of the diameter and decrease for $n_w \geq 2$. It

decreases sharply for low diameters and varies in to $1/n_w$ for very high diameters (Fig. 1.12.a). The second plot is very interesting to find the optimal parameters in the research of a specific density. Depending on the couple diameter - number of walls, the density can vary from 6 to theoretically 0 g.cm^{-3} if the diameter tends to infinity (Fig. 1.12.b). In practice, SWCNT aerogels may reach an ultralow density of 0.007 g.cm^{-3} [122].

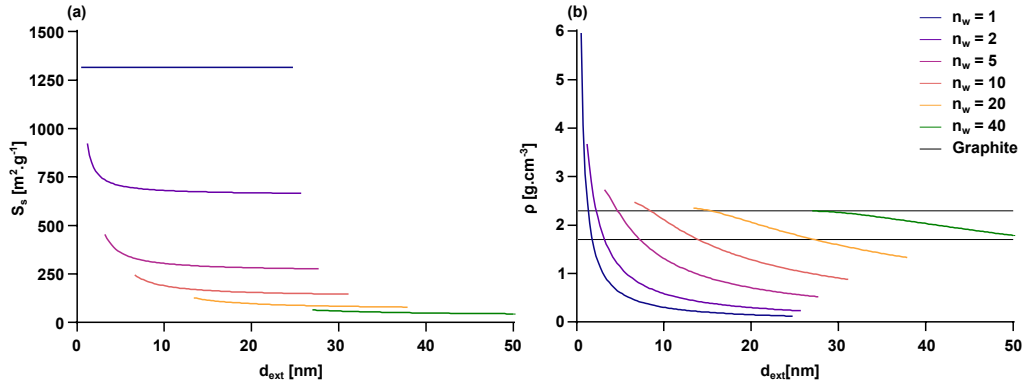


Figure 1.12: Theoretical specific surface area and density of CNTs: (a) Specific surface area of CNTs based on the calculation of the equation. (1.59); (b) Density of CNTs based on the calculation of the equation. (1.63). For both plots, the specific surface area and the density depends on the external diameter d_{ext} and the number of walls n_w . SWCNT are represented in blue, DWCNT in dark purple, MWCNTs in light purple, coral, orange and green. Graphite is plotted in black for extrema limits (1.7 g.cm^{-3} and 2.3 g.cm^{-3}) (from Ref [120, 121]).

1.3.3.2 Mechanical properties

1.3.3.2.1 Young's modulus

The C-C bond of graphite is a strong chemical bond. Indeed the dissociation energy of this bond is 348 kJ.mol^{-1} . This is the reason why fibers (and in particular nanotubes) have unique mechanical properties. Since their discovery, many theoretical works have predicted very high Young's moduli for carbon nanotubes. The use of empirical model predicts a Young's modulus of 0.97 and 1.1 TPa for multi-walled and single-walled nanotubes respectively [123, 124, 125]. It also shows that the Young's modulus, as well as other elastic moduli (shear, ...) are independent of the geometric characteristics of the tubes (chirality, diameter...). By comparison, cobwebs, which are reputed to have high tensile strength, have a Young's modulus comparable to that of Kevlar (4 GPa) [126]. CNT can be subjected to significant stresses (longitudinal or transverse) without showing any sign of plastic deformation, brittle fracture or atomic rearrangement [127]. They also show that classical theories using a continuous model can be used for large deformations of nanotubes (for which linear elasticity is no longer valid). With a free end, the nanotubes vibrate under the effect of Brownian motion. By studying their vibration by TEM (Transmission Electron Microscopy), it is possible to trace their Young's modulus. Using this method, a value of $1.25 \pm 0.5 \text{ TPa}$ for SWCNT was obtained [128]. By

AFM, it has been estimated that the Young's modulus of strings of single-walled nanotubes at 1.04 ± 0.17 TPa [129]. They also show that it is necessary to exert a constraint of the order of 0.40 TPa to break the rope. By measuring 3 points in AFM, the same stress was estimated at 0.45 ± 0.07 TPa. These values are about 20 times higher than those of the stiffest steels. The assumption that a CNT is a structural member allows the elastic properties to be obtained by experimental observation. Different types of models were considered such as bar, beam and shell models. The bar model was used in an experiment in which the compression response was measured using Raman microscopy [130]. Young's moduli of 2.8 to 3.6 TPa for SWCNTs and 1.7 to 2.4 TPa for MWCNTs have been reported. Direct measurements made by tensile tests gave Young's moduli between 320 and 1470 GPa, with an average of 1002 GPa for SWCNTs [129] and between 250 and 950 GPa for MWCNTs [131].

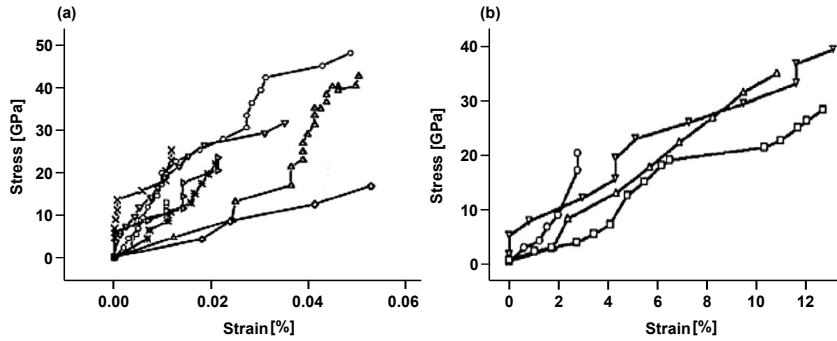


Figure 1.13: (a) Stress versus strain curves obtained from the tensile-loading experiments on individual SWCNT (from Ref [129]); (b) Plot of stress versus strain curves for individual MWCNTs (from Ref [131]).

1.3.3.2.2 Strength

The mechanical strength of CNTs is mainly driven by the distribution and type of defects in the structure. The nature of these defects is closely linked to the manufacturing process. Measuring the tensile strength of nanotubes is not easy. Direct method tests have been performed on SWCNT bundles and values of 13 to 52 GPa for tensile strength have been reported [129]. The maximum strain at break was measured at 5.3 %, a value close to theoretical predictions [132]. Tests on MWCNTs have shown that only the outermost wall breaks during traction. The tensile strength corresponding to this wall varies between 11 and 63 GPa [131]. Estimates of this characteristic can also be obtained by indirect methods, in particular by the use of AFM (Atomic Force Microscopy). Values of 45 ± 7 GPa have been obtained for SWCNTs assuming a Young's modulus of 1.25 TPa [133]. Tests performed on long MWCNTs aligned by CVD showed much weaker results with "only" 1.72 ± 0.64 GPa [134].

In order to overcome the technical difficulties of pulling a nanoscopic sample, indirect methods have also been used. Among these approaches, CNT charge transfer is through a matrix material, showing values from 22 to 55 GPa for SWCNTs

[135, 136].

Although experimentation is the best way to obtain raw data, it is now easier to model and calculate the effects of different factors such as the structure and nature of defects, the loading rate and the temperature. The limit to this is that the results obtained depend strongly on the model. Molecular dynamics (MD) simulations gave maximum breaking strengths of 150 GPa for a strain to failure of 30 % [127, 137, 138, 139].

The currently synthesized CNTs are not individually synthesized. The end product is usually a random clump of MWCNTs or bundles of SWCNTs [95, 140]. It is necessary to understand that the very large mechanical properties of individual CNTs do not necessarily imply these same properties in a micro or macroscopic assembly.

1.3.3.3 Thermal properties

Measuring the thermal properties of a nano-object like the nanotube is tricky. Diamond has the highest thermal conductivity of any 3D material known to date (about 1000 - 2500 W.m⁻¹.K⁻¹). The graphene plane has a higher conductivity (4000 - 5000 W.m⁻¹.K⁻¹). The particular structure of the nanotubes makes it possible to think that their conductivity must also be important along the axis of the nanotube.

Simulations showed a dependence of the thermal conductivity with the temperature, with a peak at 37 kW.m⁻¹.K⁻¹ for a temperature of 100 K. For ambient temperature, the thermal conductivity is around 6600 kW.m⁻¹.K⁻¹ [141]. Moreover, studies showed the dependence of the thermal conductivity for a SWCNT with the length and the diameter of the tube as well as the thermal conductivity of a graphene sheet [142]. It increases with the increase of the length and with the increase of the diameter. The defect concentration and vacancy concentration in the structure of the CNT has also been studied showing a decrease of the thermal conductivity with the increase of the defects and vacancy concentration [143].

Experimentally, the thermal conductivity can be measured by Joule self-heating. At room temperature and for a SWCNT of 1.7 nm in diameter and 2.6 µm long, the thermal conductivity has been measured at 3500 W.m⁻¹.K⁻¹ [144]. To remove the error from the influence of the thermal contact resistance, the Raman shift method is a good solution and showed values of 2400 W.m⁻¹.K⁻¹ and 1400 W.m⁻¹.K⁻¹ for individual SWCNT and MWCNT respectively [145].

These results are a priori disappointing, but they can be explained by considering the fact that all the sheets participated in the thermal conduction while only the outer sheet was used for the measurements. These considerations make it possible to estimate the magnitude of the conduction of a single-walled tube to the same order of magnitude as that of graphite.

As for the mechanical properties, the thermal properties of the individual CNTs cannot be directly transposed to the properties of the assemblies of CNTs.

1.4 CARBON NANOTUBE WIRES

We have just briefly described what carbon nanotubes are, their structure, their method of synthesis and the two main intrinsic properties that interest us: mechanical resistance to rupture and thermal conductivity. This state of the art is not exhaustive but it allows us to have references in our field of interest, the assembly of these nanoscopic tubes in a microscopic structure.

The description of these microscopic assemblies in cylindrical structure is done by the use of several terms: fibres, yarn, rope. As our study relates to the use of these assemblies for the wire-scanners, I will use for the remaining part of this thesis the term of CNT wire.

1.4.1 Production

The manufacturing process for the production of CNTW are various [146]. Each growth present specific advantages and weaknesses. The CNTs are the base of several micro/macro material such as wires [147, 148], sheets [149, 150] and films [151, 152, 153]. Two spinning families exist: the wet-spinning and the solid-state-spinning. The wet-spinning presents different techniques but the base is the same. For the solid-state-spinning, the difference in the CNT supply implies different methods.

1.4.1.1 Wet spinning

The wet-spinning, corresponding to a liquid state processing, has been developed on the bases of what is done for synthetic fibers [154]. However, it has been very challenging due to the hard work of processing CNTs in a liquid state [155]. The wet-spinning method requires a CNT pre-processed as a powder. CNTs can be produced in-house with a separate process such as CVD [156] or purchased. This powder is then mixed either with a surfactant-based mixture (Maijo Nano Carbon Co., Ltd) or in a super-acid bath (DexMat). This mixture forms the CNT dispersion [157].

The dispersion and coagulation of the wet spinning process can be performed by four main techniques:

- surfactant dispersion coagulated in water-Polyvinyl acetate (PVA) [155, 158, 159, 160]
- surfactant dispersion coagulated in ethanol/glycerol [161]
- surfactant dispersion coagulated in acid or base [162]
- sulfuric dispersion coagulated in water [163]

The surfactant is used to stabilized the CNT dispersion by forming micellar structures around the tubes. For SWCNT, the only known solvents which can be used are the super-acids [164, 165]. The assembly CNT/micelle is stable and the specificity of this structure prevent an aggregation in bundles. The parameters to

get the optimum surfactant concentration depends on the type of CNT (SWCNT, DWCNT, MWCNT [151, 162, 166, 167]) but also the diameter and the length of the CNTs.

The dispersion is then continuously injected in an alcohol bath through a tube with a diameter equal to the diameter of the desired final wire. It causes the clumping and coagulation of the individual CNTs into microscopic fibers (Fig. 1.14.a). One or even several post-processing steps are necessary to eliminate impurities. The surfactant is eliminated after immersion in a water bath for several hours. Depending on how the liquid is removed, the wire may show an hollow structure [157]. This kind of wires shows interesting properties for very specific applications, but they have weak mechanical properties.

This manufacturing method without post processing, does not allow to have a uniform fiber structure. Depending on the input parameters (type of surfactant, sonication time and others), the threads show very different profiles: ribbon-like shape [168] , structure with a void in the center, density from 0.3 to 0.9 g.cm⁻³ [157].

The use of acid during and after the production has yielded the most highly ordered and dense CNT fiber [169]. The molecules composing the acid may surround the individual nanotubes by creating a thin layer. It forms an energetically favorable charge-transfer complex and increase the mechanical properties [163].

The fact that CNTs have a high stiffness prevent their solubility in organic or aqueous solvent. The tubes are forming bundles instead of dissolving because of the van der Waals forces between them. In order to overcome this problem, techniques have been developed consisting to mix CNTs with other elements making them soluble in common solvents [170, 171]

For fibers obtained by super-acid dissolution of SWCNT, the alignment of the tubes is determined by the concentration of the acid. Three regimes were observed. For low concentrations, CNTs are randomly oriented in the acid. At medium concentration, there is a biphasic balance between coexisting isotropic and liquid-crystalline phase. At the highest concentrations, the solution is fully liquid-crystalline [172].

Wires produced with this methods show a high degree of alignment and purity and a relatively dense packing of the CNT. The different parameters of the CNT (type, diameter, aspect ratio) can be correctly controlled due to the high purity of the provided CNTs to be spun [173]. However, that high purity has a cost limits the production of large volume of material.

1.4.1.2 Solid-state or dry spinning

The solid-state-spinning allows two types of CNT production : from the gas-phase CVD [174, 175] linked with the cotton cloud [176, 177] and from a vertically-grown CNT array [178, 179, 180, 181, 182].

Obtaining CNT wires from the solid-state spinning method, or dry spinning, is inspired by traditional methods for obtaining cotton or wool wires. The wire is made up of a set of fibers with specific characteristics (diameter, length). A CNT wire obtained by dry-spinning consists of fibers of CNTs, generally produced by gas-phase CVD.

The first technique to produce CNT wires is the Floating Catalysis Chemical Vapor Deposition (FCCVD) also referred as a direct-spinning method. It provides wires by drawing continuously on a CNT aerogel directly from the gas phase inside the oven. To do so, a liquid or gaseous feedstock containing the carbon source (toluene [183], butanol [183]), the catalysis particles (ferrocene [184, 185]) and the precursor (sulfur [183, 184]) are injected in an oven at high temperature (1200 K). All molecules decompose and CNTs are formed. The final wire is obtained by hooking the aerogel directly in the oven and prime it to a windle. The winding speed is a critical parameter. The faster the winding, the greater the alignment of the CNTs and the greater the density. The maximum speed is limited by the breaking strength of the aerogel.

Another possibility to get a dry-spinning wire is to use a CNT array vertically grown on a flat substrate (Fig. 1.14.c1,c2) [186]. They are drawn from the edge of the array and each tubes are then held together by the van der Waals forces. CNT's vertical forests feature disordered regions at the bottom and top of the array. These messy areas play a key role in preserving the integrity of the wire by interconnecting the tubes [147]. The produced wire is similar to a CNT network [187]. At the end of the growth of CNT arrays, it appears a thin "crust" at the top surface. While the CNTs in the array are well aligned, the ones on the surface are entangled together. The thickness of the layer is about 1 μm for a film of 200-600 μm . The CNT not located on the top surface form thick vertical bundles. From these bundles goes out sub-bundles that can create bonds with other sub-bundles by van der Waals. The CNT bundle has the same structure in the middle and at the bottom of the film and show a very good alignment of the arrays, except at the top surface. During a pulling process, two kinds of junctions appear between bundles. A first one where the bundle ends are held together by van der Waals forces. This junction is quite fragile and easily detached during the pulling. The second one, more robust, is formed by an entanglement of CNT bundles. These junctions allow to form a continuous wire and are only located in the region near the top and bottom parts of the film.

On the other hand, studies have shown that highly aligned CNTs without amorphous carbon are a critical condition for the success of dry spinning [188, 180]. For CNTs from carbon sources containing oxygen, the wires are successfully obtained by dry spinning, while for aromatic hydrocarbon sources, the CNTs are not easily weaved. This is explained by the strong presence of amorphous carbon in the aromatic sources.

The intrinsic properties of CNTs are also key factors for obtaining a good quality wire. The diameter, the number of walls and the length of the CNTs come

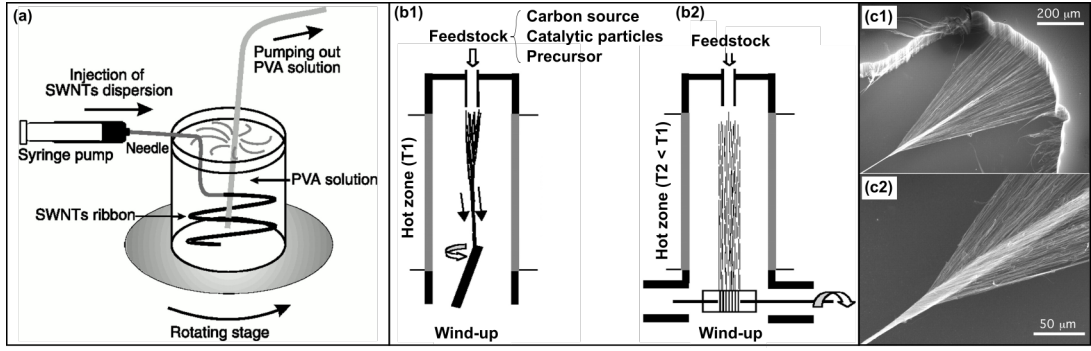


Figure 1.14: (a) Schematic of the experimental setup used to make nanotube ribbons from wet-spinning process [155]; (b1) Dry-spinning process. Schematic of the direct spinning process from CVD decomposition. The liquid feedstock is mixed with hydrogen and injected into the hot zone, where an aerogel of nanotubes forms. This aerogel is captured and wound out of the hot zone continuously as a fiber or film; (b2) Schematic of the wind-up assembly that operates at a lower temperature, outside of the furnace hot zone (from Ref [174]); (c1) and (c2) SEM images of a carbon nanotube wire in the process of being simultaneously drawn and twisted during spinning from a nanotube forest. A forest strip (width $\sim 600 \mu\text{m}$) formed the pictured twisted wire, $3.2 \mu\text{m}$ in diameter. The MWCNTs, $\sim 10 \text{ nm}$ in diameter with length 10000 times their width, form small bundles of a few nanotubes each in the forest, with individual nanotubes moving in and out of different bundles (from Ref [147]).

into account. Large-diameter CNTs with one or two walls at most are particularly interesting due to their ability to flatten, which increases the contact surface between adjacent CNTs and therefore improves charge transfer at the molecular scale. Long lengths of tubes make it possible to increase the surfaces and therefore the charge transfers and reduce the defect densities in the fibers (Fig. 1.17). However, for CNTs in a vertically aligned array, obtaining long CNTs implies an increase in amorphous carbon, which limits the maximum spinnable length.

We have just explained two of the three methods for obtaining CNT wires. The last, the direct spinning of CNT fibers from aerogels, is the one used to produce the samples tested in our experiment. This method will therefore be detailed later in the corresponding chapter (Section 3.2.5.1).

1.4.2 Structure of the wire from CVD

Since the CNT wires are composed and formed by individual CNT bonded together with weak van der Waals forces, if no other chemistry treatment was applied, the general behavior of the wires is driven by these interactions. If the idea of an individual super tube of very low density and high mechanical resistance is attractive, the intrinsic properties of these tubes have little influence on the final properties of the microscopic assembly.

The main factors that influence the mechanical responses of the wire can be summarized in three points: the cross-links between CNTs and bundles, the densifica-

tion and the alignment of the individual tubes. The intrinsic mechanical properties of individual tubes have very little influence.

The mechanical properties of wires obtained by dry spinning are limited by the porous structure of the wire. Typical wires involve three or four levels of organization (Fig. 1.15). The wire is made of intertwined fibers (or super bundles) containing bundles with typical diameters between 20 and 60 nm. These bundles are much smaller than the "super ropes" obtained via the super acid process. Finally, the CNTs are the building blocks of this structure. The superposition of different levels of organization leads to very low density (30-50 %) compared to a perfect CNT packing, even after the densification post-processing.

In macroscopic CNT assemblies, the individual tubes as well as their microscopic assembly in bundle could maximize the axial stiffness as well as the mechanical resistance to fracture. New discoveries and progress made in manufacturing processes have shown that it is possible to obtain structures with a strength up to 8.8 GPa and a stiffness of 357 GPa [175]. However, it was unfortunately observed a great variability in the mechanical properties of these CNT wires, with strengths ranging from 0.2 to 8.8 GPa and Young modulus between 30 and 357 GPa, whatever the method used [174, 186, 163]. Such variation in properties complicates their use, and at present they are not at the level to compete with traditional fibers (carbon, Kevlar, glass). It is therefore important to understand the key parameters that affect the mechanical properties of CNT wires.

The structure of fibers applied to textiles has been widely studied for many years [189, 190]. The development of theoretical models for CNT wires considering the many parameters at the nanoscale (type of CNT, impurity, diameter, chiral angle, length, alignment, inter-tube distance) and microscale (number of tubes in a bundle, alignment, density) are widely inspired from these models. But the implementation of such models presents real difficulties resulting from the hierarchical structural complexity of the wires and the interactions between building blocks. In this paragraph, various models expressing the maximum resistances to the rupture as well as the Young's moduli are presented. After optimization of the production parameters, empirical evidence showed that CNTs with few walls and large diameters lead to higher mechanical properties of the wire and best samples.

A CNT wire is a hierarchical structure organized in several scales. The basic element is the CNT, with a diameter of a few nanometers (Fig. 1.15.a). These CNTs agglomerate together to form bundles of several tens of nanometers in "diameter" and several micrometers in length (Fig. 1.15.b). These bundles intertwine to form "super-bundles", or fibers (Fig. 1.15.c). Depending on the production process, these fibers can be stretched unidirectionally and the assembly forms the final wire in the case of direct spinning, or they can be twisted in the case of dry spinning from a vertically CNT array (Fig. 1.15.d).

CNT wires are porous and often have irregular cross section. Different cases are possible considering the bulk density/packing density. If all the tubes are straight, aligned and closely packed, a parallelogram unit cell can be defined. The minimum

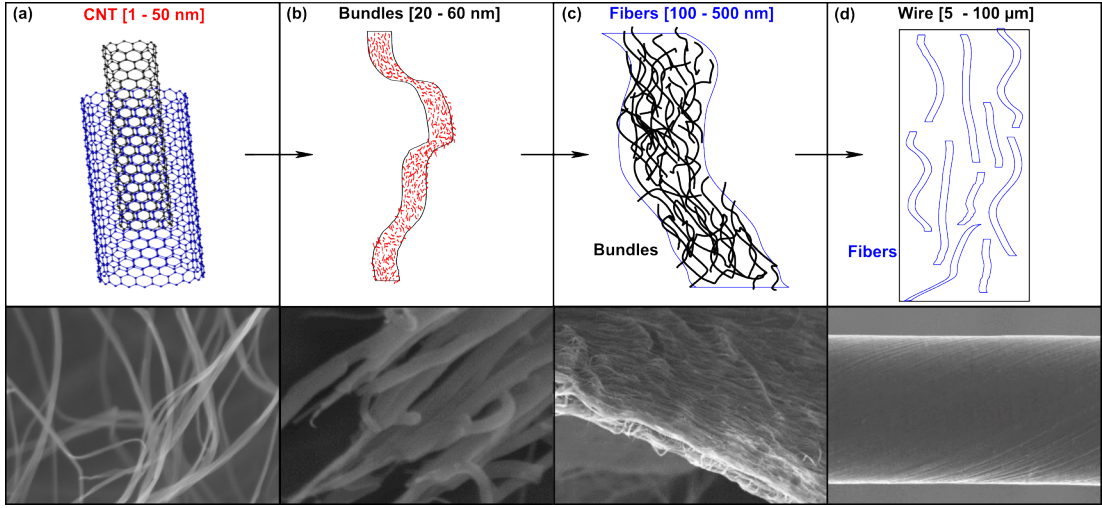


Figure 1.15: Wire structure at different scale: (a) Individual elementary CNT; (b) Bundles with a width of several tens of nanometers; (c) Fibers of several hundreds of nanometers thick; (d) Wire, final product, some micrometers thick.

porosity of the wire is expressed by

$$p_{min} = 1 - \frac{\pi \left[1 - \left(\frac{d_i}{d_{CNT}} \right)^2 \right]}{2\sqrt{3}} \quad (1.64)$$

with d_i the diameter of the inner diameter of the CNT and d_{CNT} the outer diameter. If the CNT is considered as full ($d_i = 0$), the minimum porosity in this configuration is 9.3 % and the maximum packing is 90.7 % [191]. The maximum number of CNTs that can be packed in a cross section of $1 \mu m^2$ is given by

$$n_{CNT-max} = \frac{2\sqrt{3}}{3d_{CNT}^2} 10^6 \quad (1.65)$$

In reality, CNT are not closely packed and not correctly aligned. The real wire porosity derived from the ratio between the density of the wire and the density of the CNT

$$p_{real} = 1 - \frac{\rho_{wire}}{\rho_{CNT}} \quad (1.66)$$

In an assembly, the packing is affected by both the compression and the alignment of the CNT. The CNT packing fraction Φ_P increases as a cubic function of the pressure P needed to compress randomly the CNT. It is defined by the van Wyk equation

$$\Phi_P = \sqrt[3]{\frac{P}{KE}} \quad (1.67)$$

with E the Young's modulus and K a proportionality factor determined by experiment.

In a CNT wire, the CNT packing fraction is higher in the center than in the periphery [192] and increases with the degree of twist.

For a 3D random orientation of CNTs composing a wire, considering these CNTs non-aligned and with a large aspect ratio, the number of contact points between two CNTs per volume unit can be expressed as $N_c = 2\Phi_V \bar{r}$ where Φ_V is the volume fraction ($\Phi_V = \text{"volume total of the fibers"} / \text{"volume of the wire"}$) and $\bar{r} = l_{CNT}/d_{CNT}$ is the average CNT aspect ratio [193]. For non-aligned and non-slender fibers, the number of contact points is $N_C \approx 8\Phi_V$ if we consider an independence of the orientation distribution. The number of CNTs per volume unit is $n_v = 4\Phi_V/(\pi d^2 l)$

Finally, the breakage behavior can be described in three different stages : a localized deformation with shrinkage in a certain region, a rotation of the wire in the opposite direction to the manufacturing twist and finally the failure of the wire [194]. To model the structure of the wire, the notion of CNT cluster is defined as a group of CNTs assembled in a hexagonally close-packed structure (Fig. 1.16.a).

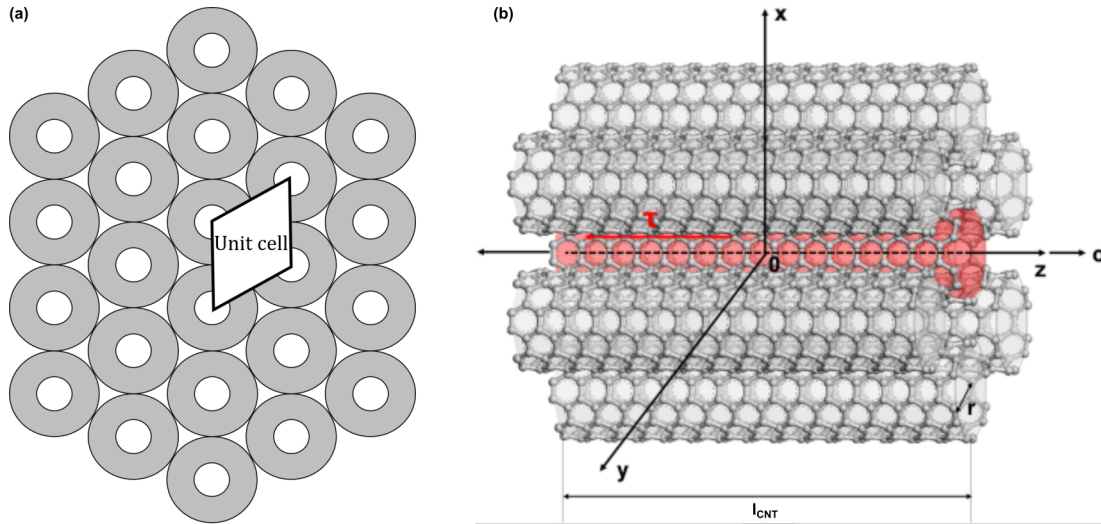


Figure 1.16: (a) CNT hexagonal packing (from Ref [191]; (b) Representative volume element (RVE) of the hexagonally close-packed CNTs (from Ref [194])

The interfacial shear stress is calculated by [194]

$$\tau = \int_0^\alpha \frac{2E_p}{\pi d_{CNT} s^2} \cos \theta d\theta \quad (1.68)$$

with E_p the variation in the potential energy caused by positional changes of the CNT cluster in the twisted wire, s the slip length of the CNTs, θ the angle between the cluster and the axis of the wire and α the twist angle of the wire.

Similarly the frictional stress can be then calculated by [194]

$$\tau_{fric} = \frac{4\mu}{d_{CNT}} \int_0^s G \left(1 - 2 \frac{s-q}{l_{CNT}} \right) dq \quad (1.69)$$

with G the shear modulus and μ is the frictional coefficient between tubes which can be fixed at 0.9 [195]. G is given by [194]

$$G = E_{CNT}\varepsilon \left[\frac{1 + \nu_y}{1 + 2\nu_{CNT}} \frac{c^2}{u^2} (1 - u^{1+2\nu_{CNT}}) - \nu_y \frac{1 - u^{2\nu_{CNT}-1}}{2\nu_{CNT} - 1} \right] \quad (1.70)$$

where E_{CNT} is the Young's modulus of a tube, ε the wire extension, ν_{CNT} and ν_y are respectively the axial Poisson's coefficient for a CNT and for the wire. The modulus and the Poisson's coefficient for a CNT were calculated within an structural mechanics approach [196] and are respectively 1 TPa and 0.2.

By defining a representative volume element (RVE) (Fig. 1.16.b), a modified shear-lag model was used to determine the slip length [197]. The axial stress which has to be applied to pull-out the CNT against the interfacial stress due to the van der Waals with neighboring CNTs is defined as

$$\bar{\sigma} = \left[\frac{\left(\frac{d_R}{2}\right)^2}{\left(\frac{d_{CNT}}{2}\right)^2 + \frac{E_R}{E_{CNT}} \left(\left(\frac{d_R}{2}\right)^2 - \left(\frac{d_{CNT}}{2}\right)^2\right)} \left(1 - \frac{\cosh(\alpha s)}{\cosh\left(\frac{\alpha l_{CNT}}{2}\right)}\right) \frac{7sE_{CNT}}{l_{CNT}} \right] \quad (1.71)$$

with d_R and E_R the diameter and the Young's modulus of the RVE, which is $d_R = (3/2)d_{CNT}$ and $E_R = (3/4)E_{CNT}$ if we consider six CNTs surrounding a CNT as the matrix. The twist angle of the wire α is defined as

$$\alpha = \sqrt{\frac{1}{1 + \nu_{CNT}} \left(\frac{d_R^2 - d_{CNT}^2}{d_{CNT}^2} \right) \left(\frac{d_{CNT}^2 + \frac{E_R}{E_{CNT}} (d_R^2 - d_{CNT}^2)}{d_R^4 \ln\left(\frac{d_R}{d_{CNT}}\right) - \frac{1}{64} (d_R^2 - d_{CNT}^2) (3d_R^2 - d_{CNT}^2)} \right)} \quad (1.72)$$

Two stresses governs the strength of the wire : the interfacial shear stress (IFS) and the frictional stress between bundles. The IFS stress was calculated by molecular dynamics simulations and the frictional stress with wire mechanics. The twist during the manufacturing induces a lateral pressure toward the axis of the wire, increasing the frictional forces. During the tension, the bundles bear most of the load. With the increase of the strain, isolated CNTs are stretched along the axis of the wire and start to bear the stress.

The model assumes a wire composed of N bundles, twisted with an angle θ . In these bundles, n bundles starts to be damaged. The summation of all the contributions and the adjustment of the bundle damaged fraction leads to the mechanical behavior during the tension.

This model does not describe fully the mechanism. The inter-tube bonding and the shear strength are not considered. Vilatela proposed such a model [198]

A synthesis of the mechanical properties depending on their manufacturing process for pristine wire is proposed in the table. 1.8 [199].

Table 1.8: Summary of the structural parameters of elementary CNTs (length, diameter, number of walls, alignment and twist angle) and mechanical properties of CNT wire (strength, Young's modulus and strain to failure) for different method of wire production (from Ref [199]).

Process	Structural parameter					Mechanical parameters			Ref
	l_{CNT} [μm]	d_{CNT} [nm]	n_w	α_a	α [$^\circ$]	σ_{max} [GPa]	E [GPa]	ϵ_{max} [%]	
Liquid crystal	-	-	1	-	-	0.12	120	-	[178]
Liquid crystal	4.3	2-4	2-3	0.99	-	1.00	120	1.4	[156]
Liquid crystal	100	50	>50	0.92	-	0.15	69	2.5	[200]
CNT array	260	≥ 6	≥ 17	-	15-20	1.17	53.5	~ 3	[201]
CNT array	650	~ 10	~ 9	-	21	1.91	330	7.02	[202]
CNT array	2100	~ 40	~ 30	-	25	0.77	51.1	~ 2	[203]
CNT array	250	~ 15	~ 15	-	7.7-9.2	1.6	110	~ 1.4	[175]
Direct spinning	-	4-10	2	~ 0.8	-	0.86	357	4-8	[204]
Direct spinning	-	-	1	-	-	-	-	~ 5	[205]
Direct spinning	-	2-7	≥ 2	0.81	-	0.36	-	22-25	[206]
Direct spinning	-	8-10	2	0.86	-	1.25	66.8	~ 18	[207]

1.4.3 Mechanical properties and improvement

Since the mechanical properties are widely dependent on the individual CNT composing the wire, the manufacturing process and the post-processing treatment, it is complicated to give accurate data on the mechanical properties.

Despite exceptional products showing strength three times larger than the strongest commercial fiber (T1000) and specific stiffness four times bigger than the specific stiffness of the M70J fiber [181], the mechanical properties of the CNT wire are usually weaker than carbon fibers and well below the mechanical properties of individual CNT. Depending on their assembly method, the strength can go from 116 MPa up to 8.8 GPa for the strength and from 120 GPa up to 357 GPa [163, 175].

It is customary to speak of linear density expressed in tex, with $1 \text{ tex} = 1 \text{ g.km}^{-1}$, unit particularly used in the textile field to describe the strength or the modulus of the CNT wire (we speak about specific strength and specific modulus) [175].

1.4.3.1 Influence of the intrinsic properties of the CNT

Studies have shown that the structure of CNTs (diameter, length, morphology, defect, purity and orientation) plays a role in the mechanical properties of the microscopic wire. As seen previously, CNTs can theoretically have perfect structures, making these tubes ideal materials at the individual level, but also in their assembly into microscopic threads. Unlike MWCNTs, SWCNTs and DWCNTs can be manufactured virtually flawless. This makes them preferential candidates for obtaining good performance. Using large diameter and long length DWCNTs with

an aspect ratio of 10^5 shows a specific strength of 2.2 N.tex^{-1} and a specific modulus of 160 N.tex^{-1} . These values are close to those of the best commercial fibers available on the market [208]. The large diameter and the flattened tubular structure of the DWCNTs make it possible to maximize the surfaces in contact with the adjacent tubes. This results in optimal charge transfer between neighboring tubes when energized.

The key parameters influencing the mechanical properties of CNT wires, microscopic assemblies, have been described by two commonly accepted models. The first consists in considering the wire as an one-dimensional material, composed of intertwined basal elements: the CNTs [147, 209, 198, 210]. Based on this principle, the mechanical tensile strength is estimated by the theory of classical wire mechanics [211]

$$\sigma_{wire} \approx \sigma_{CNT} \cos^2 \alpha \left[1 - \left(\frac{k}{\sin \alpha} \right) \right] \quad (1.73)$$

where σ_{wire} and σ_{CNT} represent the tensile strength of the wire and the tensile strength of the CNT respectively, α the twist angle of the wire, l_{CNT} the length of the CNT, d_w the diameter of the elementary CNT, Q the migration length of the CNT from the surface to the core and μ the friction coefficient of a CNT with the neighboring CNTs and k a "slip" factor defined as

$$k = \frac{1}{3l_{CNT}} \sqrt{\frac{Qd_{CNT}}{\mu}} \quad (1.74)$$

This equation indicates that the tensile strength for the wire increases with the decrease in diameter of the elementary CNTs and the increase in their length, the decrease of the migration length and the increase of the coefficient of friction between the CNTs [199]. However, this equation does not seem to be applicable in a practical case because of two factors. The first is the dimension difference between the CNT nanoscale and the microscale of the wire. The second is the deformability of the CNTs. This equation is derived from textile field and the elementary fibers are microscopic scale with millimeter lengths. The hypothesis for this model are fibers considered as non-deformable cylinders with a circular cross-section. CNTs do not fit these conditions.

To overcome these limitations, a new equation considering the failure of the wire as the rupture of the weak inter-tubes interactions was developed [198]. This model takes into account the contributions of the shear force between adjacent tubes, the deformability of the CNTs and even some of their geometry characteristics such as the number of walls and the diameter of the tubes, in the calculation of the strength of the wires [212].

Various studies analyzed the different mechanical properties and the parameters which influence the results. It has been shown that, for wires produced by wet-spinning, the strength and the Young's modulus increase with the increase

of the CNT length [213] with strengths and moduli from near 0 to 300 MPa and from near 0 to 25 GPa respectively for effective length between 500 and 4200 nm [157]. This is explained by the larger surface available to interact with neighboring CNTs. The density of end to end connections (indicated by the purple circles) between CNTs decrease with the increase of the CNT length. The extremities of a tube are defects because the inter-tube bonds between CNTs are much weaker than the covalent bonds within a single molecule (Fig. 1.17). The length of the CNT is a determinant factor in the mechanical characteristic of the wire. The assembly of long individual tubes reduces the number of end to end connections between CNTs, which are the weak points of the wire [214]. The wire strength and the Young's modulus may reach 1.35 to 3.3 GPa and 100 to 263 GPa respectively. The relation between the strength and the length of the array can be explained by two possibilities. As the packing is very low, the tubes are not in close contact with other tubes. The load transfer between CNTs is then very weak. The longer the length, the larger the fraction of tubes that can support the load. Moreover, the area where tubes end, is a weak point. If the length of the tubes is short, there is more area where tubes meet. It leads to an increase of weak points and a decrease of the strength [147, 215, 199]. With the increase of the array length, it has been observed a linear increase of the tensile strength [202, 203].

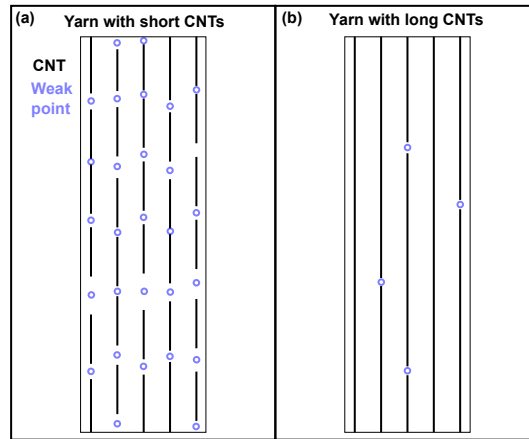


Figure 1.17: Schematic depicting two wires composed of CNTs of different lengths: (a) Short CNTs; (b) Long CNTs (from Ref [154]).

For macro-composites, fibers reinforcing the material is continuous and σ_L is the strength of the fibers. For CNTW, individual tubes are finite elements implying a load transfer between tubes through shear. σ_L is then the maximum axial stress in a bundle before shear failure. To adapt the model for a CNTW, σ_L and τ_b are linked with two crystalline elastic constant parameters e_c and g [216]. This model implies that the wires is considered as a network of individual tubes network and with a fibrillar crystallite model, the strength of the CNT wire can then be written as [217]

$$\sigma \approx \left[\frac{\cos^4 \theta}{\sigma_{mb}^2} + \left(\frac{1}{\tau_c^2} - \frac{1}{\sigma_{mb}^2} \right) \cos^2 \theta \sin^2 \theta \right]^{-\frac{1}{2}} \quad (1.75)$$

with θ the angle between the direction of the load and the parallel aligned CNTs, τ_c the critical shear strength of the wire and σ_{mb} the maximal axial stress in the CNT bundle before shear failure which was estimated from a polymer fibres of very long chains context as

$$\sigma_{mb} = 1.14\tau_c\sqrt{\frac{E_c}{G_c}} \quad (1.76)$$

where E_c is the Young's modulus in the direction normal to the c-axis (≈ 540 GPa by fitting) and G_c the shear modulus between planes oriented normal to the c-axis (8.1 ± 1.8 GPa by fitting).

Adapted from composite material [218] and specifically a modified Tsai-Hill criterion model, the Young's modulus, with the consideration of small misalignment, can be explicated as [217]

$$\frac{1}{E} = \frac{1}{E_c} + \frac{\exp\left(\frac{\sigma}{G_\nu}\right)}{G_c} \frac{\sqrt{\left(\tau_c^{-2} - 3\sigma_{mb}^{-2}\right)^2 - 4\left(4\sigma_{mb}^{-2} - 2\tau_c^{-2}\right)\left(\sigma_{mb}^{-2} - \sigma_b^{-2}\right)}}{2\left(4\sigma_{mb}^{-2} - 2\tau_c^{-2}\right)} \quad (1.77)$$

with E the Young's modulus of the wire, σ the ultimate tensile strength of the wire, G_ν is a shear modulus to consider additional CNT bundle alignment through plastic deformation ($G = 0.7$ GPa by experimental data and 1.1 GPa for few-walls-MWCNT by fitting equation). In principle, only τ_c and G_c are unknown but can be obtained by fitting experimental data.

From the relationship between stress and stretch-ratio of a tendon [219], the equation can be modified to introduce the stress / strain relation [220]

$$\sigma(\varepsilon) = \int_0^\varepsilon f_W(\varepsilon_s)\sigma_{CNT}(\varepsilon_c = \varepsilon - \varepsilon_s)d\varepsilon_s \quad (1.78)$$

where f_W is a Weibull probability density function, $\varepsilon = (l - l_0)/l_0$ is the engineering strain of the wire, $\varepsilon_s = (l_s - l_0)/l_0$ is the straightening strain, σ_{CNT} is the stress on the tubes and ε_c is the real strain of tubes. f_W represents the straightening behavior and σ_c the extension behavior. To adapt the equation from bio-materials to CNT wire (Fig. 1.18), f_W is replaced by

$$f_W(\varepsilon_s) = \frac{\beta_W}{\sigma_0} \left(\frac{\varepsilon_s}{\sigma_0}\right) \exp\left[-\left(\frac{\varepsilon_s}{\sigma_0}\right)^{\beta_W}\right] \quad (1.79)$$

with β_W a shape parameter, σ_0 a scale parameter.

Finally, the strength of the wire is fully described by

$$\sigma(\varepsilon) = \int_0^\varepsilon \left[\frac{2}{\delta} \left(\frac{\varepsilon_s}{\delta}\right) \exp\left[-\left(\frac{\varepsilon_s}{\delta}\right)^2\right]\right] \left[k \left(1 - \frac{\varepsilon - \varepsilon_s}{2\varepsilon_{max}}\right) (\varepsilon - \varepsilon_s)\right] d\varepsilon_s \quad (1.80)$$

with $0 \leq \varepsilon - \varepsilon_s \leq \varepsilon_{fail}$ and $k = 2\mu G/d_{CNT}$.

Four parameters influence the shape of the stress/strain curves (δ , k , ε_{max} and ε_{fail}). A high δ means a wide distribution of ε_s . A high k means strong interactions between CNTs and then a high strength. A high ε_{max} means long characteristic length of CNTs and a high ε_{fail} means a long slippage length driven by the frictional force between CNTs. The ratio $r_\varepsilon = \varepsilon_{fail}/\varepsilon_{max}$ is the critical factor representing the degree of slippage between CNTs at failure. If

- $r_\varepsilon \approx 1$: CNTs slip past each other continuously until the contact area becomes zero
- $\varepsilon_{fail} \ll \varepsilon_{max}$: the contact area becomes constant during the tensile test (similar to an elastic material)
- $r_\varepsilon \approx 0$: CNTs are suddenly pulled apart without frictional force (similar to brittle material)

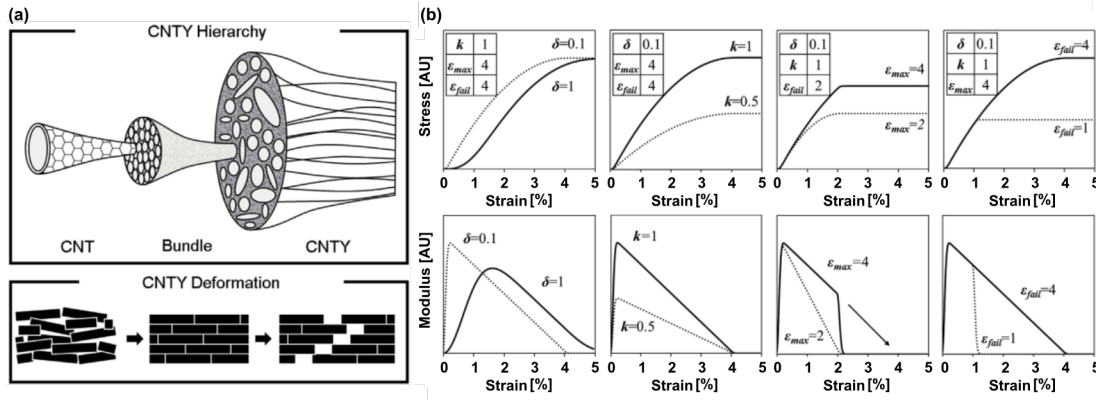


Figure 1.18: (a) Illustration of hierarchical structure and deformation process of a carbon nanotube wire; (b) Stress-strain curves and modulus-strain curves obtained from the developed mathematical model in order to show the effect of four parameters (δ , k , ε_{max} and ε_{fail} (from Ref [220])).

1.4.3.2 Influence of the assembly parameters

1.4.3.2.1 Cross-links between CNTs

The concept unanimously accepted to improve the mechanical properties of CNT wires is the creation of chemical bonds between the tubes [155, 221]. Linking CNTs inside bundles is usually achieved by infiltration of a chemical promoter that creates functional groups between CNTs [222] (Fig. 1.19.a). Covalent bonds being the strongest, it is these bonds that are sought in priority for the improvement of mechanical properties [223]. Many chemical techniques have been tested and used, but a singular improvement in mechanical properties to compete with current commercial carbon fibers requires more research [223, 224, 225]. Infiltration methods inspired by molecular mechanics have shown drastic improvements in CNT wire strength up to 470 % [226].

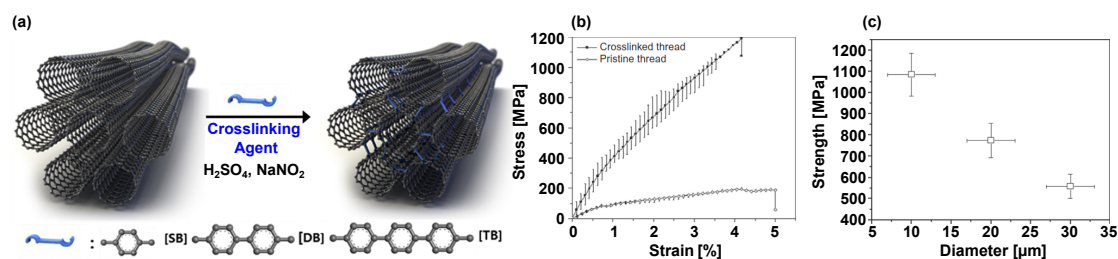


Figure 1.19: (a) Schematic illustration of CNT cross-linking within a bundle (SB: single benzene, DB: double benzene, TB: triple benzene); (b) Tensile strength of cross-linked CNT wires compared with pristine wires; (c) Tensile strength of different diameter CNT wires after cross-linking reaction (from Ref [222, 227])

Stimulation of bonding between CNTs by UV-initiated photopolymerization is a post-processing method that uses the infiltration of HDE (1,5-hexadiene) inside the wire [228]. The results obtained show an increase in the specific strength of the CNT wire by 100 %. A simpler method is the one-step cycloaddition reaction of azide linker molecule and showed an improvement of 600 % with a new tensile strength of 1.2 GPa [223] (Fig. 1.19.b). The reactants used allow the creator molecules to create the bonds directly on the surface of the wall of the CNT. This one-step method is very useful and convenient because it eliminates many usual processes.

Other infiltration methods use free radicals and ionic species to create active sites on the surface of the CNT to create bonds with the molecules of ethylenediamine (EDA) [227]. These amine functional groups correctly react with epoxy-based cross-linker such as MBDGA (4,4-methylene bis(N, N-diglycidylaniline)). This showed impressive improvement by 600% in strength of the CNT wire. A molecule of MBDGA reacts with four amine groups bonding one or more functional groups at the surface of the CNT. Material composed of epoxy resins are widely used for applications in which there is a need of high modulus.

As well as the increase of the number of functional group to improve the mechanical properties of the wire, the effectiveness of the cross-linker infiltration is a major point. The presence of a large number of CNT and bundles already formed makes the infiltration of the cross-linker infiltration more difficult. A solution is to reduce the diameters of the wire. It has been shown that for diameter above 10 μm, the strength of the wire after infiltration of EDA and MBDGA decrease with the increase of the diameter (Fig. 1.19.c). This is probably due to the thickness available in the wire for the creation of amine groups by plasma functionalization. The number of functional groups decreases in depth of the wire. Finally, the solvent penetration is more efficient when infiltrating small diameter wires.

The infiltration of the wire with epoxy allows to constrain the interbundle slip leading to a great improvement in the tensile strength close to 1.5 GPa. The use of PVA in the wire as an infiltrated polymer showed a different coupling role [229]. A plastic flow of the polymer chains occurs when the wire is under strong stress. By doing so, the stress concentration is attenuated and the interbundle contact

areas are protected. Thanks to this PVA infiltration method, a tenacity of 50 J.g^{-1} was achieved, one and a half times greater than for Kevlar and four times greater than for graphite fibers.

The creation of chemical bonds between individual tubes can also be performed by benzene cycles links, obtained with aryl diazonium salt reaction. After 24 h of cross-linking reaction, the nitrogen and oxygen contents, present in diaminobenzene ($\text{C}_6\text{H}_8\text{N}_2$), do not decrease anymore, indicating sufficient time for the creation of cross-linking of fibers. However, this does not mean that all amine groups are consumed and the reaction may not be complete. The individual tubes are bonded through the aryl coupling reaction.

The combination of Raman³ and XPS⁴ showed that diaminobenzene is able to act as an effector to create bonds between CNT. It reacts with sp^2 hybrid carbon lattice on the surface of the fiber. It extracts an electron from the carbon atom of the surface to form a new covalent bond. The specific tensile strength and the specific modulus increased with the time spent in that cross-linking reaction. It increased from 1.6 to 3.7 N.tex^{-1} and from 46 to 210 N.tex^{-1} respectively, corresponding to 130 and 350% of improvement [222]. It has been shown that the shorter the length of the cross-link (SB), the higher the mechanical properties. For single bond and triple bond respectively, the tensile strength decreases from 3.5 ± 0.61 to $2.2 \pm 0.25 \text{ N.tex}^{-1}$ and the modulus decreases from 180 ± 32 to $79 \pm 9.8 \text{ N.tex}^{-1}$ [222].

Contact esterification with sulfuric acid and concentrated nitric acid [163] can also introduce cross-linking reactions, creating functional groups such as carboxylic acid. This treatment makes it possible to eliminate the tubes of smaller diameters, indicated by the disappearance of the peak at 294 cm^{-1} of the radial breath mode (RBM). The second peak of the RBM at 134 cm^{-1} is still present and indicates that the SWCNTs are not damaged by esterification. The specific strength decreases after acid treatment from 0.43 to 0.17 N.tex^{-1} . The treatment provides functional groups needed to create cross-linking reaction, but also defect sites on the wire surface. The introduction of ester linkages between tubes interconnect CNTs bundles with covalent bonding. The specific strength and the specific modulus increase to 1.12 and 47.57 N.tex^{-1} respectively [231].

Work has shown that CNTs with a cross-linked architecture both function as continuous charge transfer pathways, but also couple strongly with polymer chains at the molecular level, resulting in unique mechanical properties completely different from those conventional composites [232]. Coupling at the molecular level between cross-linked CNTs and polymer chains could limit the free deformation of CNT networks and drastically improve the strength of interbundle bonds.

³ The degree of functionalization of the CNT can be checked by Raman spectra, which shows the quantity of defects in the structure following the modification of the sp^2 hybridization ($I_D = 1590 \text{ cm}^{-1}$) to sp^3 ($I_G = 1330 \text{ cm}^{-1}$). The I_D/I_G ratio increases with the increase of the number of functionalization [230].

⁴ The X-ray Photoelectron Spectroscopy is a method used to identify elements and bonds in material using the photoelectric effect. Each bond has a specific binding energy such as: C-C (284.6 eV), C-N (285.7 eV), C=O (288.1 eV) and C-O (286.8 eV) [222].

Thermal annealing also allows to fix a specific geometry of the wire as well as to increase the mechanical properties by the creation of covalent bonds [233, 234]

1.4.3.2.2 Densification

The densification process is probably one of the easiest post-processing to improve the mechanical properties of CNT wires. During the process to obtain the wire, the introduction of vacuum or inclusion in the structure, between the tubes as well as between the bundles, is inevitable. The wire porosity is defined as the volume fraction of spaces between the nanotubes [210]. Accurate measurement of packing or void volume fraction is complicated. The objective of this method is to get the individual tubes closer to each other by removing the voids within the tubes and the bundles created during the assembly [235]. It is important to note that these studies are based on conventional mechanical tests. Observing and analyzing the precise contribution of individual tubes is therefore very complicated. The discontinuous characteristics of the different sub-elements inside the CNT wire (fiber, bundle, individual tube) limit the overall effective shear load strengthening. It is therefore necessary to improve the inter-bundle and inter-tube connections at the micro and nanoscopic scales.

Various methods exist to improve the mechanical properties of CNT wires, which all consist in increasing the contact between individual CNTs. Highly corrosive acids which can partially dissolve the impurities (residual catalytic particles) inside the CNT structure can be used to densify the wire [169]. However, the most popular technique is to use a sacrificial organic solvent which can be directly incorporated during the continuous spinning process [235, 236]. The combination of the use of an organic solvent and a twisting process shows very interesting results in term of density [235].

Twisting process

Geometric constraints during the manufacturing of the wires are factors to be taken into account. To get an effective twisting post-processing, the diameter of the wire must be smaller than the length of the CNTs that constitute it [147]. It has been shown that pure twisting may reduce the porosity by 50 % [191]. A rank of spinnability [237] allows to characterize the ability to spin a wire, with a higher number representing better spinnability, based upon the relative ease, uniformity, and repeatability of the spinning. For low spin rank ($\sim 7-8$), it is difficult to get wires with large diameter ($> 11 \mu\text{m}$). The strength shows a decrease by 43 % for an increase of the number of CNTs in the forest. The width of the CNT sheet used to get the assembly with a twist angle of 20° cannot exceed 7 mm if we want to avoid non-linearity in the fracture load. For high spinnability (> 10) the non-linearity appears for sheet widths higher than 30 mm [200]. During a tensile test, the CNTs on the surface are mostly stretched. The outermost layer carries, then, most of the stress. Higher the diameter of the wire, lower the fraction of such layer. Hence the lower ultimate tensile strength. For very thin sheet, from 3 to 5 mm, the wire can be stronger than the strip. It comes from the densification

impediment due to the small quantity of CNT and the irregular cross section for strips. Strength of 1.89 GPa was obtained with a density of 0.78 g.cm^{-3} . The strips fracture at an average strain of 2.7% while the wires fractures at 3.6%. For strips and wires made with 75 mm sheet width, the load to failure of strips is 177 mN higher than for the wire. The toughness is approximately 78 % larger for wire than for strips (13.7 mN against 7.7 mN). A twisted structure shows an advantage for high energy absorbency [238].

However, a theoretical study shows a dramatic increase of the strength with the increase of the twist angle, but there is an optimum twist angle of 25° for the Young's modulus [239].

Physical densification

The physical densification can be obtained by twisting the wire. This twist modifies the load distribution and the bearing mechanism. This point can be avoided by rolling compression of the wire. However, it imposes the roller to move with a speed close to the pulling speed. A solution is to use a teflon slit which removes any harm that the wire could encounter on a rough surface (Fig. 1.20). After the compression through the teflon slit, the wire is collected and twisted to have more regular cylindrical cross-section. This technique allows an improvement of the tensile strength from 80 MPa for the pristine wire to 900 MPa for the processed wire.

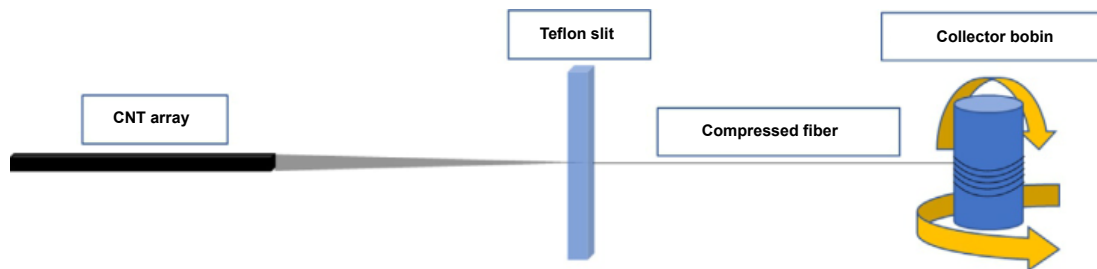


Figure 1.20: Schematic of the slit-assisted densification of CNT fiber directly from the CNT array (from Ref [240]).

The densification by compression can also be performed after the product procurement. The rolling compression of the sample implies an increase of the density. Some CNT wires achieve a density of 1.8 g.cm^{-3} reaching mechanical strength up to 5.5-9.6 GPa [168].

The densification with this method already allows an increase of the mechanical properties, but the combination with organic solvent densification can provide even better products.

Temperature-assisted organic solvent densification

Densification by chemical effect, or surface-tension-driven densification is a technique which makes it possible to increase the density of the wire. A lot of various solvents have been used, for the densification of CNT wires, some with low-boiling-point such as acetone and some with high-boiling-point such as N-methyl-

2-pyrrolidone (NMP). The infiltration through the inside of the CNT increases with the polarisation and the wettability of the solvent [236, 241]. The structure shows a high porosity for pristine wire (Fig. 1.21.a.b) requiring a densification. The process used involves soaking the wire in a solvent batch and then heating up the wire to remove the solvent, to finally obtain a densified product [240]. A process optimisation is needed because of the NMP viscosity, and the combination with the slit-assisted densification can be done to get a better final product. The relatively high viscosity of NMP interferes with the infiltration process and the elimination of the solvent. To overcome this issue, an increase of the temperature reduces the viscosity that increases the efficiency of the solvent and removes more efficiently impurities (Fig. 1.21.c.d.e.f). The porosity can be quantified by calculating a maximum theoretical packing density (1.5 g.cm^{-3}) from observations with a high-resolution TEM [169] or by image analysis of the cross-section [240, 192]. The wire shows a porosity of 46 %, 18 % and 4 % for the pristine wire, the densified wire at a temperature of 23 °C and the densified wire at temperature of 153°C respectively. This shows the significant improvement in term of densification with this process. An acetone densification can increase the density from 0.46 g.cm^{-3} to 0.96 g.cm^{-3} . The use of NMP or chlorosulfonic acid (CSA) doubles the density of an acetone-densified wire, which shows a density of 1.4 g.cm^{-3} for a porosity of 84 % [220]. It is comparable to a 40° twisted wire [242]. Results of the mechanical tests shows a direct implication of the porosity on the tensile strength. The contact between individual CNT and bundles get closer with the decrease of the porosity, leading to a better load transfer. The tensile strength goes from 600 MPa for an ambient temperature densified wire to 1200 MPa for a wire heated up to 153°C [240]. Other studies shows a linear increase of the strength and the modulus with the increase of the density between 0.2 and 0.9 g.cm^{-3} from near to 0 to 300 MPa and near to 0 to 25 GPa respectively [157].

Using a silk fibroin coating on an ethylene glycol densified wire, the improvement may be an increase by 35 % in strain to failure and 83 % in strength [238]. The densification by shrinkage effect of polymere may have a large impact on the new diameter which can be 30 % less than the sum of the individual fibers [229].

The mechanical performance research is often the purpose of the studies, usually for practical applications [243, 244, 173] but a low density can also be a target [58]. CNT wires can achieve a very low density such as $0.2 \pm 0.01 \text{ g.cm}^{-3}$, corresponding to 10 % of a commercial carbon fiber. The extremely low density comes from two distinct factors: large diameter of individual DWCNT (7 nm) and very low packing factor [181].

1.4.3.2.3 Alignment

We just saw above the importance of the cross-linking and densification for the mechanical properties improvement. The last key is the effect of the CNT alignment in the structure of the wire.

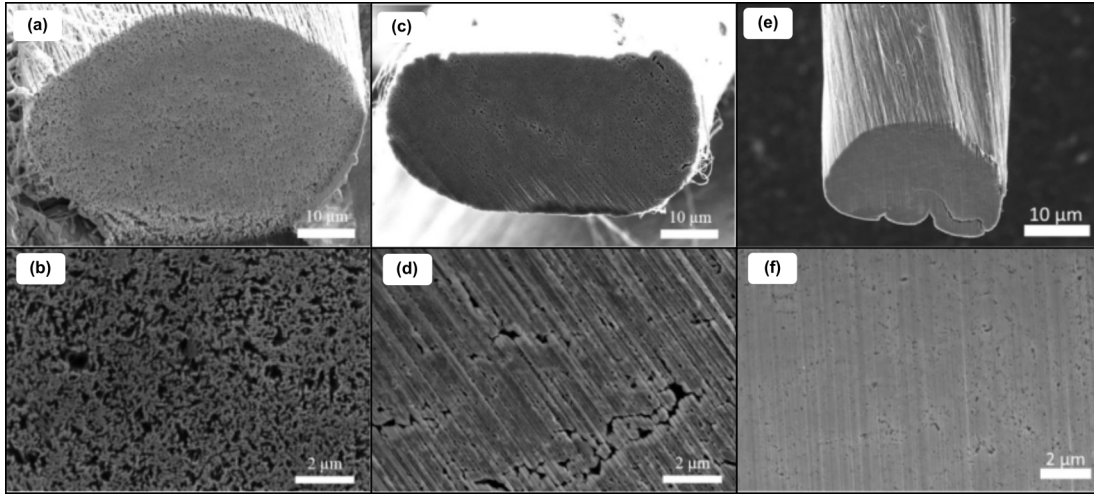


Figure 1.21: SEM images of the cross-section for (a) and (b) a pristine wire; (c) and (d) a wire densified with at 298 K; (e) and (f) a wire densified at 153°C (from Ref [245])

Table 1.9: CNT wire strengthening through densification techniques (from Ref [199]).

Method	σ_{max} [GPa]		Increase [%]	Ref
	Pristine	Improved		
Solvent densification	0.54	1.16	124	[246]
Mechanical densification	0.36	4.34	120	[206]
Polymer infiltration	0.54	1.95	151	[246]
Polymer infiltration and crosslinking	0.55	2.50	438	[226]
Carbon reinforced	0.60	1.70	283	[247]
Surface modification (N-doping)	0.32	0.65	203	[248]
Covalent crosslinking	1.65	3.70	231	[222]

The alignment describes the preferential direction of the CNT and the bundle along the axis of the wire. The CNTs on their own tend to follow the maximum disorder law. That is why they are curved on themselves and entangled with neighbors. The random orientation of CNTs as well as the weak van der Waals forces present at the macroscopic scale drastically degrade the mechanical properties of CNT wires [249, 250]. By aligning the CNT of the wire during the manufacturing process or through post processing treatment, sub-elements get more straight and less wavy. This means that the alignment can be managed during the formation of the individual CNTs or after. With a good alignment, CNTs can work simultaneously and improve the mechanical properties [217].

During the wire procurement, the alignment can be managed by controlling the winding rate, applying a nominal load to remove some of the crimps, adjusting the drawing direction, slicing the CNTs and using very high arrays of CNTs.

The post-processing methods are the mechanical stretching and pressing, solvent casting or soaking, and application of a magnetic or electric field. During the synthesis, CNTs tend to align in parallel during the growth to keep their axial properties [251]. The alignment can be improved by growing super high CNT arrays, in the range of millimeters or longer.

The alignment occurring during the production of the wire is obtained during the drawing and along the drawing direction. By controlling and increasing the winding rate up to 20 m.s^{-1} , the degree of alignment can be widely improved [252, 242] but is also affected by the application of a twist [213]. After getting a first version of the wire, various techniques can be applied to optimize and improve the alignment of the CNTs. By applying an electric or magnetic field on the CNTs during the formation of the wire, the alignment is getting better. An electric field produces an alternative dielectrophoresis current allowing the alignment of the CNTs. By increasing the voltage and the frequency, the alignment increases [253].

Another technique to improve the alignment is to mechanically stretch the wire. However, this method reduces the entanglement between bundles and CNTs which is a factor required to get a high tensile strength [252, 254].

Still another way to get a better alignment is by using a solvent, meaning that the densification process also plays a role in the alignment of the CNTs. The obtained results greatly depend on the choice of the solvent, particularly if the stretching technique is used simultaneously. The use of acetone allows a global increase of the alignment. Since the ethanol has lower wettability and viscosity than acetone, the alignment is slower but the largest bundles become more aligned. However, the densification with acetone is more efficient [254].

The maximum tensile strength shows a direct relation with the waviness (CNTs alignment). The degree of alignment of the CNTs decreases with the waviness. For as spun fibers, the Young modulus increases and the strain at failure decreases ($\sim 7\%$ to 2%) with the increase of the alignment of the tubes. For a poor alignment, the tensile stress does not increase linearly. However, the tensile strength is approximately the same, around 830-880 MPa for all these conditions. The infiltration of polyvinyl alcohol (PVA) does not change the behavior (non-linearity for high waviness wires, increase of the modulus and decrease of the strain at failure). However, the ultimate tensile strength increases from 1 to more than 1.3 GPa [212] and can reach 2.8 GPa, which represents an increase of 910 % compared with the pristine wire [252].

The Young's modulus of the wire as a function of the degree of alignment can be described with a combination of intrinsic properties of CNTs and their organization in the structure [255]

$$\frac{1}{E} = \frac{1}{E_c} + \frac{\langle \cos^2 \Phi_0 \rangle}{G} \quad (1.81)$$

where E_c is the modulus in the direction normal to the c-axis, G is the shear modulus between planes oriented normal to the c-axis, and $\cos^2 \Phi_0$ is the second moment of the c-axis orientation distribution in the unloaded state.

The elastic modulus and the shear modulus of the CNTs can be obtained from the fitting of experimental data from tensile tests. Values of 540 GPa 8 GPa are found respectively [217]. Improving the degree of alignment of the CNTs in the structure of a wire makes it possible to increase the contact surfaces between adjacent CNTs. This increases friction and reduces the likelihood of slippage when stressed. The tensile strength of the wire is therefore better, allowing improvements of up to 147 % in tensile strength [256] and 5 % in strain to failure [257].

If various methods allow a better alignment of the CNTs in order to improve the mechanical parameters, some characteristics of the wire can limit the degree of the alignment. The natural curvature and the defects located into the wire may cause issues to most of the alignment methods [166]. During the stretching of the wire, defects such as empty spaces within the wire may occur, leading to the risk of a necking or slimming in this specific area, compromising the uniformity of the wire.

Different methods exist to quantify the degree of alignment which is an important step in the characterization of the wire. A very easy way is the direct observation using SEM imaging (Fig. 1.22).

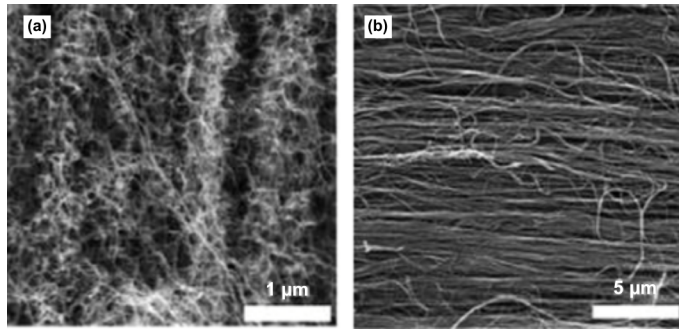


Figure 1.22: SEM images of acetone-densified sheets, with alignment increasing from image (a) to (b) (from Ref [254])

These electron microscopy images provide a visual of the uniformity of the orientation of the CNTs, but only on the surface and therefore do not give the overall alignment inside the wire. In order to overcome this problem, other techniques such as X-ray diffraction, X-ray scattering, Raman spectroscopy as well as calculations using Herman's orientation functions exist.

The use of polarized Raman spectroscopy allows the quantification of the alignment thanks to the study of the G band which is sensitive to the orientation of the optical electric field with respect to the axis of the CNT [213]. The calculation of the alignment is realized by measuring the G band for two particular positions of the laser: parallel and perpendicular to the axis of the wire. The ratio of parallel

G peak to perpendicular G peak evaluates the degree of alignment which increases with increasing ratio (Fig. 1.23).

X-ray diffraction as well as small-angle (SAXS) and wide-angle (WAXS) X-ray scattering also allow the quantification of the alignment of CNTs in the wire [241, 258, 259].

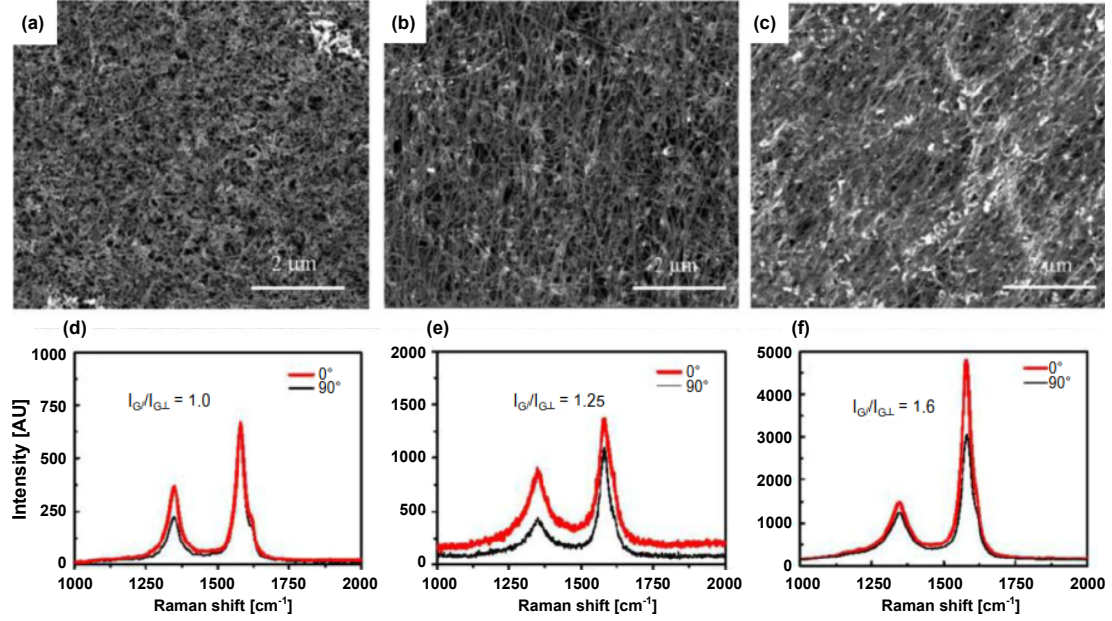


Figure 1.23: CNT orientation characterization. The orientations seen from SEM images (samples with deposition of a few layers of CNT cylinders) (a)-(c) from G peak intensity ratios from polarized Raman spectra along longitudinal and transverse directions for unaligned (a) and (d), slightly aligned (b) and (e), and well-aligned samples (c) and (f) (from Ref [252]).

1.4.4 CNT wire under irradiation

The great interest in studying the effects of radiation on carbon nanotubes comes from their important mechanical and electrical properties and possible future uses in radioactive environments [260]. Carbon nanostructures have a unique ability to restructure their atom network under irradiation. Carbon in pure carbon structure can have sp^2 (CNTs, graphene ...) but also sp^3 (diamond) and sp^1 (linear carbyne chain) hybridization. Irradiation-induced structural changes can lead to property improvements.

The change of structure and the creation of defects in individual CNT have been well studied for years [261]. Molecular dynamic simulations showed that at the nanoscale, irradiation by incident particles of sufficient energy can displace atoms from a SWCNT. It enables to form one or more vacancies which leads to a carbon network reconstruction (Fig. 1.10 and Fig. 1.24) as well as a certain number of primary recoil atoms which leave the tube and can act as crosslinks between tubes (Fig. 1.25). For MWCNTs and SWCNT bundles, more complex defects may

appear as intershell covalent bonds formed by two free bonds at gaps in adjacent walls and mediated by carbon atom recoils [262, 263].

An electron beam may affect MWCNT by inducing a fracture in the external wall whereas the core stay intact [264]. At very high doses the core may be completely amorphized.

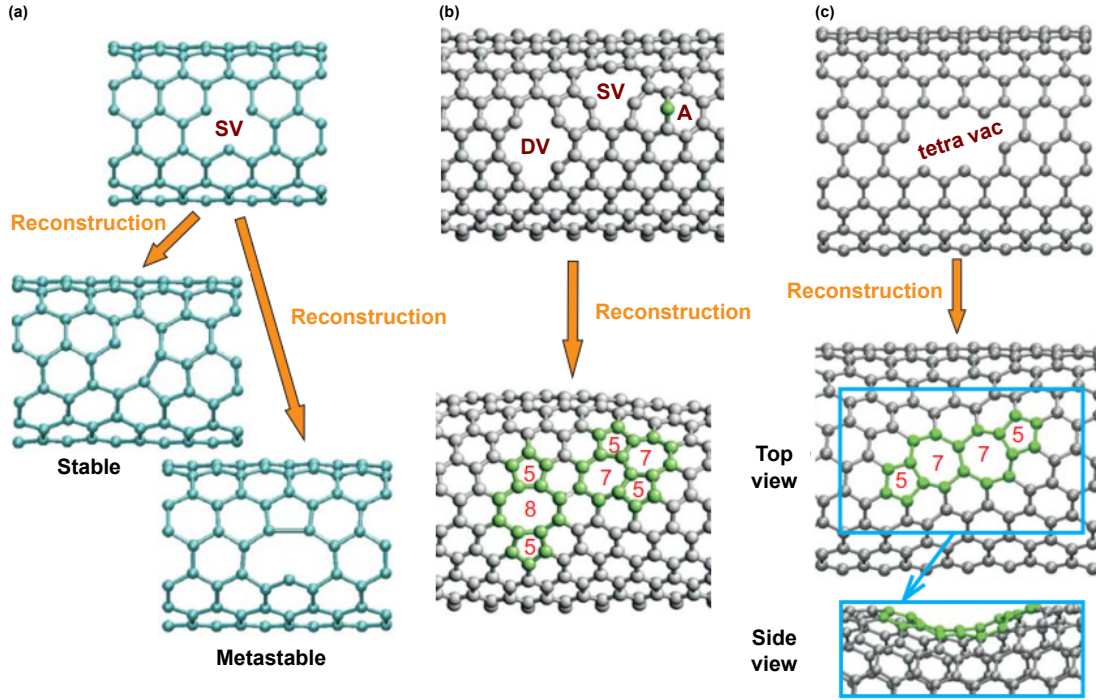


Figure 1.24: Reconstructions of the atomic network near vacancies in single-walled carbon nanotubes only front walls of the nanotubes are shown. (a) Reconstruction of a single vacancy (SV) in a (6,6) armchair carbon nanotube. The top panel shows the original nonreconstructed configuration which is unstable with respect to the Jahn–Teller distortion. Depending on the orientation of the new weak "pentagon bond", the reconstructed configurations can be metastable (when the bond is parallel to the tube axis) or stable (when the bond is almost perpendicular to the tube axis). The latter configuration has lower energy; (b) Reconstructions of the atomic network of a (10,10) carbon nanotube with several point defects. A double vacancy (DV) transforms to an agglomeration of five- and eight-membered rings. A SV and an adatom may form a metastable Stone–Wales (5757) defect; (c) Reconstruction of a tetravacancy. Note that the effective size of the "hole" decreases at the expense of local diameter reduction, so that carbon nanotubes can be referred to as self-healing materials (from Ref [261]).

At the scale of the individual tube, an increase of the number of defects increase the intershell load transfer. An optimal load transfer can be achieved by very few crosslinks [264]. Molecular dynamic models are very useful for simulating the atomic-scale structural changes of molecules. These simulations performed for carbon ion irradiations [87] with energies varying from 50 to 300 eV were tested on bundles of SWCNTs. In the case of a perfect structure, without defects, the energy required to fully move an atom from its lattice is 21.9 eV. In this condition,

the incident atom takes a place in the tube and the displaced atom moves towards the opposite wall. To knockout an atom from his lattice, the energy of the incident particle needs to be much larger than T_d which is the displacement threshold energy [261]. This knockout energy is expressed as

$$E_{ko} = \frac{(m_c + m_{ip})^2}{m_c m_{ip}} T_d \quad (1.82)$$

where m_c is the mass of the carbon atom, m_{ip} is the mass of the incident particle (ion, proton, electron...).

It has been shown that whatever how high is the fluence, if the energy is not high enough, very few bonds are created. At high energy and low fluence the creation of inter-tube bonding is significant in the center of the beam. However, a too high energy and fluence produce important damages and the CNT can be amorphized. As seen previously, the irradiation can create different kinds of defects in the structure (single atom vacancies, Stone-Wales defects ...). The size of the bundle plays a key role in the inter-tube bonds creation, due to the distance between the tubes and the center of the bundle. This model shows how a carbon ion beam [87] with the adequate parameters of energy and fluence could increase the mechanical properties of bundles (Fig. 1.26).

Carbon atoms knocked out by irradiation from the individual tubes or from purification residues can become moving interstices. These atoms are candidate portals for the creation of intertube bonds. They can move in the space between the tubes before finding an ideal position for the creation of an ideal bridge. The irradiation in the air is necessary to create new bonds by increasing the oxygen concentration and increasing the number of carboxyl groups [265]. Two carboxyl groups in the vicinity of the tubes can create stable bonds: carbon oxygen double bond and carbon oxygen carbon bond. Crosslinks can form either with the participation of interstitial carbon atoms created by irradiation or by chemical reaction induced by irradiation between carboxyl groups. This last process requires the simultaneous presence of two chemical groups on opposite tubes which is very unlikely [266]. The presence of air for proton irradiation seems to play a key role to enhance the mechanical strength by creating intertube cross-linking. The irradiation induces an ionization and an dissociation of air molecules which could lead to a higher probability of interaction with the carbon atoms of the wire [267].

Equations allow to describe the number of defects induced by the irradiation [268]. The characteristics distance l_D between defects in a CNT and the areal density of defects ρ_D can be described as

$$l_D^2 = (1.8 \cdot 10^{-9}) \lambda_{laser}^4 \left(\frac{I_D}{I_G} \right)^{-1} \quad (1.83)$$

and

$$\rho_D = (\pi l_D^2)^{-1} \quad (1.84)$$

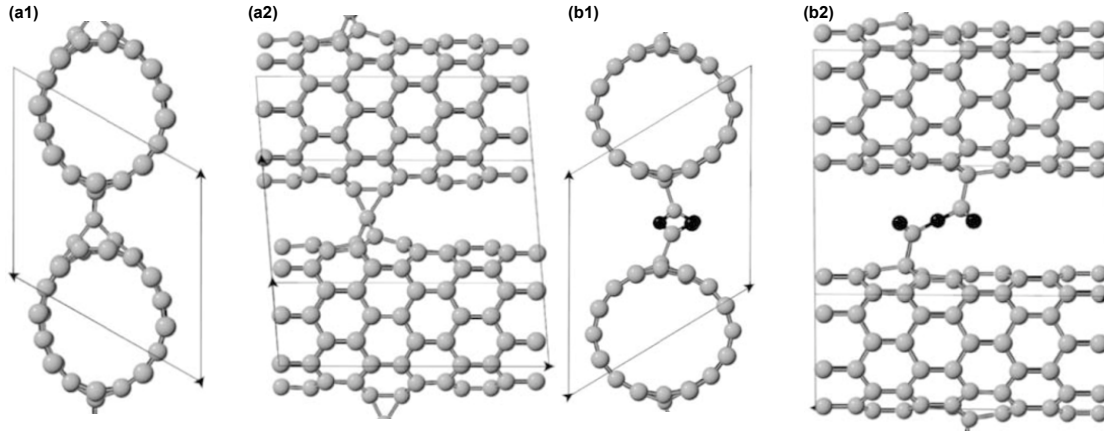


Figure 1.25: Atomic models based on DFT calculations. Two different kinds of bridges formed between neighboring carbon nanotubes: (a1) frontal and (a2) side view of a bridge involving carbon atoms formed through a fourfold coordinated interstitial carbon atom; (b1) frontal and (b2) side view of crosslink formed in an electron-beam-induced chemical reaction between carboxyl functional groups attached to neighboring nanotubes. Oxygen atoms are shown in black. (from Ref [266]).

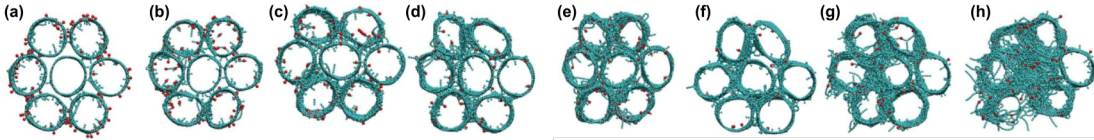


Figure 1.26: Modification of the the SWCNT bundle structure after irradiation: (a) $2.10^{14} \text{ cm}^{-2}$ fluence, 50 eV/ion; (b) $2.10^{14} \text{ cm}^{-2}$ fluence, 100 eV/ion; (c) $2.10^{14} \text{ cm}^{-2}$ fluence at 150 eV/ion; (d) $1.2.10^{14} \text{ cm}^{-2}$ fluence at 200 eV/ion; (e) $2.10^{14} \text{ cm}^{-2}$ fluence at 200 eV/ion; (f) $4.10^{13} \text{ cm}^{-2}$ fluence at 300 eV/ion; (g) $1.2.10^{14} \text{ cm}^{-2}$ fluence at 300 eV/ion; (h) $2.10^{14} \text{ cm}^{-2}$ fluence at 300 eV/ion (from Ref [87]).

with λ_{laser}^4 is the Raman laser wavelength, I_D and I_G the intensity in the I and G band of the Raman spectra respectively.

Then, the number of defects induced by irradiation in a CNT can be estimated by

$$n_{defects} = \pi \rho_D d_{CNT} l_b \quad (1.85)$$

with l_b the length of the bundle.

For a wire of CNTs, theoretical [264, 87] and experimental researches with different types of irradiation have been conducted. Gamma [269, 270], proton [267], electron [266] and ion [87, 271] irradiation induce an increase of the mechanical and electrical properties in air. The mechanical properties, strength and Young's modulus increase whereas the strain at failure and the diameter decrease for an optimum irradiation dose which depends on the irradiation type and material [272]. Two mechanisms of improvements were highlighted : 1- for low dose, the improvement is significantly due to the intra-bundle bonds, 2- at high dose this is the inter-bundles cross-links which dominate [268].

At a microscale, individual tubes located between bundles can be a bridge for the creation of bonds between adjacent nanotubes or bundles and fix their mutual position [266]. The manufacturing process and more specifically a high packing factor of the microscopic wire is a key factor to minimize the intertube distance and increase the probability of bonds creation [270, 271]. However at high doses, the concentration of defects is too high and the interconnected network of CNT collapses leading to a decrease of the mechanical and electrical properties and a change in the structure [269].

Considering a CNT mat composed of DWCNTs as a network of randomly oriented bundles of equal length (l_b) and equal width (w_b), model was constructed that express Young's modulus of a CNT mat as a function of the properties of the CNT bundles and the cross-linking [268]

$$E_{mat}(z) = \frac{E_b w_b}{8L} q \left(\left(\frac{z w_b}{l_c} \right)^2 E_1(z) + \left(3 + \frac{1}{2(1+\nu)} \right) (1 - e^{-z}(z+1)) \right) \quad (1.86)$$

where E_b is the Young's modulus of the bundle, q the areal density of CNT bundles, ν the Poisson coefficient of the mat, and l_c the critical length of the bundle. If the length is above l_c the deformation comes only from bending. If the length is smaller than l_c the deformation is only from stretching and shearing. z is a dimensionless parameter related to the number of irradiation-induced crosslinks per nanotube n_{links} by $z = l_c/l_b n_{links}$. E_1 [273] and l_c [274] are expressed as

$$E_1(z) = \int_1^\infty \frac{e^{-zx}}{x} \quad (1.87)$$

$$l_c = (1.9w + 0.11) \sqrt{2(1+\nu)} \quad (1.88)$$

The model can be modified by replacing z with $(z + z_0)$, where $z_0 = l_c n_0/L$, where n_0 is the number of initial effective crosslinks before irradiation ($n_0 = 2q/\pi$). Furthermore, to describe the reinforcement at the tube-bundle level, E_b increases with the number of crosslinks generated by irradiation.

$$E_b(z) = E_0 + E_{max} (1 - e^{-Cz}) \quad (1.89)$$

where E_0 is the bundle Young modulus before irradiation, E_{max} represents the maximum improvement of the modulus upon irradiation and C is a constant that defines the rate of the bundle stiffness. The Raman measurements showed that even at high doses (10^{16} ions.cm⁻²), the CNTs kept their level of graphitization ($I_G/I_D \geq 2$).

The irradiation of DWCNT-epoxy mats with a 3 MeV He⁺ ion beam showed a drastic increase of the Young's modulus as well as of the strength from ~ 5 to ~ 20 GPa and ~ 200 to ~ 550 MPa respectively with the increase of the dose from pristine wire to 10^{16} ions.cm⁻²) [268]. The improvement seems to be the most effective for low doses with almost 100 % of increase before a slower linear increase.

Studies on the irradiation with the same conditions of CNT wires showed that a low fluence induces a removal of defects, leading to the increase of the strength. But a too high fluence create destructive defects which conduct to the deterioration of the wire [271]. This was supported by molecular dynamic simulation highlighting the different stages of cross-linking creation to the damages of the structure [87].

These different studies are interesting since they give a starting point and the basic knowledge of the physical phenomena occurring during the irradiation of CNT materials under different conditions. However, the topic of this PhD work is about the irradiation at very high energy and intensity, making it difficult to compare with these works.

1.5 CONCLUSION

As seen previously, there are a lot of studies on the mechanical properties of CNT wires and the parameters allowing their improvement are plentiful. We have tried here to put forward some references that we think important, in order to be able to refer to them. This is however not an exhaustive list. Furthermore, we encountered difficulties in comparing these results since the high number of varying parameters makes each study an almost unique experience.

The main point for this work, is the study of the modifications of the mechanical properties of CNT wires under irradiation. For this specific theme, the searches are much less numerous, and the experiences less. Molecular dynamics models can give theoretical predictions but experimental studies are more limited. The irradiation conditions are not very diversified, probably due to the lack of interest shown in the subject, but also to the technical possibilities.

The main experiment of this thesis carried out within CERN involves energies (450 GeV) and intensities ($3.2 \cdot 10^{13}$ protons per pulse) achievable only... at CERN. A comparative analysis of the results with previously published results is therefore difficult. However, understanding physical phenomena occurring at low energy remains an excellent starting point.

"Imagination is more important than knowledge..."

Einstein

Chapter 2

Mechanical characterization

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2.1 INTRODUCTION

Wire-scanners (WS) are devices used since the 1980s [40, 41] to measure the transverse beam density profile, by moving a thin wire across the particle beam and detecting the secondary particles. The interaction between the beam and the wire creates secondary particle showers with an intensity proportional to the number of particles crossing the wire. These secondary particles are intercepted by a scintillator positioned downstream of the wire, coupled to a photo-multiplier which amplifies the resulting signal. The acquisition of the wire position and the signal intensity are combined to reconstruct the transverse beam density profile. The first WS moved linearly with stepper motor [42, 43] which required very low scan speeds, of up to 1 m.s^{-1} [44, 45]. Various studies on this instrument were carried, from the influence of the diameter of the wire [46] to its thermal behavior [43, 47, 48]. Another generation of rotary WS was then designed to meet higher specifications [49] in the Proton Synchrotron Booster (PSB) and the Proton Synchrotron (PS). However, in the coming years, the beam will progressively be smaller and more intense. This will lead to two main issues with the current generation of WS: an overheating of the wire [50, 51, 52] and an insufficient resolution for the beam size. In order to address the overheating issue, a new generation of fast wire-scanners [35, 54] was developed at CERN, for the whole accelerator chain, from PSB to the Super Proton Synchrotron (SPS). The whole engineering concept has been redesigned with new machining and manufacturing concepts [55, 56]. The major improvement is the increase of the scan velocity up to 20 m.s^{-1} , which allows to reduce the beam/wire interaction time and consequently the increase of temperature. Everything in this new WS was changed, except the centerpiece of the instrument: the wire. The same material with the same mounting system has been used from the beginning, *i.e.*: partially copper coated carbon fiber. The problem is that first, most common wires (carbon fiber, gold coated tungsten,...) will be destroyed in the HL-LHC conditions[50] and second, the precision of the wire installation is currently mainly dependent on the technicians who install the wire manually and is prone to large variations in the calibration results.

As an alternative to conventional carbon fibers, we considered fibers made of Carbon NanoTubes (CNT), a new exciting material formed by one (Single Wall Carbon NanoTube - SWCNT) or several (Multi Wall Carbon NanoTube - MWCNT) sheets of graphene, coiled on themselves. Those relative densities that can be as low as 0.5, depending on the CNT structure and packing [120] is an interesting characteristic for our purpose. The nanometric size, with diameters varying from less than 1 nm for SWCNT [174] to more than 50 nm for the bigger MWCNTs [275] and typical lengths of the order of the micrometer, [275], the millimeter [276, 277, 202] or even the centimeter [278], are particularly adapted for new technologies such as nanoelectronics [279, 280] or material composites [281, 282, 283, 284]. The "macromechanics" would also be interested in these fabulous characteristics, but individual tubes are not practical for most engineering applications due to their size. More recently, the technology to assemble these

tubes together to make long stranded yarns has opened up a new world of potential applications. Different methods were developed such as dry spinning [178], wet spinning [163, 285], in situ direct spinning [174] and spinning from gas phase [286] that offer a wide panel of possible configurations.

Many academic institutes and industrial companies are interested in investing and developing these new Carbon NanoTube Yarns (CNTY) further. Nevertheless, several studies [257, 287, 288] showed a large difference between the properties of a single nanotube and the properties of the wire assembly. Indeed, the bonds between carbon atoms inside the coiled graphene layers are strong covalent bonds, whereas the inter-tubes bonds inside the wire are weak van der Waals forces. It led to a disappointment for some applications needing high mechanical properties and a microscale structure. Methods to improve the mechanical properties of pristine yarns exist such as chemical treatment [222, 229, 231] or densification by twisting [168, 210, 238]. However, the influence of a partial copper-coating on the mechanical properties has not been studied. Hence, the goal of our study is to determine the best configuration for this new material considering several diameters (10, 20 and 30 μm), and two different mounting systems: 1- glued on a paper frame (PF) and 2- partially copper coated (CC). This new input will be carefully analyzed and will be determinant for the upgrade of the wire scanners used at CERN.

We will first describe the material, methods and protocols used, then describe the results of our measurements and the Weibull analysis done to estimate the maximum tensile stress that we will be able to impose safely to the yarns during their installation on wire scanners of the new generation. This point is crucial to achieve fast scan speeds with minimal deformations as required to monitor with a sufficient precision the beam profile in enhanced luminosity conditions. An in-depth discussion of our results, with a comparison with other results from the literature will then lead us to give some perspectives and propose recommendations for the configuration to be used at CERN.

This section describes the experimental part of the article by A. Mariet et al. [192]. The methods and protocols used are precisely detailed before studying and comparing the internal structure of the commercial yarns tested. The mechanical properties of these wires are analyzed according to two types of assembly (Paper Frame (PF) and Copper Coating (CC) conditioning). A Weibull analysis is used in order to obtain the maximum breaking stresses for the samples in the case of operational threads. Finally, a part on the study of the impact on the mechanical properties of the speed of loading comes to close this section.

2.1.1 Material, methods and protocols

2.1.1.1 Preparation of the tensile tests

The wires used for this study were provided by Hitachi Zosen Corporation. CNTs were obtained by Chemical Vapor Deposition, producing a vertically aligned sheet. [101, 289, 290]. The as-grown CNTs have an outer diameter of 10 to 12 nm. These are typical values which can be found in many productions [178]. The average number of walls composing the tubes are between 5 and 10. wires were then obtained by extracting long strips CNTW of CNT bundles from the sheet. These bundles were twisted and pulled out together [235].

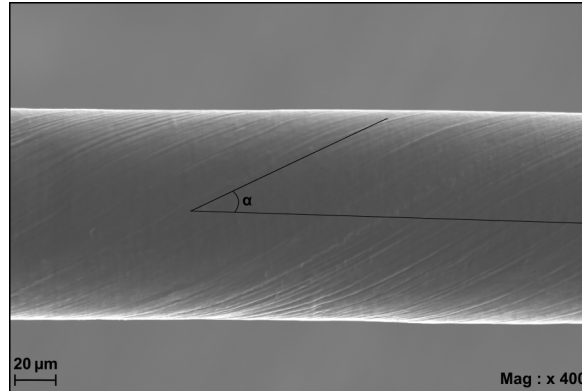


Figure 2.1: SEM image of a pristine CNTW at a magnification of 400. The twist angle α is defined as the angle formed between the axis of the wire and the average direction of the bundles.

The nominal diameters of the wires that we used for our mechanical tests were 10, 20 and 30 μm , but were measured with an electron microscope (SEM) to be 12.71 ± 0.32 , 21.95 ± 0.32 and 32.43 ± 0.27 μm respectively. The real cross-section areas were measured to be 127 ± 7 , 379 ± 13 and 826 ± 14 μm^2 respectively, which are very close to values that can be computed for circular cross-sections. The twist angle α (Fig. 2.1) depends on the rotation speed combined with the pulling out speed during the manufacturing process [291]. There were measured at $16 \pm 1^\circ$, $20 \pm 2^\circ$ and $19 \pm 1^\circ$ for wires of 10, 20 and 30 μm respectively (Table. 2.1). The supplier gives a density between 1.2 and 1.4 g.cm^{-3} for the wires. Because of the

limited amount of available wires and technical problems, we could not measure the density of the wires with nominal diameters 10, 20 and 30 μm . However, for a 100 μm CNTW sample, we measured with an hydrostatic balance based on Archimedes' principle a density of $1.3 \pm 0.2 \text{ g.cm}^{-3}$, which we used in our mechanical simulations (together with a density of 2.1 g.cm^{-3} for 34 μm carbon fibers (CF34)).

Table 2.1: Real measured diameters and twist angles.

	$\varnothing[\mu\text{m}]$	$\alpha [^\circ]$
10 μm	12.71 ± 0.32	16 ± 1
20 μm	21.95 ± 0.32	20 ± 2
30 μm	32.43 ± 0.27	19 ± 1

Our study concerns the effects on the mechanical properties (strain at failure (ε_{max}), tensile strength (σ_{max}), Young's modulus (E) and toughness (T)) of the PF or CC preparations and of the diameters of the wires. Tensile tests were made using a Dynamic Mechanical Analyzer (DMA) (Bose Electroforce3200) at ambient temperature (21 $^\circ\text{C}$) equipped with a 22 N load cell (precision of $\pm 5 \text{ mN}$) and displacement sensor of $\pm 1.5 \text{ mm}$ (precision of $\pm 1 \mu\text{m}$). The gauge lengths of the specimens were 10 and 30 mm for the PF and CC preparations, respectively.

Tests were performed with a displacement control at a speed of 1 mm.min^{-1} . The Young's modulus quantifies the tensile stiffness of a material. It was determined by linear regression of the stress/strain curves on a range of strain comprised between 0 and 1 %.

As already mentioned, two different mountings have been prepared (PF and CC). For PF, we used a self-adhesive paper frame [292] of 12 mm length. The wires are positioned across the frame and fixed with a basic cyanoacrylate glue (Fig. 2.2.a). Both sides of the frame are mounted between two aluminum plates and screwed with four M4 screws. To avoid pre-tension in the wire, the assembly is mounted on the clamps before cutting the frame to free the wire (Fig. 2.2.b). CC samples were also tested since wires currently used in WS are coated on the extremities with a layer of copper (350 μm in diameter). This coating allows the soldering of the wire on the forks through a copper insert. It also allows a better electrical conductivity, used to monitor the good state of the wire. A grid is prepared using a classic printed circuit board (Fig. 2.2.c). A thin layer of copper is deposited around the frame. The wires are then stretched by hand, positioned on the frame and fixed by tape. They are glued with a conductive quick curing bi-component epoxy adhesive Epotecnny E207. The frame is dipped in an electrolytic bath composed of copper sulfate (CuSO_4 , 75 g.l^{-1}), sulfuric acid (H_2SO_4 , 10 % v/v), sodium chloride (NaCl , 0,075 g.l^{-1}), copper gleam PC make up (4 ml.l^{-1}) and demineralized water. A small current of the order of 1 mA is used. Despite

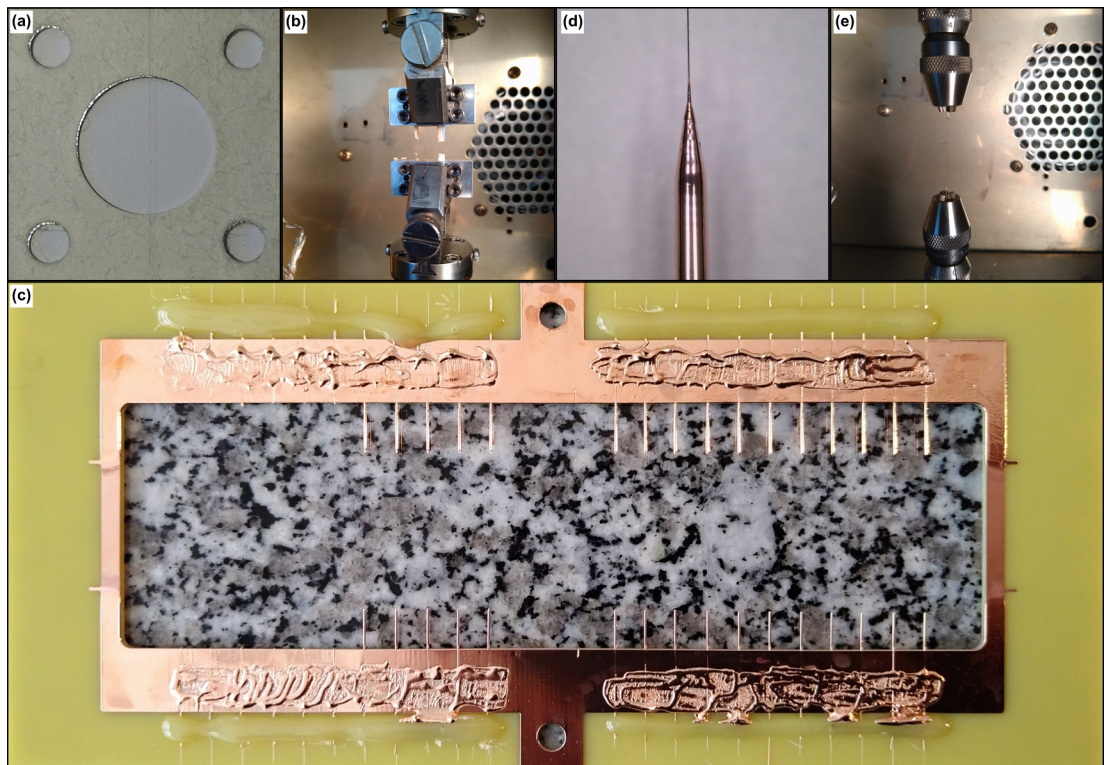


Figure 2.2: (a) Mounting of the wire on the Paper Frame (PF); (b) Assembly for clamping system in the tensile machine; (c) Typical preparation grid for the copper-coating of the extremities; (d) Quality control of the coating for a wire of diameter 20 μm ; (e) Clamping systems for the CC conditioning test.

precise specification of the outer diameter of the coating, the complexity of this step implies a wide range in the results (Fig. 2.2.d). To ensure the good alignment of the CC wires, a cylindrical clamping system has been used (Fig. 2.2.e).

2.1.1.2 Focused Ion Beam (FIB) microscopy

To prepare the samples, mechanical cutting with scissors was performed. The specimens were glued with silver paste to an aluminum stub (standard consumables for SEM). In order to gain improved insight into the microstructure of the CNTs, a cross section was milled by FIB into the samples. For the transverse cross-section, a 20 (radial) $\times$ 2 (axial) $\times$ 1 (thickness) μm^3 platinum (Pt) polishing barrier was initially deposited onto the sample surface at a current of 300 pA in order to facilitate effective polishing. A milling current of 3 nA and accelerating voltage of 30 keV were then used to mill out a section using the silicon (Si) ablation ratio. Subsequent polishing milling steps were performed at 700 pA and 100 pA and accelerating voltage of 30 keV and a final polishing was performed at a milling current of 50 pA and accelerating voltage of 30 keV to leave a smooth surface that could be effectively imaged. For the longitudinal cross-section, a milling current of 3 nA and accelerating voltage of 30 keV was used to mill out the material up to approximately half of the wire diameter. Subsequent polishing milling steps were

performed at 1.5 nA and 700 pA and accelerating voltage of 30 keV and a final polishing was performed at a milling current of 100 pA and accelerating voltage of 30 keV to leave a smooth surface that could be effectively imaged.

2.1.1.3 Analyzing tools

The data analysis was performed by using a combination of Excel worksheets (Microsoft, Redmond, Washington, USA) and python Jupyter Notebooks [293]. Graphs and statistics have been plotted with GraphPad Prism 9 (GraphPad Software, San Diego, California, USA). Images were processed with ImageJ [294] to set up brightness, contrast and get measurements. Figures have been assembled with the free software Inkscape [295]. The void density was calculated using a python script developed internally.

2.1.2 Results and discussion

2.1.2.1 Internal structure analysis

A CNTW can be described as a hierarchical structure. Individual CNTs self-assemble forming bundles and bundles are bonding together in successive layers to form the final wire [198]. A good knowledge of the characteristics of the individual CNTs composing the wire and their packing would be an asset for developing an effective composite model for the wire [173, 198].

A transverse cross-section has therefore been milled in several wires using gallium ions from the FIB, to observe their internal structures (see Fig. 2.3.a for the 20 μm wire). This allows us to measure the packing and the distribution of the CNTs and CNT bundles in the cross-section. For the diameter 20 μm , two sites of interest (SOI) along the diameter have been zoomed in (Fig. 2.3.b). SOI-1 is an area located on the external layer of the wire (2 μm away from the surface). It was observed with a magnification of 7700 (Fig. 2.3.c). This image was binarized (Fig. 2.3.d) from a grey level picture using a threshold manually set to 122 in the python script, in order to visually preserve the extents of the voids while filtering out artifacts. The void density is then estimated by the ratio of black pixels over white pixels. For SOI-1 of the 20 μm wire, we found 0.63 %. SOI-2 (Fig. 2.3.b) is located at mid distance between the outer part and the center of the layer. A picture was taken with a magnification of 52000 (Fig. 2.3.e) and binarized (Fig. 2.3.f). Using a threshold of 110, the void ratio was then estimated to be 0.28 %.

The same approach has been used for a 30 μm wire (Fig. 2.3.g), but due to the larger dimensions, only the external diameter area of the wire was prepared by FIB cross section milling. Two sites of interests have been chosen (Fig. 2.3.h), the first on the external layer of the wire (Fig. 2.3.i) and the second slightly lower but still on the outer layer (Fig. 2.3.k). Both images have a magnification of 50000. The binarisation gives for SOI-1 a void density of 11 % with a threshold of 60 (Fig. 2.3.j) and 10 % with a threshold of 45 for SOI-2 (Fig. 2.3.l). The cross-

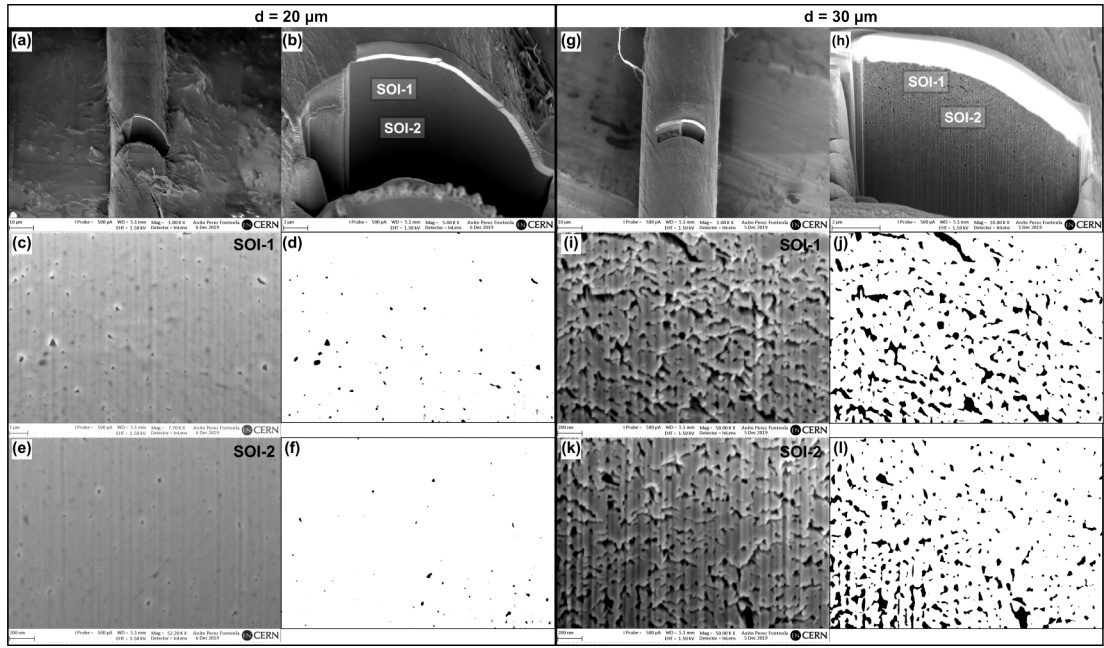


Figure 2.3: FIB images of: (a) Transverse cross-section of a 20 μm diameter wire; (b) Two sites of interests; (c,d) SOI-1 (outer position) and SOI-2 (inner position) have been zoomed in; (e,f) Binarized for the external position with grey level thresholds set at 122 and 110 respectively; (g to l) Same process has been performed for the 30 μm wire with thresholds of 60 and 45 for SOI-1 and SOI-2 respectively.

section at the external layer of the 30 μm presents a similar aspect in the two sites compared. Groups of well aligned nanotubes are discernible and also, locally, small groups of non-aligned nanotubes are visible in the voids. SOI-1 and SOI-2 had a similar aspect with discernible groups of well aligned nanotubes and also, locally, small groups of non-aligned nanotubes, visible in the voids.

From the above results, we can see that the packing is denser (less voids) for the 20 μm wires than for the 30 μm wires, which is in the continuity of the results of a previous study carried out with wires with diameters varying from 30 to 83 μm [296]. This study showed a decrease of the apparent density from 1.2 to 0.6 g.cm^{-3} with increasing diameter. However, despite the fact that various previous studies showed that the packing of twisted wires increases with the twist angle [147, 191, 168, 210], our observations do not seem to support this correlation, since we find quite different packings for similar twist angles ($20 \pm 2^\circ$ and $19 \pm 1^\circ$ for wires of 20 and 30 μm respectively (Table. 2.1)). This discrepancy may be due to the non-uniformity of packing in the 30 μm CNTW versus the uniformity of the packing in the 20 μm CNTW. Indeed, we found that the void ratio at the site of interest located on the outer layer of the 20 μm wire is twice larger than the one at the site of interest located midway between the center and the surface of the wire. This indicates that for this diameter, the packing is not radially uniform. For the 30 μm , both sites of interest show very similar packings, given the uncertainties inherent to the manual setting of the binarization threshold.

2.1.2.2 Copper-coating

Wires used in the current wire scanners are copper coated on the extremities for integration reasons. However, coating the wires with copper is a delicate step in the wire preparation. Therefore, we prepared a new batch of copper-coated wires in order to study the dispersion of their geometrical and mechanical characteristics. Results on the copper-coatings external diameters were very variable compared with the design specifications. They also showed different profiles for a same batch (Fig. 2.4.a). To observe the interface between the wire and the copper-coating, we performed transverse cross-section using the FIB method. The samples were positioned with the appropriate angle with silver paste (Fig. 2.4.b). Different levels of zoom were applied (Fig. 2.4.c). The zoom x200 allows a global view and shows the ratio between the diameters of the coating and the wire. The coating was specified at a diameter of 400 μm to fit in the 500 μm copper insert of the instrument, and is measured at 340 μm . The wire is very regular and measured at 34.4 μm . Increasing the zoom in, we observe a transition layer between carbon and copper with a thickness of about 5 μm . This transition layer was analyzed by Energy-Dispersive X-ray Spectroscopy which revealed the presence not only of carbon and copper, but also of oxygen and sulfur (Fig. 2.4.d). These last two elements are part of the electrolytic bath of the coating. They are non uniformly distributed in the transition region (Fig. 2.4.e). Chlorine and silicon visible in the spectrum are thought to be measurement artifacts.

The observation of the interface between the copper-coating and the wire could lead to the supposition that while filling the voids in the structure of the wire, copper could form new bonds stronger than the weak van der Waals forces between CNTs, which could reinforce the wire. It would then lead to mechanical properties slightly better than for a pristine wire, by strengthening the coating region. However, we observed that the strength results of the tensile tests for d20_{CC} and d30_{CC} batches are well below the results for the d20_{PF} and d30_{PF} batches, which would prevent use of higher tensile forces in order to straighten the wires and, consequently, to improve the accuracy of the WS by decreasing the amplitude of the vibrations. Our FIB/SEM/EDXS observations of cross-sections of CNTW before tensile tests showed that the coating does not modify the shape of the wire but that copper penetrates in the superficial layer of the CNTW. However, we have no information on the type of bonds created between the CNTs and the coating. Furthermore, all the samples broke at the end of the copper plating showing that there is probably a stress concentration at this point, leading to a point of weakness. Hence, it seems that partial copper coating is definitely not a good solution from a mechanical perspective. Solutions to improve the mechanical properties by fully coating the wire have been suggested [297], but not adopted in our case. The goal is to reduce the number of interactions with the beam. A coating with a high Z material would lead to an increase of beam interactions. Another alternative solution would be to use iron or tungsten coatings, since these elements are more soluble in carbon than copper and can form carbides, but this solution implies to

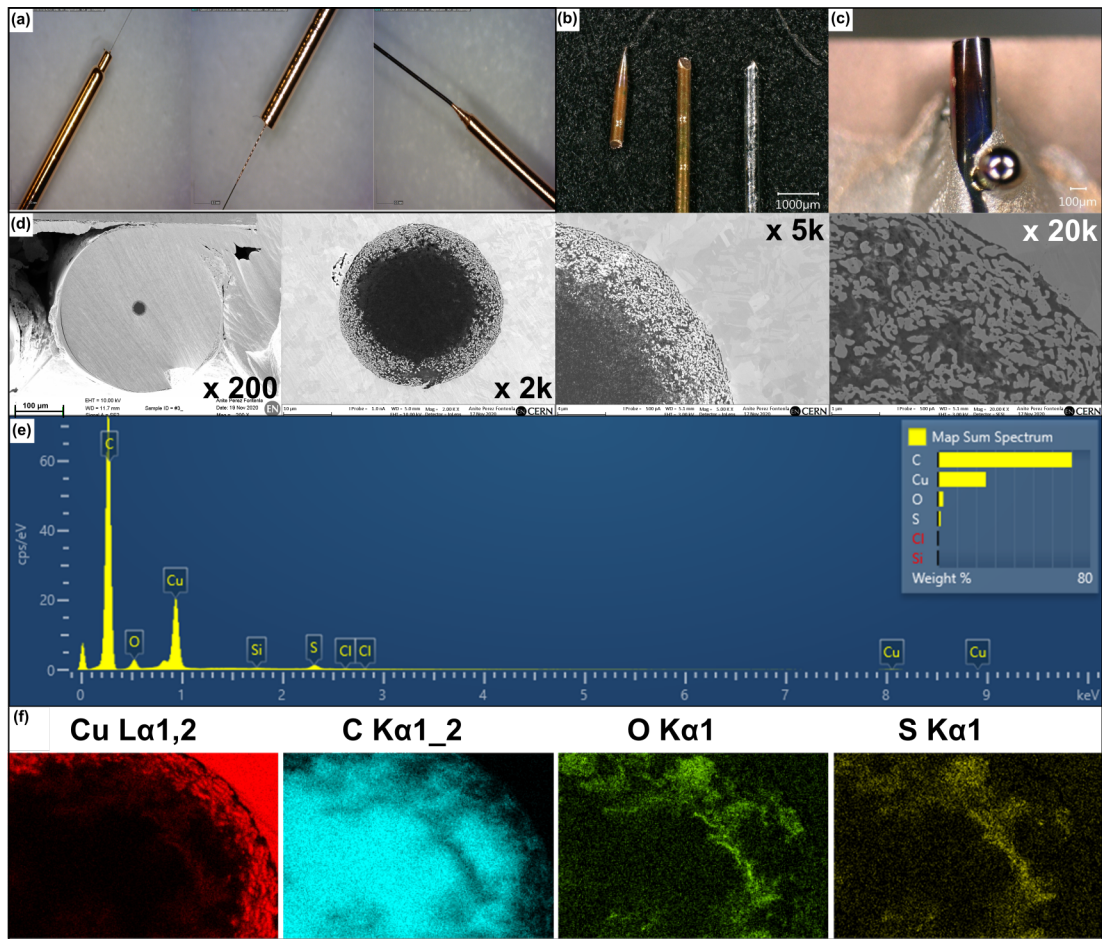


Figure 2.4: (a) Different shapes of copper-coating; (b) Extremities of three wires after soldering; (c) Samples after FIB milling; (d) Grey level SEM images of a cross-section (x200, x2000, x5000 and x20000). Copper appears grey while the wire appears black; (e) Energy-dispersive X-ray spectrum of a sample highlighting the presence of copper, carbon, oxygen and sulfur; (f) Maps of these four elements showing the presence of oxygen and sulfur in the transition region between the inner carbon wire and the outer copper-coating.

add significant mass, which could be a problem. Indeed, a local increase of mass implies larger deflections during the scan due to the acceleration.

Moreover, we saw that the reproducibility of the coating process is not very high and is depending on a lot of factors, such as the cleanliness of the tank, precision of the current going through the grid, but also the thickness and the width of the copper layer on the printed circuit board during the grid preparation. This is not acceptable for such a high precision project.

2.1.2.3 Mechanical analysis by Weibull method

2.1.2.3.1 Raw parameters calculation

The goal of this study is to find which kind of wire, with which mounting, could replace the existing carbon fibers used in the current WS. To do so, tensile tests have been performed on 93 samples, combining the two parameters of interest: diameter (10, 20 and 30 μm) and mounting conditioning (PF and CC). Labels and numbers of samples tested are given in the [table. 2.2](#).

Table 2.2: Labels and numbers of samples for each couple of parameters.

	PF	CC
10 μm	d10 _{PF} (15)	d10 _{CC} (6)
20 μm	d20 _{PF} (12)	d20 _{CC} (19)
30 μm	d30 _{PF} (16)	d30 _{CC} (25)

Typical raw tensile stress/strain curves show various behaviors for our samples depending on the diameter and conditioning ([Fig. 2.5](#)). The left axis of each graph indicates the load (in N) applied to the wire during the test while the right axis is for the corresponding stress (in GPa) calculated as load divided by the previously measured real cross-sections of the wires. For the CC samples, the diameter used to calculate the stress is also the diameter of the wire. We consider that the coating parts are part of the clamping system. Looking at the three PF figures corresponding to the three diameters ([Fig. 2.5.a.b.c](#)), we can see that the curves are quite similar since they all correspond to a brittle behavior of these non-coated CNTW, with a strain to failure about 2 %. There is a linear increase of the stress between 0 and 90 % of the strain to failure (ε_{max}) and a very short plastic zone before the failure. The behavior for the CC samples is more disparate. Samples from batch d10_{CC} more or less have the same brittle response as for the PF conditioning, whereas samples of d20_{CC} and d30_{CC} batches show a much larger discrepancy between samples, with larger intervals of variation than for the PF conditioning, since the strain at failure varies from 1.4 to 4 % and from 1.7 to 5 % for 20 and 30 μm respectively. The maximal load before the failure also shows large variations, from 140 to 357 mN and from 229 to 638 mN for 20 and 30 μm respectively, corresponding to maximal stresses smaller than in the PF and d10_{CC} cases.

Strain to failure (ε_{max}), average strength (σ_{max}), Young's modulus (E) and toughness (T) were evaluated from the raw curves of the [figure. 2.5](#).

Strain to failure

The average strain at failure (ε_{max}) ([Fig. 2.6.a](#)) is relatively constant for the PF conditioning for the three diameters, with 1.93 ± 0.54 % for 10 μm , 1.86 ± 0.21 % for 20 μm and 2.28 ± 0.34 % for 30 μm , corresponding to relative variations of 28 %, 11 % and 15 % respectively. For CC conditioning samples, both ε_{max} and its variations seem to increase approximately linearly with the diameter, with slopes of 0.84 and 0.86 respectively.

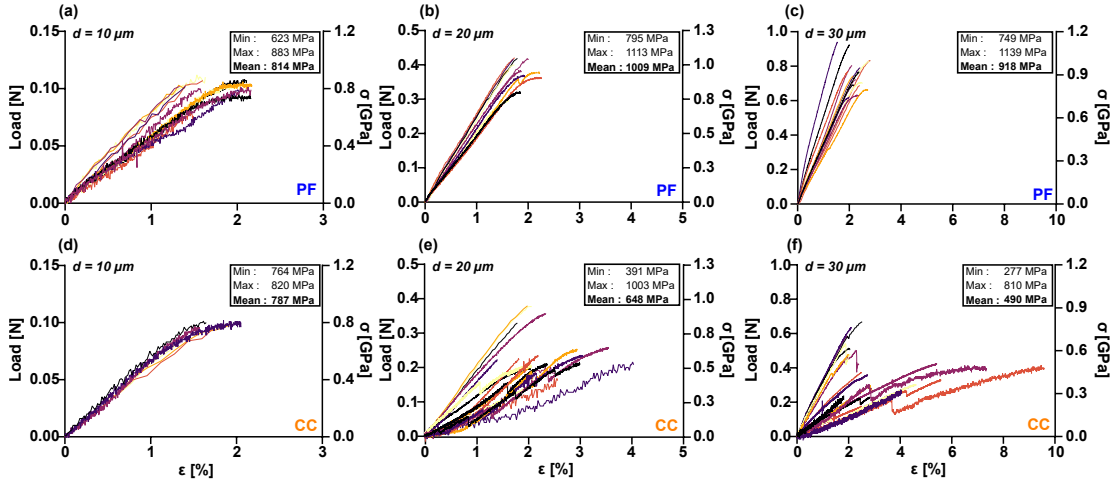


Figure 2.5: Typical stress / strain curves for the PF (a, b and c) and CC conditionings (d, e and f). For each of these conditionings, response of samples from three different diameters: 10 μm (a and d), 20 μm (b and e) and 30 μm (c and f) have been plotted.

Stress to failure

The average stress at failure (σ_{max}) for the raw data are shown in the figure. 2.6.b. Comparing the PF and CC conditionings, we can see that results for the 10 μm nominal diameter are the most balanced, with an average fracture stress of 814 ± 65 MPa for PF and 787 ± 22 MPa for CC. The difference between the average fracture stresses of d20_{PF} (1009 ± 102 MPa) and d20_{CC} (648 ± 178 MPa) or d30_{PF} (918 ± 114 MPa) and d30_{CC} (490 ± 164 MPa) is much larger and corresponds to a strong decrease of the fracture stress between the PF and the CC conditionings for these diameters.

Young's modulus

The Young's modulus (E) (Fig. 2.6.c) was calculated using the slope of a linear regression of the stress-strain curve between 0 and 1 % of strain. It is interesting to notice that for the wires with a nominal 10 μm diameter, the modulus is a larger for CC with 51 ± 3 GPa than for PF with 44 ± 14 GPa. For the diameters of 20 and 30 μm , it is the opposite, with a modulus for PF twice larger than CC, with 58 ± 7 and 26 ± 15 GPa and 43 ± 11 and 20 ± 12 GPa respectively. We can see that the relative uncertainty is much larger for the d20_{CC} and d30_{CC} batches than for the other batches, as already visually evident in the figure. 2.5.

Specific toughness

The specific toughness (T) expressed in J.g^{-1} quantifies the ability of a material to absorb energy by deforming elastically without breaking. It is defined as the area under the stress/strain curve up to the elastic limit, divided by the density. The average specific toughness (Fig. 2.6.d) for the d10_{PF} batch is 66 ± 18 J.g^{-1} , 78 ± 14 J.g^{-1} for d20_{PF} and 89 ± 16 J.g^{-1} for d30_{PF}. These data could be fitted by a linear increase with diameter of equation $T = 1.2d + 54.7$. The wires with the

CC conditioning show another behavior with a specific toughness quite similar for the three diameters, with 62 ± 12 , 59 ± 21 and 71 ± 44 J.g⁻¹ for d10_{CC}, d20_{CC} and d30_{CC} respectively. We note that for this conditioning, this is the standard deviation which increases linearly.

The raw data of the stress/strain curves for d10_{PF}, d20_{PF}, d30_{PF} and d10_{CC} batches show similar behaviors with a linear increase, then some leveling off or linear increase with a smaller slope and finally failure. However, the transition between the two linear regimes varies widely from a sample to another. For the d20_{CC} and d30_{CC} batches, some partial fractures appear for some samples and the leveling-off regime is rarely attained. Another difference between batches is that all PF samples broke in the middle of the wire while CC samples broke at the extremity of the copper-coating. This indicates that the coating plays a keyrole in the failure mechanism and that the assumption of an improvement due to the filling of the voids is definitely not confirmed. An explanation may be that the coating on both extremities over-constrains the outer layers. Under the tension, the external layers break first which induces the successive partial fails before the final failure. For smaller diameters, the coating could stress more homogeneously the wire which may lead to a single and abrupt failure.

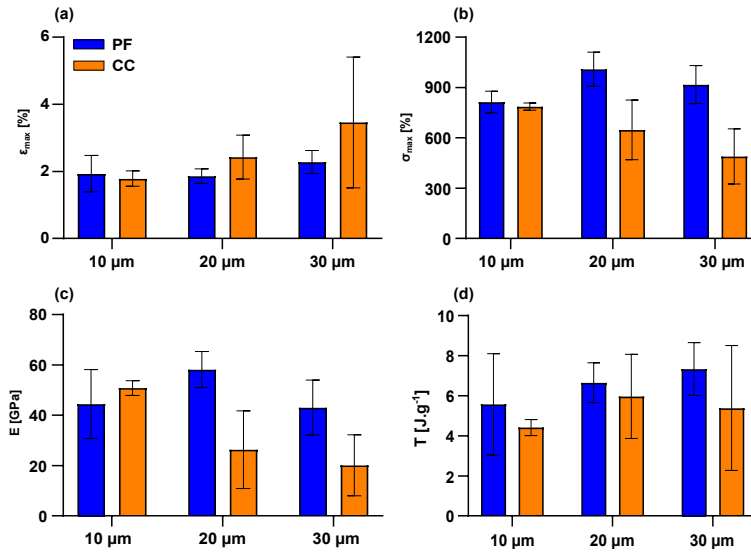


Figure 2.6: Mechanical parameters synthesis: (a) Strain to failure; (b) Stress to failure; (c) Young's modulus; (d) Specific toughness.

2.1.2.3.2 Weibull parameters calculation

For many years, scaling models for composite materials have been the subject of intense research. Empirical, phenomenological or progressive damage methods, all aim to optimize and improve the performances of these materials [298]. Our study also fits in this framework, with the idea of testing small-sized samples of short

CNTW and adapting the results to the longer wires used in wire scanners, using Weibull methods. Main tests are performed with small gauge length samples [212, 299] as limits on translational stroke of the tensile machine does not accommodate samples of length used in WS. This kind of analysis allows scaling the dispersion of the mechanical strength results without explicitly modeling the defects, using the hypothesis that a higher degree of similarity of defect type and uniformity results in a smaller dispersion of failure stresses. In this approach, the state of the material (failed or not) is represented by a random variable which is distributed according to a probability law that depends on the applied stress and can be experimentally identified by a series of tests (see e.g. [300, 301]). The probability of survival follows a Weibull distribution and is then expressed as [302, 303]:

$$P_s(\sigma) = \exp \left[-V^* \left(\frac{\sigma}{\sigma_0} \right)^m \right] \quad (2.1)$$

where σ is the applied stress, σ_0 , the scale parameter, and m the shape parameter (Weibull modulus). V^* is the reduced volume equal to the actual volume divided by a reference volume. First, to get the scale and shape parameters, we consider that the volume of reference is the volume of the sample, so that $V^* = 1$. In order to compute σ_0 and m , we first sort the samples by stress at failure (σ_{max}) from lowest to highest. Each sample then has an associated probability of survival that can be evaluated using its rank in the list (n_{rank}) with formulas that depend on the number of samples ($N_{samples}$). For moderate size samples [303]

$$P_s(\sigma_{max}) = 1 - \frac{n_{rank}(\sigma_{max}) - 0.3}{N_{samples} + 0.4} \quad (2.2)$$

Then (for $V^* = 1$), we can obtain m and σ_0 thanks to a linear fit using the following equation derived from Eq. 2.1

$$\ln(-\ln(P_s)) = m \ln(\sigma_{max}) - m \ln(\sigma_0) \quad (2.3)$$

Indeed, using a plot (Fig. 2.7) of $\ln(-\ln(P_s))$ as a function of $\ln(\sigma)$, the slope of the linear regression gives the modulus m and the Y-intercept y_0 gives $\sigma_0 = \exp(y_0/m)$. Results for m and σ_0 for the three diameters and two conditionings, are summarized in the ?? along with r^2 which characterizes the quality of fit.

For the 10 μm batches, Weibull moduli are 19.18 and 28.11 respectively, for PF and CC conditionings. This is quite high compared with the results for the 20 and 30 μm batches (between 3.05 and 8.25). This is normal since a mathematically known property of the Weibull modulus is that the lower m , the greater the dispersion of the variable characteristic of the failure. This has been seen for composite materials with fibers oriented at random, lower values of m corresponded to greater dispersions of the tensile strength. Hence, the high m for the low diameter indicates a defects distribution more homogeneous than for the larger diameters, which agrees with the previous observations of the packing.

The results for σ_0 show 6 % of difference between the PF and the CC conditionings for the diameter of 10 μm , while this difference increases up to 32 % for the 20 μm batches and 43 % for the 30 μm batches. The r^2 values indicate a fair quality of the linear fits, with a minimum of 0.88 for the d20_{CC} batch and a maximum of 0.96 for the d30_{PF} batch. In order to compare the new CNT wires with the existing CF wires, other tests have been performed with 8 samples of carbon fibers (as used in current WS) of diameter 34 μm and length 10 mm, with a PF conditioning only. Results for those tests are summarized in the last line of the ??, in *italic*. They give a modulus of 11.8 and a σ_0 of 928 MPa. With $r^2 = 0.93$, the fit is as good as for the CNT wires.

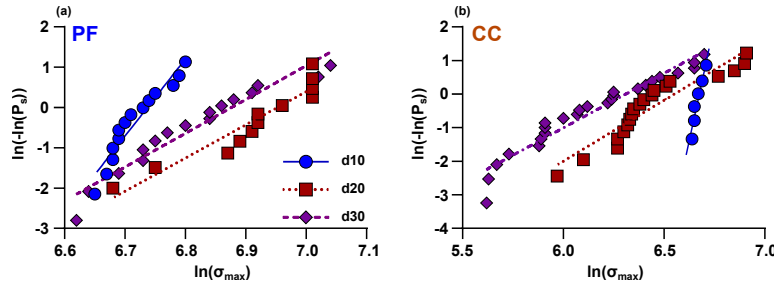


Figure 2.7: Linear fits of results of tensile tests using a Weibull failure probability law: (a) PF samples (10 mm gauge length); (b) CC samples (30 mm gauge length).

Table 2.3: Weibull parameters m and σ_0 and goodness of fit r^2 .

$\emptyset$	m		σ_0 [MPa]		r^2	
	PF	CC	PF	CC	PF	CC
10	19.8	28.1	843	793	0.92	0.91
20	8.3	3.6	1045	700	0.90	0.88
30	7.7	3.1	957	537	0.96	0.96
34	<i>11.8</i>	-	<i>928</i>	-	<i>0.93</i>	-

Our study for diameters from 10 to 30 μm , completes the study of Sugano et al. [296] in which 4 wires with diameters larger than 30 μm were used. They found a decrease of the shape parameter m from 9.4 to 3.1, and scale parameter σ_0 from 938 to 217 MPa with the increase of the diameter, which is coherent with our results.

2.1.2.3.3 Operational parameter calculation

The interest of this study is to verify if these new wires could be installed on operational WS. If so, the length of the wires will not be 12 or 30 mm as in the tests, but 150 mm. In order to extrapolate our experimental results to wires of 150 mm, Weibull analysis is a good approach, since the stress-strain curves showed a brittle behavior, sometimes with significant variations, which excludes using the classic sizing criteria used for ductile materials [304, 305, 306]. Furthermore, CNTWs

broke at maximal stresses (σ_{max}) which varied greatly from a sample to another, particularly for the d20_{CC} and d30_{CC} batches. This dispersion, probably due to the presence of defects in variable quantities and distributions, undermines the classical assumption of homogeneity of mechanical behavior.

To make this extrapolation, we used an equivalent reduced volume V^* expressed as $V^* = V_{op}/V_{sample}$, with V_{op} the volume of the operational wire and V_{sample} the volume of the samples used for the mechanical tests [307], either for PF or CC conditionings. Using Eq.2.1, we see that the larger the operational volume, the smaller the survival probability, which is normal since the number of defects is increased. In fact, we can also define $V^* = 150/l_{cond}$, since the wires in the future operational WS will have one of the diameters used during the tests and consequently the same cross-section as the CNTWs with that diameter. We therefore find $V_{PF}^* = 12.5$ for the PF conditioning (gauge length of 12 mm) and $V_{CC}^* = 5$ for the CC conditioning (gauge length of 30 mm). The problem is now to define a new characteristic strength σ_{0-op} lower than σ_0 if $V^* > 1$. Since the Weibull modulus m characterizes the inhomogeneity in the population of defects (the bigger m , the more homogeneous the population), it can be considered independent of the gauge length. In the test case ($V^* = 1$), $P_s(\sigma) = 1/e$ if $\sigma = \sigma_0$. Similarly, in the operational case ($V^* > 1$), we define σ_{0-op} such that $P_s(\sigma_{0-op}) = 1/e$. This implies that $V^*(\sigma_{0-op}/\sigma_0)^m = 1$, which gives

$$\sigma_{0-op} = \sigma_0 (V^*)^{-\frac{1}{m}} \quad (2.4)$$

Using the values of σ_0 and m from the ??, the new σ_{0-op} for the PF conditioning decreases by 12 % with respect to σ_0 for a diameter of 10 μm and by around 25 % for diameters of 20 and 30 μm . The values decrease much more for the CC conditioning and diameters of 20 and 30 μm , due to the combination of a larger reduced volume and smaller modulus, up to a 41 % decrease for the diameter 30 μm (Table. 2.4).

Table 2.4: Corrected σ_{0-op} for the calculation of the failure probability.

$\varnothing$	σ_{0-op} [MPa]	
	PF	CC
10	739 (-12 %)	749 (-6 %)
20	769 (-26 %)	449 (-36 %)
30	691 (-28 %)	317 (-41 %)
34	764 (-18 %)	-

The impact of the gauge length has also been studied by Zhang et al. on fibers composed of MWCNT of around 6-8 nm but they found that variations of the gauge length between 1 and 20 μm gave apparently randomly scattered results without any specific trend [257]. However, Wang et al., while studying CNTW transformed in rolled ribbons [168], found a large evolution of the curve

shape, with a linear increase followed by plastic deformation over a large range of deformations for 10 mm gauge length vs. almost no plastic deformation for 20 mm gauge length. Finally, the differences in strength and strain at failure were +17 % and +50 % respectively in favor of the 10 mm samples. This is slightly different with our observations, where the shape of the curves is very similar for the three diameters, with a sharp increase up to failure. As the gauge length is correlated with the copper coating, it is difficult to determine if the increase of the strain to failure of the CC conditioning is due to the gauge length or to the coating. It is particularly true if we consider the 10 μm where the variation is only 5 %.

2.1.2.3.4 Comparison of mechanical parameters

The Weibull survival probability distribution for a given stress, defined in Eq.2.1, is complementary to the probability of failure before that stress ($P_f(\sigma) = 1 - P_s(\sigma)$) which is itself the cumulative density function (CDF) corresponding to the following probability density function (PDF) (in the case $V^* = 1$):

$$f(\sigma) = \frac{m}{\sigma_0} \left(\frac{\sigma}{\sigma_0} \right)^{m-1} e^{-\left(\frac{\sigma}{\sigma_0} \right)^m} \quad (2.5)$$

On the [figure. 2.8](#), we plot the Weibull probability of failure before a given stress ($P_f(\sigma) = 1 - P_s(\sigma)$), first for the raw test data ([Fig. 2.8.a.b](#)) and then for the operational WS ([Fig. 2.8.c.d](#)) with a solid blue line for $d = 10 \mu\text{m}$, dotted orange line for $d = 20 \mu\text{m}$ and dashed purple line for $d = 30 \mu\text{m}$. We observe that both for the PF and CC conditionings, the transition from 0 to 1 is quite sharp for $d = 10 \mu\text{m}$ and smoother for the other two diameters. We also see that for the operational conditions, the difference between σ_0 and $\sigma_{0\text{-op}}$ shifts this transition to lower stresses, which is due to the higher number of defects in the increased volume. For the 10 μm wires, the shift is small (11 % and 5 % for PF and CC respectively), but much larger (46,36 %) and (28,41 %) for (PF, CC) and for $d = 20$ and 30 μm respectively. However, the slopes of the transition remain much smaller for $d = 20$ and 30 μm than for $d = 10 \mu\text{m}$, corresponding to a broader range of stress at failure, *i.e* an increased range of strengths for the two larger diameters.

The average strength can be computed from the PDF f defined by

$$\bar{\sigma} = \int_0^\infty \sigma f(\sigma) d\sigma = \sigma_0 \Gamma \left(1 + \frac{1}{m} \right) \quad (2.6)$$

where Γ is the gamma special function. Results for the average strengths are summarized in the [table. 2.5](#), along with values for other mechanical parameters that will be discussed later. We observe that considering the length of an operational WS, Weibull analysis show a reduction of the average strength decreasing by around 10% with respect to the lengths used for the mechanical tests, for $d = 10 \mu\text{m}$, for both conditions. For the diameters 20 and 30 μm , the variation is much higher with 28 % for $d_{20\text{PF}}$, and up to 40 % for $d_{30\text{CC}}$. For comparison, we note that $\bar{\sigma}$ varies by only 20 % for the $d = 34 \mu\text{m}$ CF.

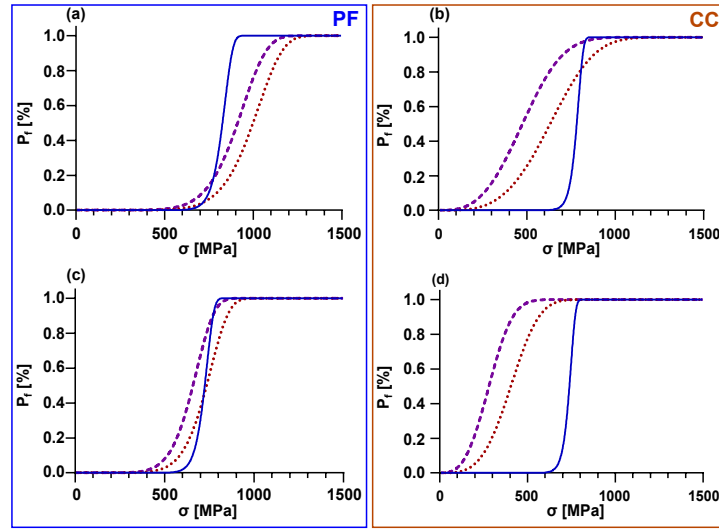


Figure 2.8: Probability of failure for a specific applied stress for: (a) PF conditioning; (b) CC conditionings for $d = 10 \mu\text{m}$ (continuous blue line), $d = 20 \mu\text{m}$ (dotted red line) and $d = 30 \mu\text{m}$ (purple dashed line) for the raw test data and (c and d) for the operational WS.

Table 2.5: Strain to failure (ε_{max}), maximum stress at failure (σ_{max}), average strength in the test conditions ($\bar{\sigma}_{raw}$, calculated using σ_0), average strength for operational WS ($\bar{\sigma}_{op}$, calculated using σ_{0-op}), Young's modulus (E) and toughness (T), first for the CNTWs with $d = 10, 20$ and $30 \mu\text{m}$, then for the $34 \mu\text{m}$ CF (only the strengths in that case).

Conditioning		10 μm PF	20 μm PF	30 μm PF	34 μm
ε_{max}	[%]	1.9 ± 0.5	1.9 ± 0.2	2.3 ± 0.3	-
σ_{max}	[MPa]	814 ± 65	1009 ± 102	918 ± 114	909 ± 60
$\bar{\sigma}_{raw}$	[MPa]	820 ± 53	986 ± 142	900 ± 138	889 ± 91
$\bar{\sigma}_{op}$	[MPa]	719 ± 46	725 ± 105	650 ± 99	731 ± 75
E	[GPa]	44 ± 14	58 ± 7	43 ± 11	-
T	[J.g ⁻¹]	5.6 ± 2.5	6.7 ± 1.0	7.3 ± 1.3	-
Conditioning		CC	CC	CC	
ε_{max}	[%]	1.8 ± 0.2	2.4 ± 0.7	3.5 ± 1.9	
σ_{max}	[MPa]	787 ± 22	648 ± 178	490 ± 164	
$\bar{\sigma}_{raw}$	[MPa]	778 ± 35	631 ± 193	480 ± 172	
$\bar{\sigma}_{op}$	[MPa]	735 ± 33	405 ± 124	293 ± 101	
E	[GPa]	51 ± 3	26 ± 15	20 ± 12	
T	[J.g ⁻¹]	4.4 ± 0.4	6.0 ± 2.1	5.4 ± 3.1	

The mechanisms increasing the strength of the wire for the packing factor is mainly the reduction of the distance between CNTs and CNT bundles. Stronger bonds are created at a nano-scale for individual tubes and at a micro-scale for

bundles with increasing the shear strength. This is coherent with the results of Vilatela et al. [198] where he reported the increase of wire strength with the increase of the CNT length and so, the surfacic area in contact with other tubes. Moreover a high packing factor implies a higher probability to create entanglements between individual tubes and bundles [308].

Liu et al. observed a decrease of the strength by 45 % for 20 mm long CNTWs of 25 μm in diameter with the increase of the twist angle from 12 to 38° [235]. An optimum twist angle of 25° has been observed for wires with diameters of around 8 μm composed of 200 μm long MWCNTs of 10 to 15 nm [307]. This mechanism of a higher packing factor could explain the increase of the strength and the Young's modulus between 10 and 20 μm .

2.1.2.3.5 Probability of failure

Considering the results from the Weibull analysis, we can evaluate the probability of failure for a stress of 327 MPa, for each wire diameter and both PF and CC conditionings. The table 2.6 summarizes the results before and after the σ_0 correction. We observe that for $d = 10 \mu\text{m}$, both for the raw test data and for the operational WS, the probability of failure is quite small for both conditionings, but lower for the CC conditioning than for the PF conditionings. The behavior is partly different for $d = 20$ and $30 \mu\text{m}$, with an increase of the failure probability P_f for the CC conditioning with respect to the PF one. However, we still have an increase of P_f for operational WS, with respect to the shorter wires used for the tests. P_f reaches 67 % for $d = 30 \mu\text{m}$ in CC conditioning after the σ_0 correction. To have an idea of what level of probability is acceptable, we note that the current 34 μm CF have a probability of failure of $4.5 \cdot 10^{-6}$ for the CF used during the mechanical tests and $4.5 \cdot 10^{-5}$ for the operational wires.

Table 2.6: Probabilities of failure for a stress of 327 MPa, corresponding to a load of 30 g applied on a 34 μm CF wire during the installation.

d	$P_f (\sigma = 327 \text{ MPa})$			
	Raw data		Op WS	
	PF	CC	PF	CC
10	$7.2 \cdot 10^{-09}$	$1.5 \cdot 10^{-11}$	$9.7 \cdot 10^{-08}$	$7.7 \cdot 10^{-11}$
20	$6.5 \cdot 10^{-05}$	$6.3 \cdot 10^{-02}$	$8.3 \cdot 10^{-04}$	$2.7 \cdot 10^{-01}$
30	$2.6 \cdot 10^{-04}$	$1.9 \cdot 10^{-01}$	$3.1 \cdot 10^{-03}$	$6.7 \cdot 10^{-01}$
34	$4.5e-06$	-	$4.5e-05$	-

2.1.3 Influence of the loading speed

In addition to the mechanical tests, we carried out electron microscopy analyzes in order to observe the structural modifications and better understand the fracture mechanisms due to the different tensile parameters. Among the factors studied, we varied the tensile speeds (0.01, 1 and 10 mm.min⁻¹) for three samples of 30 μ m in diameter.

The figure. 2.9 shows the fracture zones for the three samples tested at speeds of 0.1, 1 and 10 mm.min⁻¹. We observe that for the lowest speed (Fig. 2.9.a), the fracture zone is elongated with a fracture angle of 45°. The longitudinal section of the zone shows a volume 11 μ m high and 40 μ m long, void of material, as if it had been torn away (SOI-1). A zoom on the outer layer of the cut zone shows a fracture initiator 500 nm thick and 20 μ m long, located 2 μ m under the surface.

At high speed (Fig. 2.9.c), the fracture zone is clear, similar to a brittle fracture, with no CNT floating on the surface, a sign of tearing. The yarn shows a slight detachment of the superficial fibers with local bumps. The longitudinal section shows a globally homogeneous structure, with a small incipient fracture (SOI-1) and a less dense zone (SOI-2).

The sample tested at $v_l = 1$ mm.min⁻¹ shows an intermediate state between low and high speed (Fig. 2.9.b).

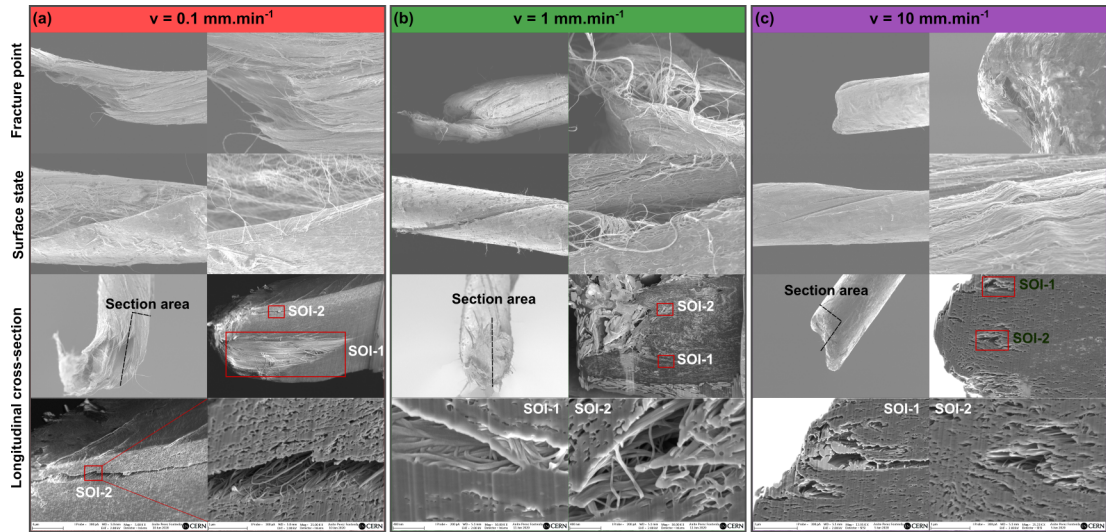


Figure 2.9: SEM pictures of the fracture area and longitudinal cross-section of 30 μ m CNT wires tested at speed: (a) 0.1 mm.min⁻¹; (b) 1 mm.min⁻¹ and (c) 10 mm.min⁻¹.

These observations highlight that in our case, with these samples, the speed of traction plays a role in the structural modification of the wire. A low tensile speed allows the entire wire to deform by sliding of the CNT bundles relative to each other leading to super aligned bundles. The fracture then occurs in a zone containing predispositions such as incipient cracks or zones of fragility with larger spacings between the twisted fibers.

For high pulling speeds, the bundles do not have time to reorganize. The entangled bundles are abruptly released leading to hard breaking, higher strength and lower strain to failure (Fig. 2.10).

Despite these observations under the microscope, the analysis of the results of the loads at failure and the strains to failure do not show any characteristic effects due to changes in tensile speed. If for wires of 10 μm we observe a decrease in strain to failure of 13 % with increasing speed, the large standard deviation does not allow a definitive conclusion on a speed effect. Concerning the samples of 20 μm , one observes an increase in the maximum deformation for the intermediate speed of 20 % then a return to the initial value for the highest speed. The 30 μm samples show an equivalent maximum strain for the low speed and the intermediate speed with a value of 2.2 % and an increase for the highest speed at 2.4 % (Fig. 2.10.a).

Regarding the breaking loads, a slight increase of 6 % is observed for the 10 μm wires and greater increases for the 20 and 30 μm wires with 30 and 13 % respectively (Fig. 2.10.b).

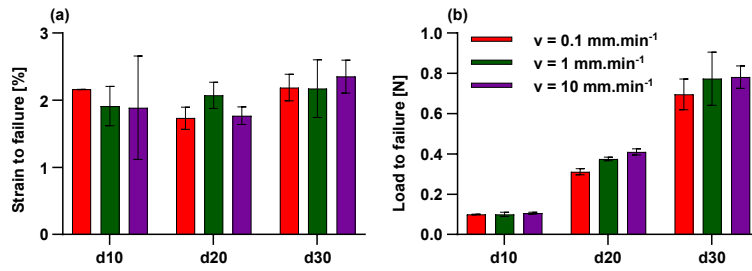


Figure 2.10: (a) Load to failure and (b) Strain to failure for CNT wires of diameters 10 μm , 20 μm and 30 μm for load speeds of 0.1 mm.min^{-1} ; 1 mm.min^{-1} and 10 mm.min^{-1} .

The mechanical results obtained experimentally do not show any effect of tensile speed on the structure of the wire. Some studies [257, 309] show a direct relation of the tensile speed on the improvement of the mechanical properties, specifically on the maximum load at failure and the Young's modulus while the strain to failure decreases. The curves include three different stages corresponding to a linear deformation at the beginning, a non-linear with a slow slope in the middle and a final sharp break. The ultimate strength, Young's modulus and fracture strain are a lot dependent on the strain rate. These three parameters have two different regimes: a viscoelasticity behavior for low strain rate (0.006 mm.min^{-1} to 0.6 mm.s^{-1}) corresponding to a sliding-to-break mechanism and an elastic one at higher strain rate (0.6 mm.s^{-1} to 60 mm.min^{-1}) corresponding to a "cascade-like" break [257].

However, these results are disputed by other studies showing effects on the mechanical properties due to the speed of traction only at very high speeds.

2.2 PRELIMINARY ANALYSIS

In this section, we introduce the mechanical characteristics interesting for our study: the number of points of measurement for the resolution and the maximal deflection for the accuracy. We start with the basic case of stationary state, considering a constant angular velocity for the existing wire situation, a twisted wire of 12 fibers of $7\text{ }\mu\text{m}$ in diameter of density 1.7 g.cm^{-3} and a single strand of $34\text{ }\mu\text{m}$ of density 2.1 g.cm^{-3} . Then, we compare the CF34 with the CNT wires considering the results found in the [section 2.1](#).

2.2.1 Number of points per sigma

As mentioned in the first chapter, the increase in luminosity targeted by current CERN projects is accompanied by an increase in intensity as well as energy and a decrease in beam sizes. The combination of small beam sizes and high intensities leads to very high beam densities. The instrumentation of these beams is a real challenge for interceptive instruments.

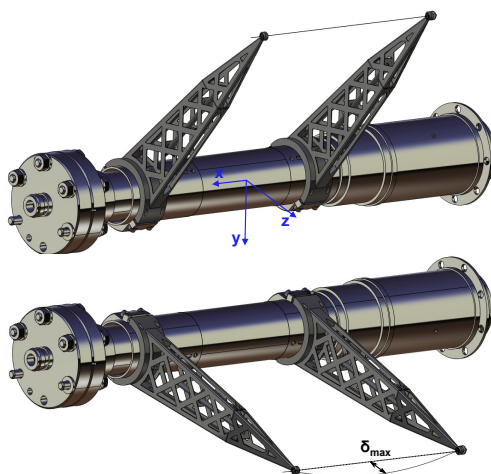


Figure 2.11: Illustration of the wire fly and deflection during a scan.

Today, wire-scanners are used to scan the beam and measure the transverse profile. In order to reconstruct the Gaussian profile, enough measurement points (therefore beam wire interactions) are necessary. It is customary to define 3 points per sigma (where sigma is the characteristic size of the beam, defined as the standard deviation of a Gaussian profile) as a sufficient number. For some conditions, we are facing a dilemma between an increase of the speed required to avoid an overheating, and a decrease of the speed to get enough measuring points. As an example, currently, a measurement of the beam with linear wire-scanners in the LHC is limited to 8 bunches out of 2808 possible, meaning that a full intensity beam cannot be measured.

The distance between two measuring points during a scan is

$$d_p = v_{scan} t_{rev} \quad (2.7)$$

with v_{scan} the scan speed, expressed in $[m.s^{-1}]$ and t_{rev} the revolution time of the beam, specific for each accelerator (Table. 1.4).

The number of points per sigma is then

$$n_{pps} = \frac{\sigma_b}{d_p} \quad (2.8)$$

with σ_b , the beam size.

Considering minimum beam sizes of the conditions defined in the table. 1.4, *ie*: 1.60 mm for the PSB, 0.80 mm for the PS, 0.20 mm for the SPS and 0.16 mm for the LHC, we obtain the number of points per sigma for scan speeds from 1 $m.s^{-1}$ to 20 $m.s^{-1}$. It clearly appears that for the PSB and PS accelerators, the beam sizes are large enough so that at full speed (20 $m.s^{-1}$), the condition of 3 points per sigma is respected. On the other hand, we note that for the SPS the maximum limit for scanning the smallest beam is 2.9 $m.s^{-1}$ and 0.6 $m.s^{-1}$ for the LHC (Fig. 2.12.a).

If we now study the scan speed as a function of the beam size in a range defined by the minimum and maximum possible sizes, we notice that for the SPS, a high speed scan can be performed for a beam of 1.39 mm, which corresponds to a factor of 7 compared to the minimum possible sigma (0.20 mm). Concerning the LHC, the maximum admissible speed for a measurement of the largest beam (1.19 mm) is 4.5 $m.s^{-1}$ and 0.6 $m.s^{-1}$ for the smallest beam (0.16 mm) (Fig. 2.12.b).

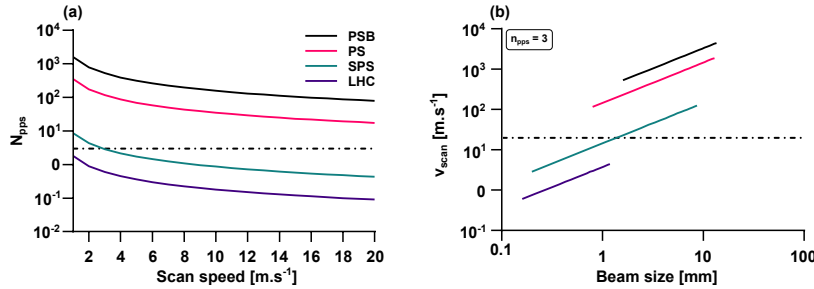


Figure 2.12: (a) Number of points per sigma as a function of the scan speed for the circular accelerators and the LHC for a beam size of 1.60 mm for the PSB, 0.80 mm for the PS, 0.20 mm for the SPS and 0.16 mm for the LHC; (b) Speed of the scan required to get 3 points per sigma as a function of the beam size.

The conclusion of this analysis is that the new rotary wire-scanners can operate at a nominal speed of 20 $m.s^{-1}$ for the whole range of beam sizes for the PSB and the PS. Regarding the SPS, for a high-speed scan, the beam size must be greater or equal to 1.4 mm. This beam size cannot be achieved for the maximum energy of 450 GeV. Finally, for the LHC (in which the new rotating wire-scanners are not installed), the speeds that can be used over the whole range of beam sizes are between 0.6 and 4.5 $m.s^{-1}$. The current use of linear wire-scanner is done at

a speed of 1 m.s^{-1} , thereby limiting the total intensity of the beam that can be measured. The use of the new wire-scanners could be useful in providing better measurement precision, but not for the possibility of high-speed scanning.

2.2.2 Deformation for a stationary state

Particle beams at CERN take different configurations depending on the accelerator and the users. As seen above (Table. 1.4), in the SPS, a beam, circulating at a frequency of 43375 Hz, is composed of 4 batches of protons of $1.9 \mu\text{s}$ length, spaced by 120 ns. Those batches are themselves made from 72 bunches of 1.5 ns length spaced by 25 ns. Each bunch is composed of about 10^{11} protons and has a two-dimensional gaussian distribution in the plane perpendicular to the axis of the beam. For each scan cycle, there is an acceleration of the wire during 15 ms, a flat top at the nominal speed (110 rad.s^{-1} for the fastest one, 20 m.s^{-1} for the linear speed) during 10 ms (during which the data acquisition is performed) and a decrease of the speed during 15 ms. This cycle is repeated a second time after a delay time of 150 ms for the scan out.

Consequently the wire is crossed by bunches of protons, several times during the acquisition time. Since it is desirable to reduce the number of crossings in order to preserve the wire from irradiation damages, the wire speed has been increased in the new WS. However, the wire now undergoing a higher rotary acceleration, deflects and moves dynamically more from its nominal position. The accuracy of the WS measurement is thus reduced by the uncertainty on the wire position due to its vibrations. The goal is then to minimize δ , the difference between the theoretical and real position (Fig. 2.11), by increasing the pre-tension force during the installation of the wire in the WS, so that it deflects less. This requires a wire material with a high tensile strength and resilience with respect to irradiation damages and thermal load induced by the energy deposited by protons.

The question of the optimal material to be used for the wire in the CERN WS has already been extensively studied. Materials such as beryllium [40] or carbon fibers [51] (already used at CERN for WS) or tungsten [42] (not used for fast WS) were considered. These studies have been extended for the mechanical aspect to other materials (copper, iron and titanium) [58]. Our goal in this study was to assess whether the CNTWs could stand the pre-tension imposed to reduce the uncertainty on the wire position due to its vibrations during the scan.

As seen in the state of the art, the wire is fixed on the forks. The length of the wire (L) depends on the model of the instrument. For the new generation of wire-scanners, the rotary WS, the length is 120 mm for the PSB and 150 mm for the PS and SPS. The length of the forks (R), which defines the distance between the wire and the rotation axis is 153 mm for the PSB and 183 mm for PS and SPS. So far, the configuration of the wire for the PSB and the PS is a twisted wire composed of 12 wires of $7 \mu\text{m}$ (Table. 2.7). The equivalent diameter of this wire can be estimated as:

$$d_{eq} = \sqrt{n_y} d_y \quad (2.9)$$

where n_y is the number of wires composing the wire and d_y the diameter of the wire. For the PSB and the PS, this leads to an equivalent diameter of 24 μm .

Table 2.7: New fast wire-scanner configuration.

Ring		PSB	PS	SPS
Wire configuration		$12 \times 7 \mu\text{m}$	$12 \times 7 \mu\text{m}$	$1 \times 34 \mu\text{m}$
Equivalent diameter	[μm]	24	24	34
Wire length	[mm]	120	153	153
Forks length	[mm]	150	183	183

During the scan, the wire will deform under the different constraints due to the acceleration of the shaft. We name deflection the variation of the wire position during the movement with respect to the position at the equilibrium state. To analyze the deflection of the wire during the scan, we model with a linear wave equation for a vibrating string submitted to a force corresponding to the centrifugal force produced by a rotation of angular frequency ω .

The equation of motion for the wire in the rotating frame is

$$\lambda \frac{\partial^2 y(x, t)}{\partial t^2} = F_p \frac{\partial^2 y(x, t)}{\partial x^2} + \lambda(R + y(x, t))\omega^2 \quad (2.10)$$

Boundary conditions

$$y(x = 0, t) = 0$$

$$y(x = L, t) = 0$$

Initial conditions

$$y(x, t_0) = f(t_0)$$

$$\frac{\partial y}{\partial t}(x, t_0) = f'(t_0)$$

with λ the linear density of the wire ($\lambda = \rho \times S$) and F_p the pre-tension load applied to the wire during the assembly. We make appear this parameter (F_p) because this is the one set and controlled during the installation.

The [equation. \(2.10\)](#) describes the general motions of the wire. We will focus on the two main states interesting for our concern: the stationary case and the dynamic case.

In the stationary state, the acceleration is null. The equation can then be expressed as

$$F_p \frac{\partial^2 y(x)}{\partial x^2} + \lambda(R + y(x))\omega^2 = 0 \quad (2.11)$$

with R the distance between the wire in the equilibrium state and the rotation axis (also the length of the forks) and ω the angular velocity in [rad.s^{-1}] where $\omega = v_{\text{scan}}/R$.

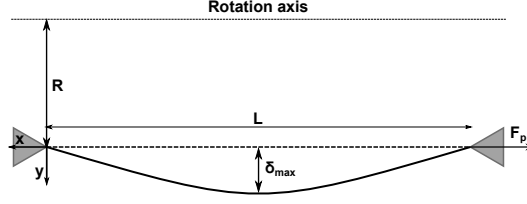


Figure 2.13: Wire deflection model for a stationary state.

Defining $\alpha^2 = \frac{\lambda}{F_p} \omega^2$, we obtain an inhomogeneous linear ordinary differential equation with constant coefficients that can be solved explicitly in terms of trigonometric functions

$$\frac{\partial^2 y(x)}{\partial x^2} + \alpha^2 y(x) = -\alpha^2 R \quad (2.12)$$

The solution can be written explicitly as

$$y_{stat}(x) = 2R \frac{\sin(\frac{\alpha x}{2}) \sin(\frac{\alpha(L-x)}{2})}{\cos(\frac{\alpha L}{2})} \quad (2.13)$$

And the maximum deflection, located at the center of the wire ($L/2$), is

$$\delta_{max} = 2R \frac{\sin^2(\frac{\alpha L}{4})}{\cos(\frac{\alpha L}{2})} \quad (2.14)$$

Since the available information on the material is the density, we can make appear this parameter as well as the stress inside the wire with

$$\alpha = \omega \sqrt{\frac{\lambda}{F_p}} = \omega \sqrt{\frac{\rho S}{F_p}} = \omega \sqrt{\frac{\rho}{\sigma}} \quad (2.15)$$

Doing that, we remark that the maximal deformation is only dependent on the angular speed, the density and the stress.

We start the analysis with the existing configurations, *ie*: with a twisted wire of 12 fibers of 7 μm in diameter and a single fiber of 34 μm . So far, the applied preload did not change and is set to 0.30 N.

From a previous mechanical test study (Hal Bigland's master thesis), CF7 shows a scale factor σ_0 from the Weibull analysis ([table. 2.3](#)) of 2.8 GPa and a shape factor m of 5.18. A new scale factor can be calculated to take in consideration the real length of the wire compared with the length of the sample during the test. The operational scale factor is explicitated with the [equation. \(2.4\)](#) and the strength of this fiber can be then estimated with the [equation. \(2.6\)](#), leading to $\sigma_{0-op} = 1.66$ GPa and $\bar{\sigma} = 1.53$ GPa.

Applying a preload of 0.30 N on the wires, leads to stresses of 663 and 330 MPa for the twisted wire of $d = 24$ μm and the mono-fiber of $d = 34$ μm respectively. These values of stress are well below the strength of the fibers.

In this first approach, we only consider the carbon fibers (CF24 and CF34) currently used in the WS (and not the CNT wires) and assuming a density of 1.7 and 2.1 g.cm⁻³ for the CF24 and CF34 respectively, we can model the shape of the deflection for scans in the SPS with limit linear speeds of 1 and 20 m.s⁻¹ corresponding to $\omega = 6$ and 110 rad.s⁻¹ (Fig. 2.14).

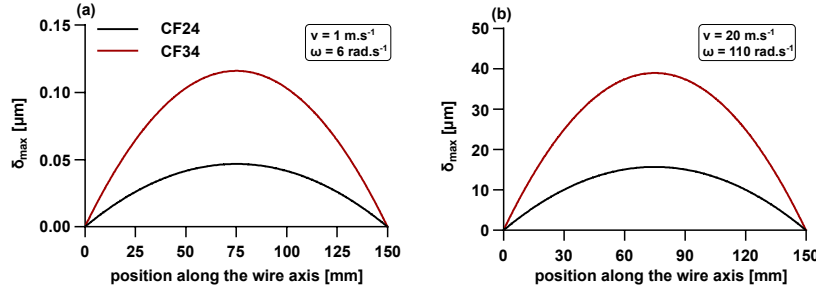


Figure 2.14: Deflection shape of the wires CF24 and CF34 for two different speeds: (a) 1 m.s⁻¹ and (b) 20 m.s⁻¹ for the couple of conditions (d, ρ) , with $d = 24$ and 34 μm , $\rho = 1.7$ and 2.1 g.cm⁻³ with a preload of 0.3 N, corresponding to the preload set for wires on WS, in the SPS.

At low speed (Fig. 2.14.a) we observe that the deformations are very small, with 0.05 and 0.11 μm for the CF24 and CF34 wires respectively. By multiplying the speed by 20 (Fig. 2.14.b), the maximal deformation is multiplied by 320 for the CF24 wire and 355 for the CF34 wire with 16 μm and 39 μm respectively at high speed.

This observation shows the importance of the scan speed. Hence taking into account the specifications required and the cases considered, we will favor low speeds if possible.

On the figure. 2.15.a, we plot the maximal deformation at the center of the wire as a function of the stress for the maximal speed in the SPS (Fig. 2.15.a). We logically observe a decrease of the maximal deflection with the increase of the applied stress. The curves are hierarchically sorted driven by the density. The deformation of the wire CF24 varies by a factor 7.5 for an increase of the stress by a factor 6.9. Concerning the wire CF7 and CF24, it varies by a factor 3.2 for an increase of the stress by a factor 3.3.

The range between the minimal and maximal deflection, taken for pre-loads between 250 MPa and 90 % of the ultimate tensile strength (657 MPa and 1347 MPa for CF24 and CF34 respectively), is similar for both wires with 62 μm .

The figure. 2.15.b shows the evolution of the maximal deformation as a function of the scan speed for the PSB, PS and SPS, for a preload of 0.3 N. The deformation is lower for the PSB, since the length of the wire as well as the length of the forks are shorter (Table. 2.7). For the higher speed, the difference between the PSB and the SPS for both wires is an increase of 30 %.

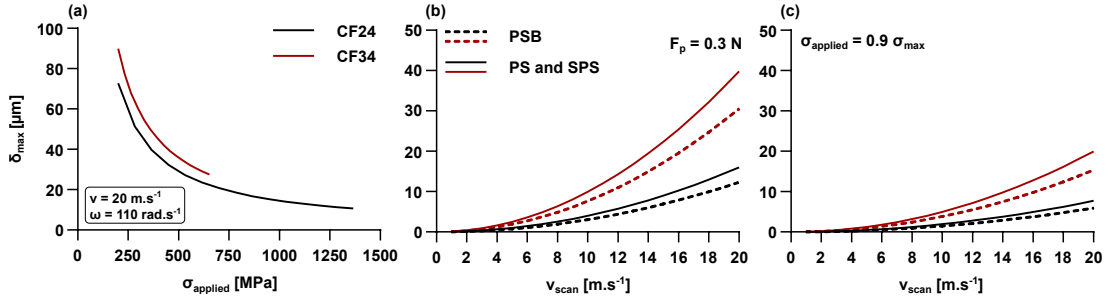


Figure 2.15: (a) Maximal deflection at the center of the wire as a function of the stress applied during the wire installation for a speed of 20 m.s^{-1} for both wires, CF24 (black) and CF34 (red); (b) Maximal deflection as a function of the scan speed for a preload of 0.3 N for the PSB (dashed line) and the PS and the SPS (continuous line); (c) Maximal deflection as a function of the scan speed for an applied stress equal to 90 % of the strength of each wire.

As seen previously, the application of a greater stress decreases the maximum deflection of the wire (Fig. 2.15.a). By applying a stress equal to 90 % of the maximum resistance (keeping a safety coefficient of 10 %) of the wires, the maximum deformation for the CF34 is 2.5 times more than for the CF24, with at the highest speed (20 m.s^{-1}), 5.9 μm and 15.3 μm for the PSB and 7.7 μm and 20.0 μm for the PS and the SPS (Fig. 2.15.c).

Table 2.8: Summary of the deformations in the middle of the wire for different applied stresses.

F_p [N]	Maximal deformation [μm]			
	Low speed		High speed	
	CF24	CF34	CF24	CF34
0.3	0.04	0.09	16	40
max	0.02	0.05	8	20

We have thus seen that rotation of the wire around an axis generates a deformation of this wire, the shape of which depends on geometric parameters (length of the wire and distance from the wire to the axis of rotation), on material parameters (density and stress applied during installation) and scan speed.

The deformation of the wire during the passage through the beam generates uncertainties on its exact position and therefore uncertainties on the measured profile. It has been seen that the faster the scan, the greater the maximum deformation (located in the center of the wire). However, we want to minimize this deformation as much as possible in order to meet the specifications required for each instrument, typically around 10 μm [34]. It can be seen that today, under current conditions, CF34 carbon wires do not allow the specification of 10 μm maximum

deformation to be met at 20 m.s^{-1} , neither for the SPS, nor for the PSB. Scans should be limited to speeds of 14 m.s^{-1} for the SPS and 16 m.s^{-1} for the PSB for a preload correspond to a stress equal to 90 % of the ultimate tensile strength.

This preliminary study highlights the need for a new material which, from a mechanical point of view, would allow the new wire-scanners to meet the specifications and to be able to use them at their maximum speed in order to reduce the risk of overheats during beam interaction.

2.2.3 Comparison with CNT wires

For existing designs, a load of 0.3 N is applied during assembly, corresponding to a stress of 327 MPa on current carbon fiber of $34 \text{ }\mu\text{m}$. Since the installation procedures are sensitive, all the changes in the accelerator complex require in-depth studies. That is why nowadays the load has not been increased. The result for δ_{max} gives us an opportunity to evaluate the influence of the pre-load.

Considering the mechanical parameters results obtained with experimental tests, the maximal deflection is therefore plotted as a function of the pre-load for the CNTWs for diameters 10, 20 and $30 \text{ }\mu\text{m}$ (density of 1.3 g.cm^{-3} , cf: [section 2.1.1.1](#)) and for the CF of diameter $34 \text{ }\mu\text{m}$ (density of 2.1 g.cm^{-3}) ([Fig. 2.16.a](#)). For each sample, the range of the load is between 1 g and the maximum pre-load considering 90 % of the strength for the PF conditioning (647 MPa, 653 MPa, 585 MPa and 657 MPa for the CNT10, CNT20, CNT30 and CF34 respectively). We observe a decrease by a factor 5, 20, 41 and 60 the maximal deflection between 1 g and the maximal pre-load for each sample. Looking at the 30 g pre-load, only the CNT30 and CF34 can reach this value. The maximal deflection is $22 \text{ }\mu\text{m}$ and $40 \text{ }\mu\text{m}$ respectively.

As already said, in the configuration currently used, we apply a pre-load of 30 g corresponding to an applied stress of 327 MPa. Using this applied stress, $\rho = 1.3 \text{ g.cm}^{-3}$ and $\omega = 110 \text{ rad.s}^{-1}$ in the [equation. \(2.14\)](#) and the [equation. \(2.15\)](#), we get the same $\delta_{max} = 25 \text{ }\mu\text{m}$ for the CNTWs independently of the diameter, whereas using $\rho = 2.1 \text{ g.cm}^{-3}$ for the CF, we get $\delta_{max} = 39 \text{ }\mu\text{m}$ for the CF ([Fig. 2.16.b](#)).

Alternatively, if we consider a preload stress equal to the previously calculated operational strength σ_{max} with a safety coefficient of 10 %, we can calculate the maximal deflection for each diameter and conditioning ([Fig. 2.16.c](#)). Similarly to what we found for the operational strengths, we can see that the maximal deflection is roughly the same for the three diameters when using the PF conditioning, with 12, 12 and $13 \text{ }\mu\text{m}$ for the CNTWs. However, when using the CC conditioning with CNTW of diameters 20 or $30 \text{ }\mu\text{m}$, the deflection is much higher than for the current CF. These later conditions are therefore not adequate to get a better precision on the measurement of the beam profile, than the current CF, contrarily to PF conditioning for the three diameters or for CNT10_{CC}.

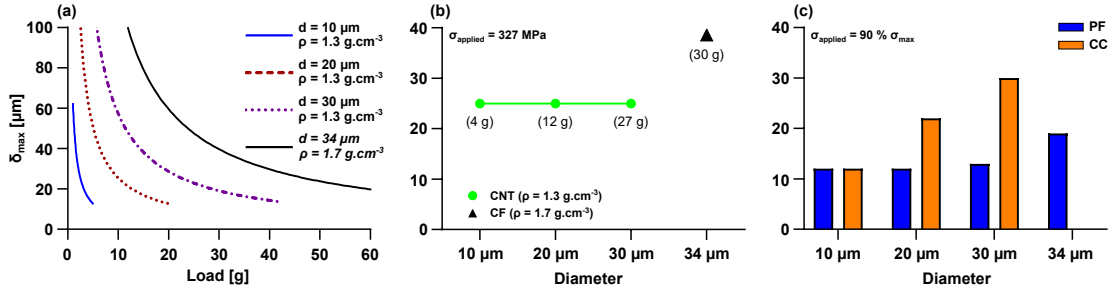


Figure 2.16: Maximal deflection at the center of the wire using a stationary state model: (a) As a function of the preload for CNTWs of 10, 20 and 30 μm with density of 1.3 g.cm^{-3} and a CF with a diameter of 34 μm and density of 1.7 g.cm^{-3} with maximal strengths calculated for PF conditioning; (b) For an applied stress of 327 MPa; (c) considering an applied stress of 90 % of the operational strength corresponding to a given couple (diameter, conditioning).

This analysis shows the importance of a high preload stress during the installation in order to decrease the maximal deflection and thus increase precision of the beam profile measurement, with respect to the current CF. In our simplified model, the important quantity is the stress and not the force (in α). Hence, thinking in terms of load, as usually done, can be misleading. The comparison of the performances of the CNTWs with different diameters, for a same stress, thus seems to be more appropriate in our case. For a constant stress of 327 MPa, we observe a decrease of 25 % of the maximal deflection with respect to the current CF, for the CNTWs with $d = 10$ and 20 μm in the PF conditioning. The copper-coating seems to degrade the properties leading to a δ_{max} twice and three times larger for the 20 and 30 μm CNTWs than for the CF. However, these results have to be considered carefully, keeping in mind that the real density could be different as the one used for the calculation, and that our model relies on assumptions.

2.2.4 Comparison with other models

Previous works [310] studied the deformation of the wire, using the elementary cable theory and treating the wire as a cable fixed on its extremities. Assuming that the wire is subjected to a uniform transverse loading w defined as

$$w = \frac{\rho \pi d^2}{4} R \omega \quad (2.16)$$

with ρ the density, R the length of the forks and ω the angular velocity, the maximal deformation is given by the relation

$$\delta_{max} = \frac{\rho v_{scan}^2 L^2}{8 R \sigma} \quad (2.17)$$

with L the length of the wire, v_{scan} the linear velocity and σ the axial stress.

To compare the difference of δ_{max} between our model and this model, we plot $\text{Diff} = y_{max} - \delta_{max}$ for low speed (1 m.s^{-1}) and high speed (20 m.s^{-1}) for the three densities of interest (1.3 g.cm^{-3} , 1.7 g.cm^{-3} and 2.1 g.cm^{-3}) (Fig. 2.17).

We observe a larger difference for larger density. Still considering a preload of 327 MPa, the difference decreases by 61 % ($4.66.10^{-8}$ to $1.79.10^{-8}$ for $v_{scan} = 1 \text{ m.s}^{-1}$ and 0.0075 to 0.0029 μm for $v_{scan} = 20 \text{ m.s}^{-1}$) for a decrease by 38 % of the density (from 2.1 g.cm^{-3} to 1.3 g.cm^{-3}). It is also interesting to notice that the difference decreases widely with the increase of the stress.

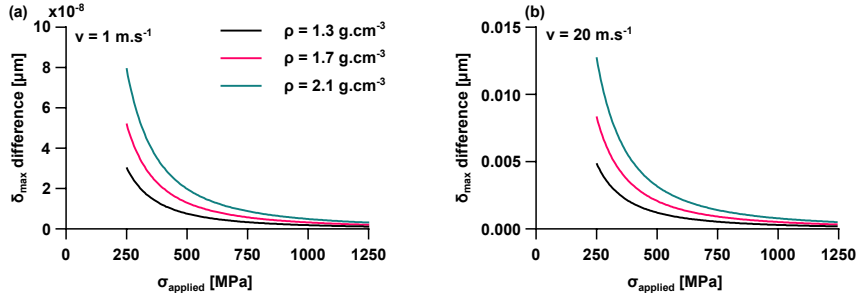


Figure 2.17: Difference of δ_{max} as a function of the stress in the SPS for densities of 1.3 g.cm^{-3} , 1.7 g.cm^{-3} and 2.1 g.cm^{-3} between our stationary model and the approximation model from Bigland et al. [310] for a linear velocity of (a) 1 m.s^{-1} and (b) 20 m.s^{-1} .

This comparison of models shows the interest of using this simplified model, specifically when using low velocities and high stresses. This easy calculation allows to get a good approximation of the maximal deflection for a stationary approximation.

2.3 WIRE DEFORMATION FOR A DYNAMIC MODEL

2.3.1 Vibrating states in regimes with constant angular velocity

If the maximum deformation of the wire considering simplified stationary models has been widely studied and used, the dynamic study has not, to my knowledge, been the subject of research. This is why in this section we detail the mathematical model to extract the explicit solutions.

In practice, the wire does not rotate at a constant speed, because the geometry and design of the instrument (presence of a cable welded to the end of the fork) does not allow it to rotate more than one revolution. The wire is therefore subjected to rapid acceleration allowing the wire to wait for its flight speed during which it crosses the beam before decelerating and ending up in the "in" position, at 180° from its initial "home" position (Fig. 2.18).

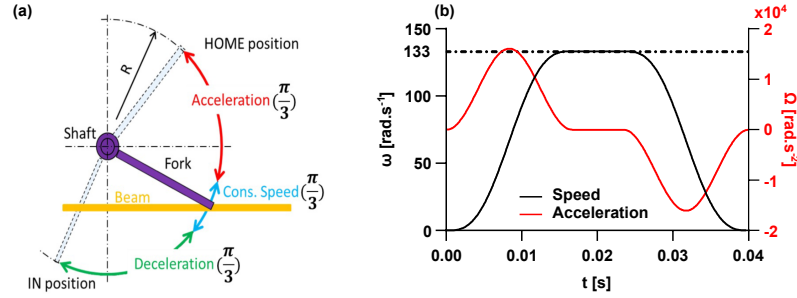


Figure 2.18: (a) Scheme of a scan profile: acceleration over an angle of $\pi/3$, constant speed during the passage through the beam followed by a deceleration; (b) Theoretical acceleration and speed profile for a scan at 20 m.s^{-1} in the PSB.

Now we consider a regime where the angular speed ω is constant. The general equation is then

$$\frac{\partial^2 y(x, t)}{\partial t^2} = \frac{F_p}{\lambda} \frac{\partial^2 y(x, t)}{\partial x^2} + y(x, t)\omega^2 + R\omega^2 \quad (2.18)$$

$$y(x = 0, t) = 0$$

$$y(x = L, t) = 0$$

The general solution of the equation of motion can be written as:

$$y(x, t) = y_{stat}(x) + h(x, t) \quad (2.19)$$

where $y_{stat}(x)$ is the stationary solution (which is unique for $\omega < \frac{1}{L}\sqrt{\frac{F_p}{\lambda\pi}}$), defined in the [equation. \(2.13\)](#), and $h(x, t)$ is the general solution of the homogeneous equation

$$\frac{\partial^2 h(x, t)}{\partial t^2} = \frac{F_p}{\lambda} \frac{\partial^2 h(x, t)}{\partial x^2} + \omega^2 h(x, t) \quad (2.20)$$

$$\begin{aligned}
h(x=0, t) &= 0 \\
h(x=L, t) &= 0 \\
h(x, t=0) &= -y_{stat}(x) \\
\frac{\partial h}{\partial t}(x, t=0) &= 0
\end{aligned}$$

Defining $\varphi_n(x) = \sin\left(\frac{n\pi x}{L}\right)$ the eigenfunctions of

$$-\frac{\partial^2 \varphi_n(x)}{\partial x^2} = \lambda_n^2 \varphi_n(x) \quad (2.21)$$

with λ_n the eigenvalues defined by

$$\lambda_n = \frac{n\pi}{L} \quad (2.22)$$

φ_n is an orthogonal basis of the space of functions that satisfies the boundary conditions

$$\begin{aligned}
\varphi_n(x=0) &= 0 \\
\varphi_n(x=L) &= 0
\end{aligned}$$

Thus the solution $h(x, t)$ of the [equation. \(2.20\)](#) can be written as

$$h(x, t) = \sum_{n=1}^{\infty} h_n(t) \varphi_n(x) \quad (2.23)$$

$$h(x, t) = \sum_{n=1}^{\infty} h_n(t) \sin\left(\frac{n\pi x}{L}\right) \quad (2.24)$$

The equation for the coefficients $h_n(t)$ is obtained by inserting the [equation. \(2.23\)](#) into the [equation. \(2.20\)](#) leading to

$$\sum_{n=1}^{\infty} \frac{\partial^2}{\partial t^2} h_n(t) \varphi_n = \sum_{n=1}^{\infty} h_n(t) \left(\frac{F_p}{\lambda} \frac{\partial^2 \varphi_n(x)}{\partial x^2} + \omega^2 \varphi_n(x) \right) \quad (2.25)$$

which can also be written with the [equation. \(2.21\)](#) as

$$\sum_{n=1}^{\infty} \frac{\partial^2 h_n(t)}{\partial t^2} \varphi_n = \sum_{n=1}^{\infty} h_n(t) \left(-\frac{F_p}{\lambda} \lambda_n^2 + \omega^2 \right) \varphi_n(x) \quad (2.26)$$

Leading to

$$\sum_{n=1}^{\infty} \left(\frac{\partial^2 h_n(t)}{\partial t^2} + \left(\frac{F_p}{\lambda} \lambda_n^2 - \omega^2 \right) h_n(t) \right) \varphi_n(x) = 0 \quad (2.27)$$

Since eigenfunctions are linearly independent and orthogonal, this implies that

$$\frac{\partial^2 h_n(t)}{\partial t^2} + \left(\frac{F_p}{\lambda} \lambda_n^2 - \omega^2 \right) h_n(t) = 0 \quad (2.28)$$

and thus

$$\frac{\partial^2 h_n(t)}{\partial t^2} = \left(\omega^2 - \frac{F_p}{\lambda} \lambda_n^2 \right) h_n(t) \quad (2.29)$$

$$\frac{\partial^2 h_n(t)}{\partial t^2} = -\omega_n^2 h_n(t) \quad (2.30)$$

Hence the solutions for $h_n(t)$ can be written as

$$h_n(t) = A_n \cos(\omega_n t + \psi_n) \quad (2.31)$$

Furthermore, the initial conditions

$$\frac{\partial h_n}{\partial t}(t=0) = 0.$$

leads us to take $\psi_n = 0$ and

$$h_n(t) = A_n \cos(\omega_n t) \quad (2.32)$$

with

$$\omega_n = \sqrt{\frac{F_p}{\lambda} \lambda_n^2 - \omega^2} \quad (2.33)$$

Inserting the [equation. \(2.32\)](#) into the [equation. \(2.24\)](#), this leads to

$$h(x, t) = \sum_{n=1}^{\infty} A_n \cos(\omega_n t) \sin\left(\frac{n\pi x}{L}\right) \quad (2.34)$$

Considering the initial conditions

$$h(x, t=0) = -y_{stat}(x)$$

$$\frac{\partial h}{\partial t}(x, t=0) = 0$$

We obtain the coefficients A_n for $t = 0$, which gives

$$-y_{stat} = \sum_{n=1}^{\infty} A_n \sin\left(\frac{n\pi x}{L}\right) \quad (2.35)$$

indicating that the coefficients A_n are the Fourier sinus series coefficient of the stationary solution y_{stat} that are written as

$$A_n = -\frac{2}{L} \int_0^L \sin\left(\frac{n\pi x}{L}\right) y_{stat}(x) dx \quad (2.36)$$

They can be calculated explicitly using the equation. (2.13)

$$\frac{2}{L} \int_0^L \sin\left(n \frac{\pi x}{L}\right) \sin\left(\frac{\alpha x}{2}\right) \sin\left(\frac{\alpha(L-x)}{2}\right) dx = \frac{2\alpha^2 L^2 \cos\left(\frac{\alpha L}{2}\right) \sin^2\left(\frac{n\pi}{2}\right)}{-\alpha^2 L^2 n\pi + n^3 \pi^3} \quad (2.37)$$

which leads to

$$A_n = -\frac{4R\alpha^2 L^2 \sin^2\left(\frac{n\pi}{2}\right)}{n^3 \pi^3 - \alpha^2 L^2 n\pi} \quad (2.38)$$

Finally, the global solution is

$$y(x, t) = y_{stat} + \sum_{n=1}^{\infty} A_n \cos(\omega_n t) \sin\left(\frac{n\pi x}{L}\right) \quad (2.39)$$

with

$$y_{stat}(x) = 2R \frac{\sin\left(\frac{\alpha x}{2}\right) \sin\left(\frac{\alpha(L-x)}{2}\right)}{\cos\left(\frac{\alpha L}{2}\right)} \quad (2.40)$$

$$\alpha = \omega \sqrt{\frac{\lambda}{F_p}} \quad (2.41)$$

$$\omega_n = \sqrt{\left(\frac{F_p}{\lambda} \frac{n^2 \pi^2}{L^2}\right) - \omega^2}, \quad n = 1, 2, 3... \quad (2.42)$$

We observe that the Fourier coefficients A_n decrease as $1/n^3$ for large n . We can thus expect that one can obtain a good precision truncating the sum with a small number of terms. The frequencies ω_n that determine the time evolution are independent of R , meaning that the only geometrical parameter matters for this consideration is the length of the wire (distance between the forks). In the case of a time dependent $\omega(t)$ one can not use the Fourier technique, since in the equation of motion there is a product of two time dependent functions, which after Fourier transformation become a convolution product, and the ensuing equations cannot be easily solved analytically.

For the analysis coming next, we consider three different type of accelerations: a sudden switching corresponding to a Dirac pulse where the speed goes from 0 to 110 rad.s⁻¹ immediately, a linear switching where the speed increases linearly from 0 to the nominal speed, and finally a real speed profile.

2.3.2 Response specific speed profiles

2.3.2.1 Sudden switching

The study of different velocity profiles makes it possible to estimate which profile is the most adequate to minimize the deformations during the scans. For the following, we focus only on an SPS configuration, with variation of different

parameters (d_w , ρ , F_p and ω_{max}).

We first consider a sudden switch of the speed from 0 to 110 rad.s⁻¹ by a pulse profile (Fig. 2.19.a).

We consider the input parameters described above, with a preload set up at 90 % of the stress to failure for each sample using the strength determined in the paper, for the paper frame (PF) conditioning and the copper coating (CC) conditioning. For the 7 μ m carbon fiber of density 1.7 g.cm⁻³, the strength is taken as 1530 MPa (from previous studies). Two preloads are considered on the 34 μ m carbon of density 2.1 g.cm⁻³ representing first the theoretical load we could applied (0.6 N) and second the load currently applied on the instrument (0.3 N). The input parameters are synthesized in the table. 2.9. Since the permissible preload for the CNT10 wire is the same in both cases (PF and CC), we will study only one of them.

Table 2.9: Summary of the input parameters used for the dynamic simulations.

d	[μ m]	7	10		20		24	30		34	
	-		PF	CC	PF	CC		PF	CC	θ	used
ρ	[g.cm ⁻³]	1.7		1.3		1.3	1.7		1.3		2.1
σ_{max}	[MPa]	1530	719	735	725	405	1530	650	293	731	-
$0.9\sigma_{max}$	[MPa]	1377	647	662	653	365	1377	585	264	657	-
F_p	[N]	0.05	0.05	0.05	0.20	0.11	0.62	0.41	0.19	0.60	0.30

To compute and analyze the deformation of the wire during the scan, we solved the equation (Eq. (2.18)) numerically with a leap frog formula for the time (t) and a Chebyshev spectral differentiation along the axis of the wire (x) (inspired from Trefethen, program 18, p 80 [311]).

We plot the dynamic response of the wire to a sudden switch of the speed from 0 to 110 rad.s⁻¹ considering a preload based on the PF results for the CNTWs wires and on a theoretical allowable preload for the CF34 (Fig. 2.19.b) and based on the CC results for the CNT wires and the current applied preload for the CF34 (Fig. 2.19.c). The red line represents the maximum deformation of the wire's centre in its stationary state. The blue line represents the oscillations of the wire when this one is submitted to a sharp increase of the velocity. We can observe that the amplitude is very large, almost the double of the maximal deformation in the stationary state. This maximum value in stationary state becomes a mean value around which the wire oscillates. We can also observe some residual oscillations when the speed decreases to 0. In this case, we do not include any damping coefficient, considering the time scale. This implies that residual oscillations do not decrease. Consequently, we are in a worse case.

The results show vibration amplitudes varying significantly depending on the case. It is observed that the greater the deformation of the adiabatic case, the greater the amplitude of the vibrations. The CF24 sample shows the weakest vibrations with an average of the adiabatic case at maximum speed, the deformation

is $6.3 \mu\text{m}$ and the vibrations oscillate between 0 and $12.5 \mu\text{m}$. During the stopped phase, the oscillations continue and are $-6 \mu\text{m}$ and $+6 \mu\text{m}$. The amplitude is therefore the same in both phases. For the CF34 sample, on the other hand, oscillations are observed in the stop phase twice as large as during the scan phase.

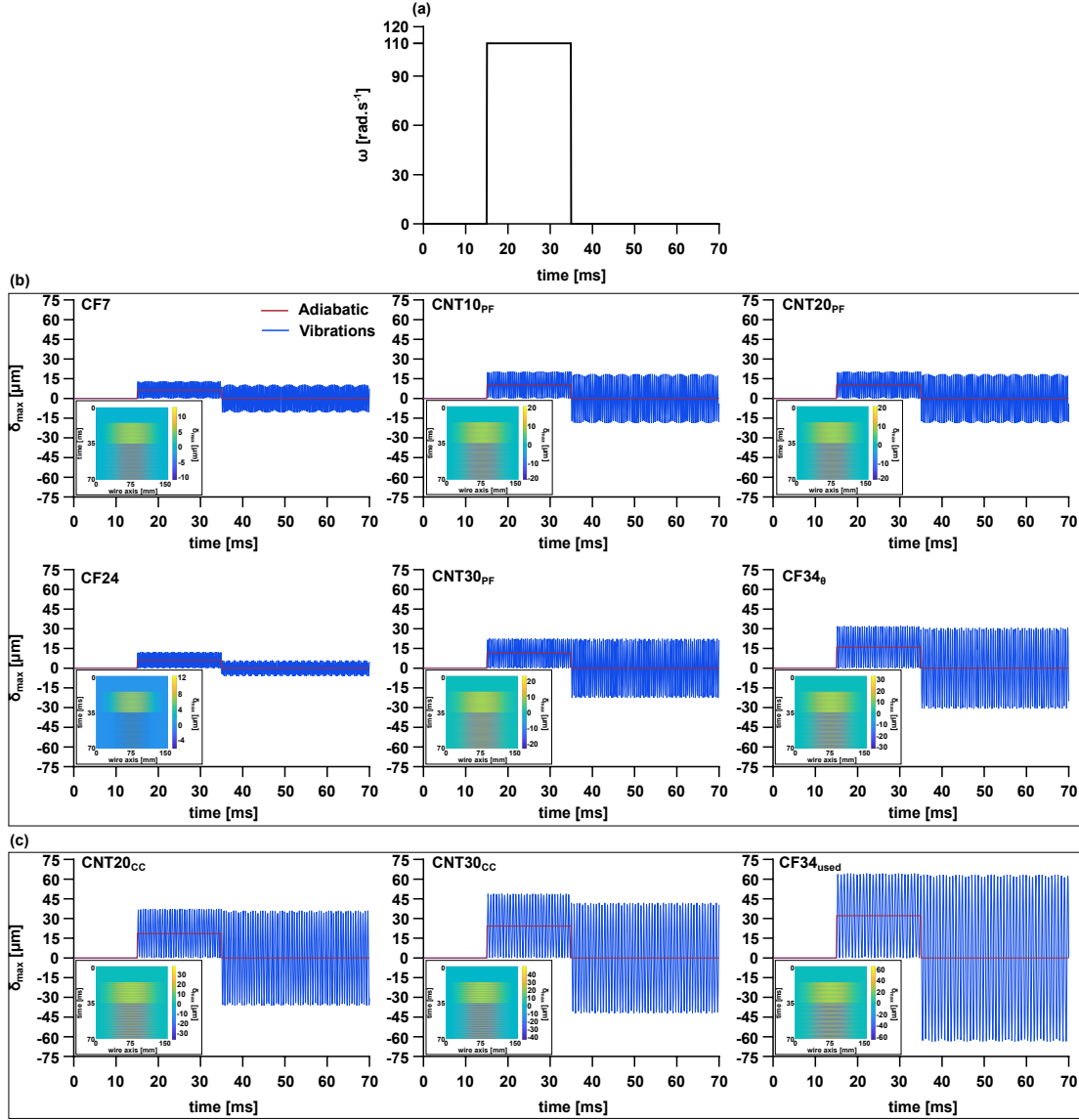


Figure 2.19: (a) Speed profile of the wire for a sudden switch between 0 and 110 rad.s^{-1} . Dynamic response of the wire after a sudden switch. The red line represents the maximum deformation for the adiabatic regime and the blue line the response considering the dynamic equation; (b) Preload applied for the CNTWs based on the the PF conditioning and on the theoretical maximal load we could apply on the CF34_θ; (c) preload applied for the CNTWs based on the CC conditioning and on the current load applied during the installation on the instrument for the CF34_{used}.

This model is clearly not optimal but interesting to look at to compare with the other different speed profiles.

2.3.2.2 Ramp switching

A model closer to reality is to consider a ramp speed profile, from 0 to ω_{max} , so as to have a constant acceleration. Hence we take

$$\omega(t) = 0 \quad \text{for } t \leq t_{rs} \quad (2.43)$$

$$\omega(t) = \frac{(t - t_{rs})}{(t_{ps} - t_{rs})} \omega_{max} \quad \text{for } t \in [t_{rs}, t_{ps}] \quad (2.44)$$

$$\omega(t) = \omega_{max} \quad \text{for } t \in [t_{ps}, t_{pe}] \quad (2.45)$$

$$\omega(t) = 1 - \frac{(t - t_{ce})}{(t_{ce} - t_{pe})} \omega_{max} \quad \text{for } t \in [t_{pe}, t_{ce}] \quad (2.46)$$

$$\omega(t) = 0 \quad \text{for } t \geq t_{ce} \quad (2.47)$$

$$(2.48)$$

with t_{rs} the time ramp start (0 s), t_{ps} the time of the plateau start ($15 \cdot 10^{-3}$ s), t_{pe} the time of the plateau end ($35 \cdot 10^{-3}$ s) and t_{ce} the time of the cycle end ($50 \cdot 10^{-3}$ s).

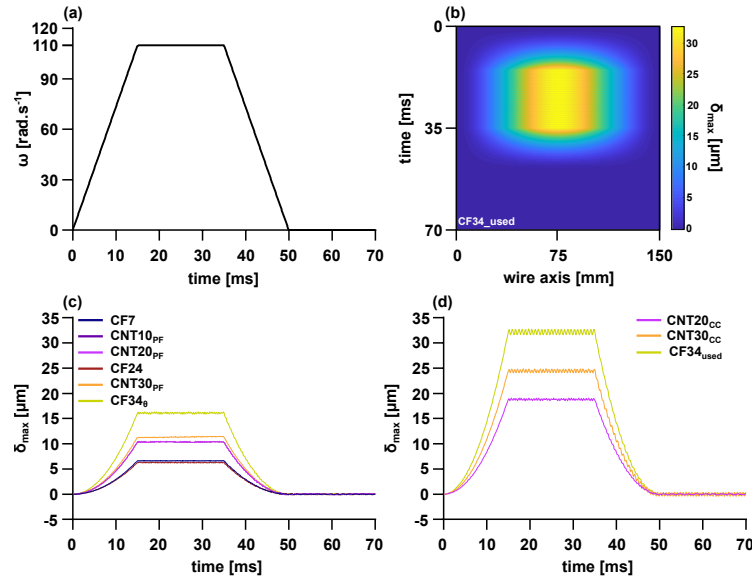


Figure 2.20: (a) Dynamic response of the wire after a ramp speed profile depicted; (b) Typical deformation of the wire as a function of the time and the position along the axis of the wire. Here is shown the response for the CF34_{used}; (c) Response in the middle of the wire for the PF and theoretical conditioning; (d) response in the middle of the wire for the CC and current setup conditioning.

The speed profile is plotted in the figure. 2.20.a. The dynamic profile for a ramping velocity profile is drastically different. The figure. 2.20.b shows the strain response along the wire and as a function of time for the CF34_{used} wire. We observe a homogeneous deformation during the scan with its maximum in the center of the wire. We also note that the general deformation profile is the same for all wires,

only the maximum values and oscillations vary, with a minimum for CF7 and CF24 with a deformation of 6.6 μm (Fig. 2.20.c) and a maximum for the CF34 θ whose preload is 0.3 N with 32.2 μm (Fig. 2.20.d).

It is observed that the greater the maximum deformation, the greater the oscillations around this equilibrium position. However, these oscillations remain much lower (between 31.6 and 32.7 μm) than those obtained with the previous velocity profile.

The analysis of the adiabatic regime, which is the maximal deformation of the stationary state for instantaneous angular velocities, allows to conclude that in this regime, the wire does almost not vibrate (compared with the sudden switching case).

We see above that the speed profile has a great influence on the amplitude of the vibrations. A slower increase of the speed up to the constant speed ω leads to smaller vibrations. Moreover, with a sudden increase and decrease of the speed, the amplitude of the vibrations during the rest time is higher than during the passage through the beam. With a ramp increase and decrease, the wire returns to its equilibrium state without vibration.

2.3.2.3 Real profile

To improve the accuracy of the model, we took a real speed profile obtained during a calibration campaign for an instrument installed in the SPS. The calibration of each instrument makes it possible to characterize the projected angular movement which varies from instrument to another due to assembly tolerances. It also allows to determine the uncertainty of the position of the wire in a controlled environment, and finally allows to test different speed profiles to minimize the vibrations of the system and the wire. To do this, a laser and a photodiode placed in opposition are moved vertically. At each position of the laser, 3 scans are performed. When the wire passes between the laser and the photodiode, the signal is cut and we can therefore know the real position of the wire and compare it with its theoretical position. This position is measured by optical sensors which read a number of slits engraved on an optical disc and whose distance between each is known precisely.

By recovering the actual velocity profile, we obtain a Gaussian fit with 3 terms for the scan in and 8 terms for the scan out (Fig. 2.21.a). The fit function can be then explicitated as

$$f(t) = \sum_{i=1}^n a_i e^{-\left(\frac{t-b_i}{c_i}\right)^2} \quad (2.49)$$

The parameters a_i , b_i and c_i for the scan in and out are given in the table. 2.10. The very good fit obtained using this method is represented by a r-squared of 0.9956 for the scan in and 0.9960 for the scan out.

Table 2.10: Parameters of the Gaussian fit for the scan in and the scan out with t in second and ω in rad.s^{-1} .

	Scan in			Scan out		
	1	2	3	1	2	3
a	61.28	83.33	43.92	81.45	72.61	88.93
b	3.68e-2	2.64e-2	1.53e-2	3.83e-1	3.91e-1	4.01e-1
c	6.89e-3	1.05e-2	7.49e-3	7.33e-3	7.46e-3	8.37e-3

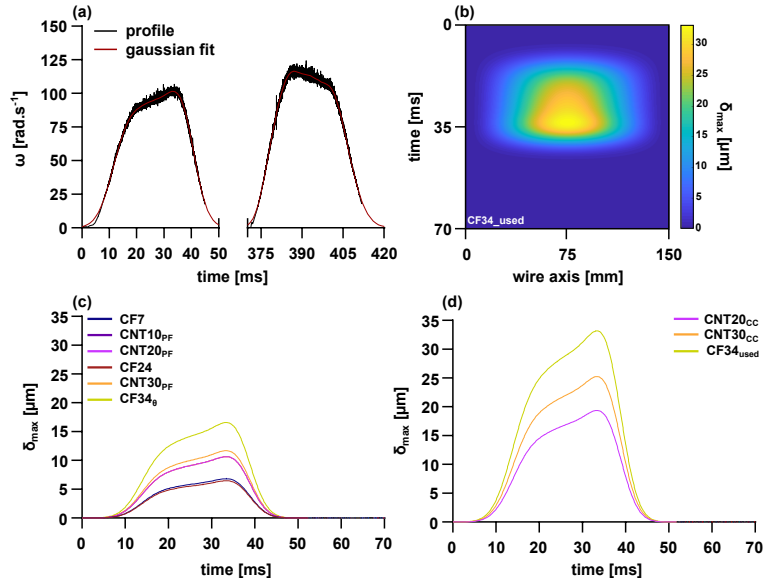


Figure 2.21: (a) Real speed profile measured during the calibration (black line) and gaussian fit (red line); (b) Typical deformation of the wire as a function of the time and the position along the axis of the wire. Here is shown the response for the CF34_{used}. Dynamic response of the wire for the real speed profile: (c) Response in the middle of the wire for the PF and theoretical conditioning; (d) Response in the middle for the CC and current setup conditioning.

The dynamic response shows the same hierarchy for the real speed profile as for the other profiles, with a minimum of deformation for the wire of 7 μm despite a higher density, thanks to its very high mechanical resistance. Considering the applied preloads, the CNT10_{PF} and CNT20_{PF} wires follow with a maximum deformation of 10.7 μm . The CNT30_{PF} wire is quite similar with a maximum deformation of 11.7 μm . The CF34_θ wire shows the greatest deformation with a maximum at 16.6 μm (Fig. 2.21.a). Considering lower preloads due to copper plating, the deformations increase drastically with values of 19.4, 25. and 33.2 μm for the CNT20_{CC}, CNT30_{CC} and the CF34_{used} (Fig. 2.21.b). Moreover, the mean value of the deformation over the entire scan in increases by a factor of 2 for the wires considered.

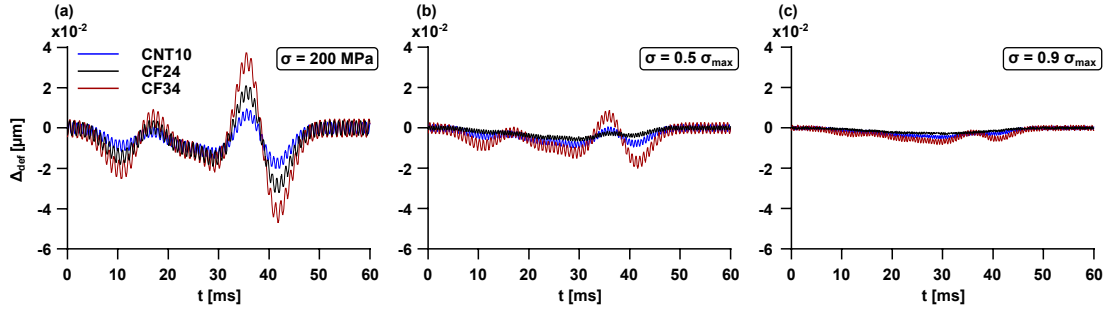


Figure 2.22: Difference between the vibrational response and the adiabatic model for the three wires for an applied stress of (a) $\sigma = 200$ MPa; (b) $\sigma = 0.5\sigma_{max}$; (c) $\sigma = 0.9\sigma_{max}$.

Technical report for LIU gives an absolute accuracy on beam sigma for several measurement less than 20 μm and a precision in a single measurement less than 10 μm

The important point of this model is the observation that the dynamic response follows the adiabatic model almost perfectly (Fig. 2.22). It is also noted that the strain profile follows the same shape as the velocity profile and can also be expressed using a three-term Gaussian. The representation of the deformation of the wire as a function of time and of the distribution along the wire (Fig. 2.21.b) makes it possible to visualize more generally the various maximum deformations at any point of the wire. It is in the center that the deformation is the greatest at the peak of the speed.

Hence, in order to avoid vibrations of the wire, one should program the swelling of the rotation such that one is well in the adiabatic regime, ie with a slow and smooth enough switching.

2.3.3 Internal stress induced by the wire deformation

We have seen previously that the wire, submitted to the centrifugal force, is subject to a deformation. This deformation is maximal in the middle of the wire. This deformation introduce an increase of the wire length. The new length can be calculated as follows

$$L_d = \int_0^L \sqrt{1 + \left(\frac{dy_{stat}(x)}{dx} \right)^2} dx \quad (2.50)$$

The derivative function of $y_{stat}(x)$ is

$$\frac{dy_{stat}(x)}{dx} = \alpha R \frac{\sin\left(\frac{1}{2}\alpha(L - 2x)\right)}{\cos\left(\frac{\alpha L}{2}\right)} \quad (2.51)$$

We can evaluate numerically this integral to get the length of the wire after deformation. The results shows a length increase of 2 nm, 6 nm, 8 nm, 7 nm and 13 nm for the samples CF7, CNT10, CNT20, CF24, CNT30 and CF34 respectively

for the PF conditioning. For the CC conditioning and real preload, the wire increase by 18 nm, 31 nm and 54 nm for the samples CNT20, CNT30 and CF34 respectively (Table. 2.11).

Table 2.11: Wire length increase due to the deformation undergone during the scan.

	Wire length increase [nm]					
	CF7	CNT10	CNT20	CF24	CNT30	CF34
PF / theory	2	6	6	8	7	13
CC / real	-	-	18	-	31	54

Note that despite an increase by a factor of 3 for CNT20 and a factor of 4 for CF34 between the test samples and the copper samples, these increases in length are negligible compared to the total length of the wire ($3.6 \cdot 10^{-5} \%$ for the worst case). It is therefore not absurd to neglect this elongation and therefore the additional stress generated by the deformation. We can therefore consider that the stress in the wire during the scan is the initial stress due to the preload during installation.

2.4 CONCLUSION AND PERSPECTIVES

This work focused on the comparison of the mechanical properties of CNT wires between a conventional assembly of pristine wires on a paper frame and other wires copper coated at their ends. The study of the cross sections has shown that the structure of these threads is not homogeneous with, in particular, a greater packing at the core than at the surface. In addition, the overall packing seems to increase with the increase in diameter. The direct copper plating without an intermediate layer on this type of wire is a novelty and has been tested to be able to meet the current specifications of the new generation wire-scanner.

The study of the copper-coating was first of all hindered by the bad reproducibility of the samples, with very variable diameters and surface conditions which lead to much higher variations in the mechanical properties compared to the PF case. The copper / carbon interface shows copper penetration into the interstices of the wire in the surface layer. The tensile tests show an increase in the disparity of the mechanical properties with the increase in diameter. Furthermore, the mechanical strength was on average 30 % higher for the PF conditioning (910 MPa) than for the CC conditioning (640 MPa), for the three diameters. Weibull analysis also shows the importance of considering changes in the length of the sample which induces changes in σ_0 [307]. The tensile tests carried out with PF conditionings used gauge lengths of 12 mm while CC batches had gauge lengths of 30 mm and the design length for the wire to be put in the WS is 150 mm. We therefore corrected σ_0 so that the average strength and probability of failure for a given load corresponds to the same 150 mm length for all the batches. We found, as expected, that the lower the m parameter, the larger the significance of that correction on the strength results. The decrease of the scale parameter (σ_{0-op}) when shifting to a larger volume model is coherent with the increase of the number of defects and their distribution in the volume. This confirmed our observations under the microscope on the non-uniformity of the structure and the variations of the numbers and types of defects. The Weibull modulus for the batch with a 10 μm nominal diameter is much larger than for the 20 and 30 μm batches, which indicates a more uniform distribution of the defects in the structure, possibly with less defects than for the 20 and 30 μm batches. Clearly, a solution to improve the reproducibility of the copper-coating process necessary to weld the wire in the inserts of the WS must be found or the entire insertion sub-system should be redesigned.

The calculation of the failure probabilities for conventional installation loads of 10 to 50 g showed the impossibility of installing 10 μm wires with these loads without breaking them. However, our model for the maximal deflection of the wire, for a given pre-load applied during the installation of the wire, showed that the relevant quantity is not the load but the stress. For a smaller diameter, a smaller load is required to get a given stress, the failure probabilities for a given

stress show a much different tendency, with the smaller diameter being the most interesting, partly due to the smaller variability of the failure stress during our tests.

The conclusion of this study is that to increase the measurement accuracy on existing instruments during scans, it will be preferable to use small diameter wires with low density, provided they exhibit the same homogeneity of strengths than during our tests, and that it would be useful to design a new fixing system to use pristine wires without copper coating.

It would be very interesting and judicious to complete these results with the work of Juan Alvarez [312] which models the different chains of uncertainty when measuring the position of the wire and the consequences on the mechanical elements caused by the acceleration.

In the present study, we saw (Table. 2.6) that the wire that can carry the highest load is the one with the largest diameter. However, the highest tensile strengths are obtained for the 20 μm diameter. A possibility to combine these two parameters would be to braid wires of small diameter and high mechanical resistance in order to get an equivalent diameter equal to 35 μm , which is the maximal diameter required for accelerator physics. This would lead to increase the cross-section of the new wire, keeping the best mechanical properties of the fibers. A braiding machine prototype has been developed at CERN and the first braided wires have been created using 100 μm CNTWs. Mechanical tests will be performed on these new braided wires to assess their strength distribution.

In the old WS we used carbon fibers. This kind of material has been extensively used in research and industry. This has led to a good understanding of their mechanical properties. On the contrary, the development of CNTWs is more recent, and little is known about their properties, especially for applications in beam instrumentation which are very rare [313, 314]. Previous studies of these wires after proton [271], ions [267], electrons [264, 272] or gamma ray irradiation [269, 270] at low energies showed an improvement of mechanical resistance under certain conditions, possibly due to the creation of chemical bonds between layers of CNTs or between CNTs themselves. However, we currently have no results at very high energies (GeV / TeV). This is why a project has been developed at CERN to irradiate CNTWs in the SPS, with protons of 450 GeV and an integrated intensity of $3.2 \cdot 10^{15}$ particles. This work is fully developed in the chapter. 3.

Chapter 3

Influence of irradiations

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3.1 INTRODUCTION

In this chapter, we discuss our experimental set up on the CERN HiRadMat experimental line allowing the $440 \text{ GeV}\cdot\text{c}^{-1}$ proton irradiation of samples of carbon nanotube wires, and the FLUKA simulations done to help us on the interpretation of the experimental data.

The first part focuses on the description of the experimental area, its specificity and constraints, particularly in terms of radioactivity due to the presence of a dump at the end of the lines, and the implementation of the experiment. We describe in detail the different elements of the experiment, the instruments used, their principle of operation and their characteristics. We also explain the challenge of the tests, the implementation of the experimental plan and the problems encountered during the experiment. A sub-part is devoted to the production method of our samples and their characterization before irradiation.

In another part, the results of the simulations of beam/matter interactions carried out with the FLUKA code are discussed, notably the influence of the level of vacuum in the tank on the production of secondary electrons and the interest of using a scintillator electronic system - PMT for the measurement of secondary particles generated. In order to estimate and predict the temperatures reached by the samples during the tests, we study the different parameters relating to the energy deposited and discuss the results of the different scorings proposed by FLUKA, a particle / matter interaction simulation software. Using these parameters we predict the secondary emission yield for the different samples and compare the different productions of secondary particles produced by each type of sample. Finally, we proceed to the thermal simulations to predict the behavior of the samples during the irradiation.

An analysis of the images produced by the BTV (Beam TV instrument, *cf: [section 3.2.2.2](#)*) is performed to know the exact position of the wire with the beam. The signal produced by the SEM-grid is normalized and analyzed to look for relations between the production of secondary electrons with the diameter and the dose deposited by the beam in the samples, with the assumption of a perfect circular and homogeneous wire.

Post-irradiation studies are carried out to analyze the deformation observed after the interaction with the beam as well as tensile tests to evaluate the influence of the irradiation on the strain to failure, the specific strength and the specific modulus. It should be noted that the great fragility of these samples caused us to break some of them during handling and installation in the traction machine, especially for the thin wires. Finally a Raman spectra analysis is operated to study the potential change of CNT structure. Due to the time needed of each analysis and the results observed, we did not test all of them.

Finally, preliminary results of scans operated with a CNT wire installed on an operation linear wire-scanner are presented.

Despite the great care with which we prepared this experiment, we were subject to several surprises. Unfortunately, we noticed that the wires which were supposed

to be identical (in structure and geometry) showed extremely variable results. The microscopic observations revealed that the wires were not cylindrical. This caused a problem to define their diameter as well as the interaction volume with the beam at the irradiation point.

Furthermore, we encountered technical difficulties within the HiRadMat experiment. The experimental design called for a homogeneous beam size. However, during the experiment, a beryllium window failed which caused a vacuum leak. The experiment was paused during three days. When we were able to resume the experiment, the operational team requested larger beam sizes (0.50 mm) to avoid another window failure. This is why we ended up with two beam sizes (0.25 mm and 0.50 mm). The obtained results for these two beams cannot be directly compared to one another.

Last, we installed carbon fibers currently used in the wire-scanners, for direct comparison with the CNT wires. During the tests, the CF samples did not show any signal with the beam interaction despite the certainty of a good alignment. Our assumption was a failure of both carbon fibers. However, the inspection of the samples after the experiment showed that the carbon fibers did not fail. We do not have reasonable explanations for that. Moreover we do not have analysis of the secondary electron emission for these samples but only the results of the tensile tests.

Because of these problems, we were unable to make all the comparisons we wished between the wires and the condition, so that it is difficult to draw as many significant conclusions as we wished.

3.2 CNT WIRE CHARACTERIZATION UNDER IRRADIATION AT HIGH INTENSITY PROJECT

3.2.1 HiRadMat: an experimental line

HiRadMat (High-Radiation to Material) is a users facility on an extraction line of the SPS. It is designed to produce high-momentum ($440 \text{ GeV}\cdot\text{c}^{-1}$) and high-intensity ($3.46\cdot 10^{13}$ protons per pulse) pulsed beam of $7.95 \text{ }\mu\text{s}$. After extraction from the SPS, the beam can be focused to reach a beam size of 0.25 mm. Three tables are available to install experiments and a dump is located 10 m downstream to absorb the beam at the end of the experimental line [315, 316] (Fig. 3.1).

After submitting and defending a project called CWCIHI (CNT Wire Characterization under Irradiation at High Intensity), we had the opportunity to get beam time in HiRadMat to irradiate in extreme conditions samples of carbon nanotubes wires (CNTWs) produced and generously provided for free of charge by the IMDEA in Madrid [317] and carbon fibers (CF) from our operational stock.

In the [section 3.2.5](#), we will discuss about the manufacturing process of the samples, the test plans and the results on the electrical properties, the secondary electron emission and the mechanical properties.

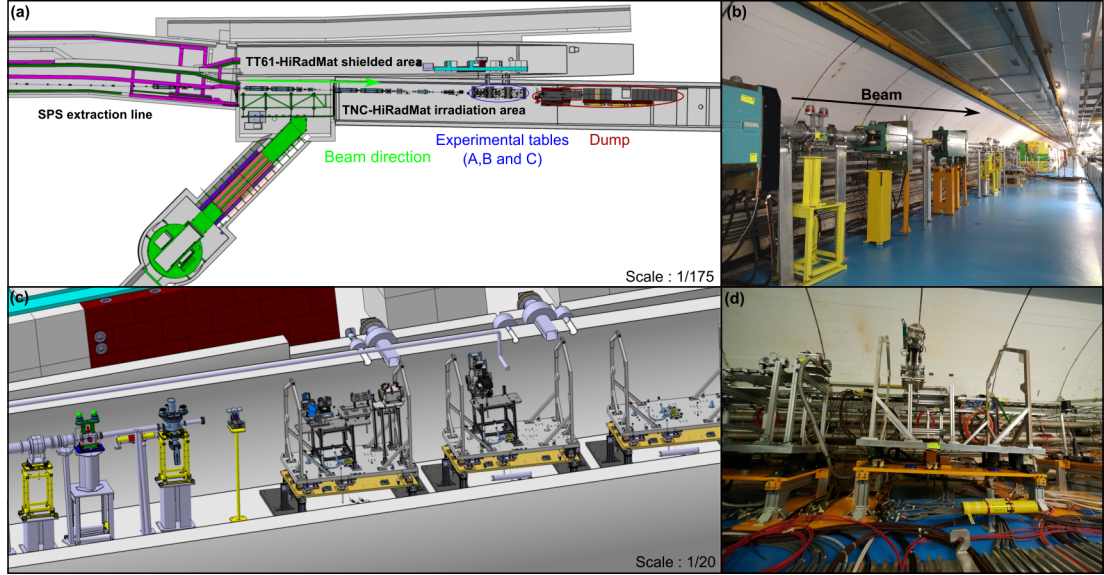


Figure 3.1: (a) HiRadMat layout with extraction line from the SPS, injection in the HiRadMat line, experimental tables and dump in the TNC. Shielded area for electronic devices in TT61; (b) picture of the HiRadMat line; (c) CAD model of experimental area; (d) picture of the experiment installed on the experimental table B.

3.2.2 Experiment description

The mechanical, thermal and density properties of CNTs are, as we have previously seen, very interesting. It was natural to look into their use for our applications. However, it was agreed very quickly that the sudden transition from using 34 μm carbon fiber to tubes a few nanometers in diameter would be difficult to handle in practice, and the production of CNTs with a length of about 20 cm corresponding to the distance between the forks where the wires are attached is relatively complicated. This is why it was decided to focus our researches on the microscale assemblies of CNTs, first with CNTWs bought from Hitachi Zosen for our mechanical tests described in the [chapter. 2](#), then with samples provided by the material department of the IMDEA, motivated for our tests in HiRadMat.

For cost and time reasons, but also reliability and available electronics and software, the experiment was developed using existing instruments: the SEM grid is a spare for the Linac 4 line, the camera to visualize the beam comes from our test laboratory, the screen and the windows come from the HiRadMat team's spare

parts. Device variable names¹ that allow to download from timber server² the data stored during the experiment were set by the software section of BI group. The experiment was entirely controlled from the CERN Control Center (CCC) (Fig. 3.2) in collaboration with colleagues from the operation group, which controls the life cycle of beams.



Figure 3.2: Control room from where the experiment was piloted (SPS area).

3.2.2.1 Secondary Electron Monitor (SEM grid)

The Secondary Electron Emission grid (SEM-grid) is an instrument commonly used at CERN for measuring transverse beam profiles. Its principle is to use a grid of wires spaced by a given distance. When the beam crosses the grid, the interaction between the beam particles and the wire creates secondary electron emission (SEE) through the ionization of atoms from the material by the primary particles from the beam. As the wires are the same, usually tungsten with gold coating of 40 μm , the number of secondary electrons emitted from each wire is proportional to the number of interacting particles such that a beam profile can be reconstructed by plotting the SEM current for each wire.

In CWCIHI, a SEM-grid is used as a support to irradiate different CNT wires. The distance between consecutive wires is set to 4 mm, a distance large enough compared to the beam size in Hiradmat so that neighboring wires are not impacted

¹ BTV of reference (image): TT66.BTV.660524.DigiCam>LastImage:image2D
 BTV in situ (image): TT66.BTV.660518/Image# imageSet
 SEM-grid (signal): BESTLD.TT61.HiRadMat:Acquisition:semRawData
 BCT (intensity): TT60.BCTFL.610225:TOTAL_ INTENSITY

² <https://timber.cern.ch>

during the irradiation test. The samples are soldered on copper tracks with a resistance of $1\text{ M}\Omega$, which allows continuous monitoring for wire breakage. The samples, through the grid, are connected to the data acquisition system.

The SEM-grid movement control is composed of a stepper motor coupled to a mechanism which extend or retracts the grid through a long rigid arm. The stepper motor has a resolution of $2.5\text{ }\mu\text{m}$ per step and a stroke of 180 mm (Fig. 3.3).

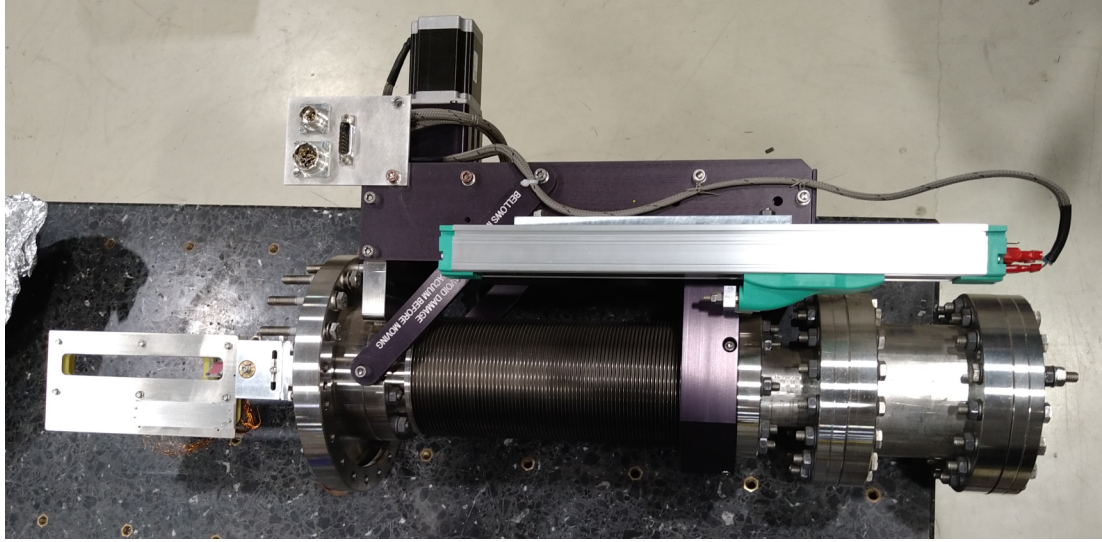


Figure 3.3: SEM grid assembly composed of the stepper motor, the mechanism, the grid and the screen, part of the BTV.

3.2.2.2 Beam Television Visualization (BTV)

The BTV is an instrument very useful which allows a direct visualization of the beam. It combines a fluorescent screen oriented at 45° to the beam axis with a camera placed on the top of the screen which captures the light produced by the interaction between the beam and the screen. Several materials are used depending on the beam characteristics, such as titanium, glassy carbon or Chromox. For our experiment, we chose for availability reasons, a screen composed of Chromox (99.5% of Al_2O_3) and 0.5% of Cr_2O_3), 500 μm thick. This material does not support high intensity which is why we worked with single bunch only. The screen was mounted on an aluminium support. Four small slits have been machined close to the edges of the screen to monitor where its center is and provide a position reference for further measurements. By comparing the beam position measured on this screen and on another BTV placed downstream on the beam line, a precise positioning of the wires can be done, ensuring the protons cross the wires in their centers (Fig. 3.4.a).

A camera Watec Wat-902H Ultimate and a density wheel were located on the top of the tank, centered in its middle. The interaction of the beam with the screen produces scintillation which is captured by the camera during the alignment phase (Fig. 3.4.b).

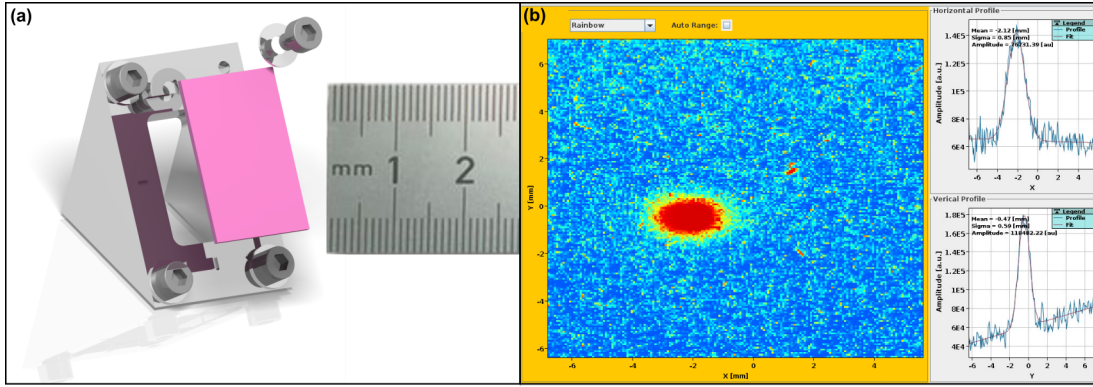


Figure 3.4: (a) CAD model of the screen and its support of the in-situ BTV; (b) Image captured by the camera after a single bunch beam @440 GeV.c⁻¹.

3.2.2.3 Vacuum tank

The tank was designed internally with the constraints of fitting with all the existing instruments (SEM-grid, BTV) and parts (windows in / out in glassy-C, vacuum line). As the idea was to have a support which could be reused for the characterization of thin wires, we gave the possibility to have a ultra high vacuum capability. The choice of the material was stainless steel 316L which is highly corrosion-resistant. Its homogeneous microstructure at the core and on the surface, making it possible to obtain surface states, quality machining and welding. The flanges were ordered on the CERN market, and certified by the CERN quality service. The tank is composed of a main tube of 204 mm in diameter, 2 mm thick and 441 mm long. Holes of 100 mm in diameter have been radially and centered machined for the entry and exit of the beam and a hole of 63 mm in diameter is radially located on the top of the tube for the camera. Chimneys of 2 mm thick are welded using a gaz tungsten arc welding without solid metal to fix the different flanges (Fig. 3.5).

As the alignment was one of the real challenge of this experiment, high tolerances have been specified (Annexe A.1) between the axis of the SEM flange with the axis of the camera and the bottom surface of the tank. As the tunnel has a significant upward slope, the alignment has to be ensured vertically as well as horizontally. To do so, two sliding plates were manufactured with fine-tuning on the horizontal axis and vertical axis. These two plates were supported by three M20 threaded rods which allow a rough alignment with the tilt.

Vacuum level was set to be as low as possible with the existing system considering the long pumping time required, and thus was $1.2 \cdot 10^{-2}$ mbar. Vacuum was ensured by a primary pump group located in the TT61. A Pirani gauge DN16 CF was installed on the tank, connected to surface of the building to measure the vacuum level during the tests.

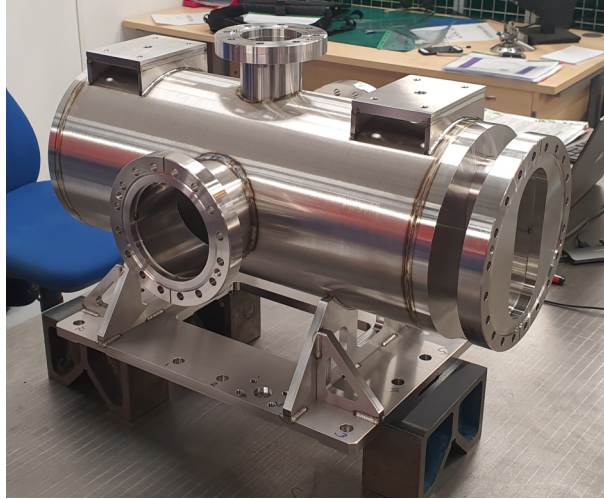


Figure 3.5: As manufactured vacuum tank from Lesker company containing two flanges in/out DN100 CF for the windows, one flange on the top DN16 CF for the camera, one flange DN160 CF for the SEM-grid and one flange DN16 CF for the vacuum gauge.

3.2.2.4 Glassy-carbon window

The choice of the material for the in/out windows (Fig. 3.6) to close the tank was glassy-C, SIGRADUR®. This material is resistant up to temperatures of 3270 K for an inert gas or vacuum environment. The combination of its mechanical properties (high hardness, high strength and low density (1.42 g.cm^{-3}) and its thermal properties (low thermal expansion: $2.6 \cdot 10^{-6} \text{ K}^{-1}$) makes this material an ideal candidate for windows [318, 319].

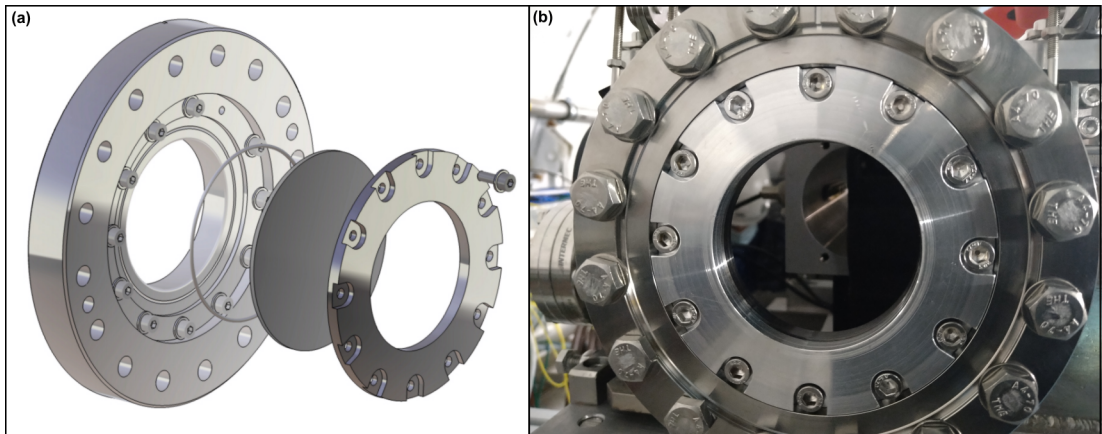


Figure 3.6: (a) Assembly of the window in interaction with the beam composed of a stainless steel flange, a glassy-C window and a counter plate. The sealing is ensured by an indium ring compressed during the assembly; (b) Picture of the window assembly.

3.2.3 Alignment

The real challenge of this experiment was to ensure the alignment between a beam of 0.25 mm and samples of 11 μm . This part was performed in several steps. First, in the lab, a calibration was made between the position of each wire and the center of the screen. The whole instrument assembly was aligned using a monocular and a level, the level was needed to ensure that there was no roll on the system. Once the system was aligned and fixed, the alignment targets where used as the starting reference. The monocular was then moved to the theoretical position of the beam as per drawing. The grid assembly was adjusted into the same roll level as the overall chamber, for this the top edge was measured in vertical direction along the horizontal edge. Adjusting the angle allowed to ensure that there was no vertical offset between the left side and the right side of the assembly. Using the theoretical position of the beam the BTV screen as well as the wires were moved one by one into this position, the position was confirmed via the monocular, at the same time the number of steps needed to reach that position for each wire was noted down. To check and confirm that the numbers were correct two full test runs using the monocular were performed which showed that the wires and the screen where at the right place (Fig. 3.7).

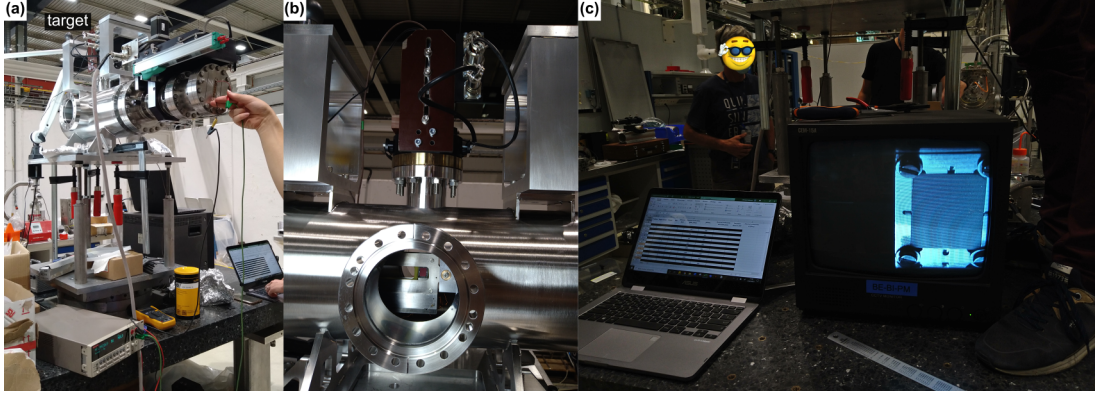


Figure 3.7: (a) SEM-grid installed on the alignment bench with the target; (b) BTV composed of the screen in position "centering" and the camera on the top of the tank; (c) Image of the screen captured by the camera during the alignment procedure.

In HiRadMat the assembly was aligned to the theoretical beam position using the alignment targets and adjustment table. In the tunnel (TNC), the alignment was verified by comparing the images produced by our in-situ BTV with the images of the BTV of reference located a meter upstream on the line. We aligned the beam on our screen with a single bunch beam.

3.2.4 Test plan

The purpose of this experiment was the study of the change of the mechanical properties of CNT wires after irradiation at high-energy and high intensity. To do so, we ordered two batches of CNTW of different diameters: thin and thick. We installed 12 thick CNTW, 10 thin CNTW and 2 carbon fibers (CF) of 7 and 34 μm .

Each group was irradiated with a proton beam of 440 $\text{GeV}\cdot\text{c}^{-1}$. The nominal intensity was set up to $1.2\cdot 10^{11}$ protons per bunch and the number of bunches was requested at 288 per pulses. Originally, the beam size should have been 0.25 mm (for horizontal and vertical directions), but due to technical issues in HiRadMat (a beryllium window failed leading to a loss of the UHV), we had to change the plans for the second part of the experiment: the beam size increased to 0.50 mm and the intensity of a pulse increased from $\sim 2.76\cdot 10^{13}$ to $\sim 2.98\cdot 10^{13}$. Since we were not sure to finish the experiment, we also modified the number of pulses irradiating each wires. The complete initial conditions are summarized in the [table. 3.1](#)

Table 3.1: Initial conditions for each sample: type of wire, beam size and number of pulses.

Wire number	Type	σ_b [mm]	N_{pulses}
1,2	Thick	0.25 / 0.5	
3	Thick	0.25	1
4	Thick	0.25	2
5,6,7	Thin	0.25	2
8,13,14,15	Thin	0.25	4
9,10,12	Thick	0.25	4
11	Thick	0.25	6
16	Thin	0.25	6
17-18	Thick	0.25	6
23	Thin	0.25	10
24	Thin	0.25 / 0.50	3 / 8
19,20	Thick	0.50	6
21,22	Thin	0.50	6
25,26,27,28	Thick	0.50	10
31,32	Thin	0.50	10

Unfortunately, the original plan of having 4 samples of the same type, with the same number of pulses to have robust statistics on the mechanical tests post-irradiation could not be completed because of the problem with the beryllium window.

3.2.5 Carbon nanotube wire samples

3.2.5.1 Manufacturing process

Since details of the manufacturing process and structure of the commercial CNT wires we used for our studies described in the [chapter. 2](#) were missing, we searched in the literature for an academic team producing CNTWs. It made us find a team in IMDEA (Madrid) that could provide such a material. We contacted them and soon set up a collaboration with them.

The samples were produced by floating catalyst chemical vapor deposition process (FCCVD). The liquid reaction solution composed of a carbon source, in our case toluene (C_7H_8), a catalyst (ferrocene ($Fe(C_5H_5)_2$)) and a sulfur promoter (thiophene (C_4H_4S)) is injected and carried by a H_2 gas in a reactor tube of mulite ceramic (SiO_2) heated to 1570 K.

At the beginning of the reaction, at moderately-high temperature, there is a pyrolysis of the catalyst precursor and ferrocene decomposes to form nanoparticles by the collision of metal clusters. At high temperatures the carbon source decomposes catalyzed by the metal nanoparticles. Simultaneously, there is a segregation of the promoter which create localized molten region at the surface of the catalyst nanoparticles. Finally, saturated carbon atoms which are localized next to the molten region precipitate to form caps and grow to form long tubes. The CNT obtained are mainly double or few multiwalls, some hundreds of μm long.

The assembly of individual tubes to form a microscopic fiber (CNTF) occurred in the gas phase during growth. CNT entangle and form a dark aerogel which is drawn through and out of the reactor tube. This aerogel of CNT deposits on the winder to produce a fiber [204, 183] (Fig. 3.8). The diameter of the final fiber is controlled by varying the spinning rate of the winder and then the wire stretching speed, from 7 m.min^{-1} for the thick diameters, to 28 m.min^{-1} for the thin diameters.

This method has numerous advantages. The main reagents being very basic, the production cost is low. Thus a large amount of material can be quickly provided. Moreover, as the process is controlled from beginning to end, it is very easy and convenient to control all the input parameters which act on the CNT structure (number of walls, diameter, length). One major advantage is the production simultaneously of the nanotube and the micro-fibers with a single facility. One of the limitations, if there is no purification post-processing, the final product presents residual catalytic particles. Depending on the needs, it can be a problem. Furthermore, the yarn is not as regular as the one we can get with other process.

The obtained samples with this manufacturing process contrary to those obtained with the dry spinning process look more like fibers than wires. As the aerogel is hooked directly in the furnace and deposited on the winder, the super-bundles agglomerate and are stretched in the same direction, giving the CNTWs

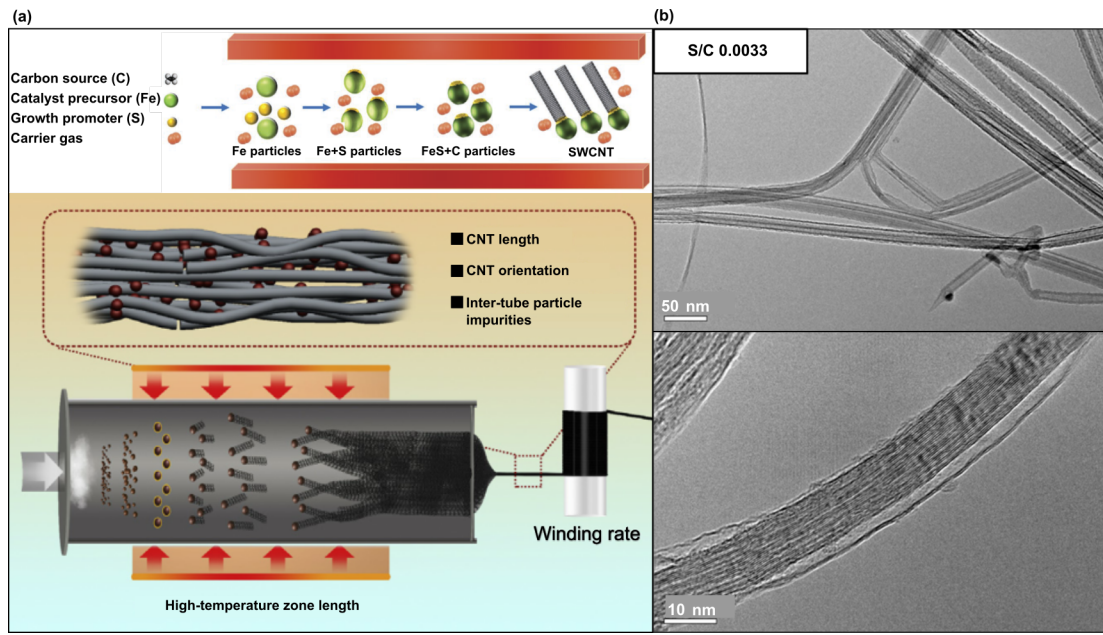


Figure 3.8: (a) Floating Catalyst Chemical Vapor Deposition (FCCVD) process (from Ref [320, 321]); (b) TEM images of CNT bundles spun (from Ref [183] - Supplementary material).

specific morphology (Fig. 3.9).

For the rest of the study I will make a misnomer by speaking of wire to qualify these samples.

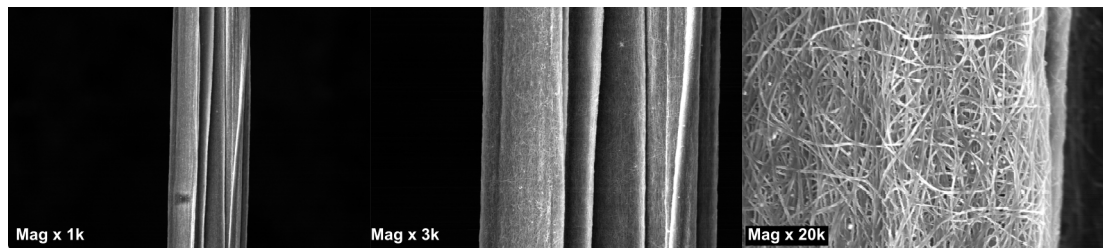


Figure 3.9: As-spun thick yarn (~ 24 μm in diameter) at three different magnifications (x 1k, x 3k and x 20k).

For our experiment, the following parameters have been chosen ([183] - Supplementary Data):

- 1- a promoter / carbon ratio (S/C) equal to 0.0033, leading to the following ratios:
 - $\text{Fe/C} = 0.00086$
 - $\text{S/C} = 0.00332$
 - $\text{S/Fe} = 3.86922$
 - $\text{C/H} = 0.02009$
 - $\text{Fe/H} = 0.00002$
 - $\text{S/H} = 0.00007$

- 2- the flow rate at the injection of the carrier gas (H_2) was 20 sccm (standard cubic centimeters per minute)
- 3- the flow rate of the feedstock (toluene) was 0.0366 sccm

3.2.5.2 Characteristics of the individual CNTs

The S/C parameter chosen for the production of the samples gave a mix of SWCNTs and few-layer CNTs. Assuming a concentration density of CNTs constant and a constant length, the fiber linear density is related to the distribution of the linear densities of the CNTs. Similarly to what was done in the [section 1.3.3.1](#), the linear density of a CNT can be calculated by

$$\lambda_{lin-CNT} = (\pi d_i (1 + 0.68(n_w - 1))) \frac{1}{S_{s-g}} \quad (3.1)$$

with d_i the internal diameter, n_w the number of walls and S_{s-g} the specific surface area of graphene ($1315 \text{ m}^2.\text{g}^{-1}$).

The average linear density of the individual tubes, including CNT with different inner diameters and different number of layers, is $2.56.10^{-8} \pm 0.03.10^{-8} \text{ tex}$. It was calculated using the data of transversal TEM analysis and the [equation. \(3.1\)](#).

3.2.5.3 Wire characteristics

Composition

Due to the manufacturing process and the lack of purification post-treatment, there are residual iron catalytic particles in the wire.

This produces filaments which are collected on a plastic reel which are densified by exposing them to a solvent. The filaments shrink to form a single fiber assembly ([Fig. 3.10.a,b](#)).

Linear density

The linear density of the wire was measured vibroscopically [322] and for each batch of different diameter, the linear density was verified gravimetrically using 150 to 300 m of material continuously collected. The linear density is then estimated at $0.114 \pm 0.023 \text{ tex (g.km}^{-1}\text{)}$ for thick wires and $0.040 \pm 0.004 \text{ tex}$ for thin wires.

Diameter

After black and white binarization (ImageJ) of the photo of the sample taken by an optical microscope, a Python script counts the number of white pixels for each column along the wire, which makes it possible to obtain the average apparent diameter ([Fig. 3.10.\(c.d.e.f\)](#)). The average diameters and standard deviation for each individual wire is plotted on the [figure. 3.10.g](#), with in red, the wires of the thin batch, in green, the wires of the thick batch, in grey the carbon fiber of $34 \mu\text{m}$ and in blue the carbon fiber of $7 \mu\text{m}$. The average diameter for each batch is $11 \pm 1 \mu\text{m}$ for the thin batch, $24 \pm 3 \mu\text{m}$ for the thick batch, $34 \pm 1 \mu\text{m}$ for the thick carbon fiber and $8 \pm 1 \mu\text{m}$ for the thin carbon fiber ([Fig. 3.10.h](#)).

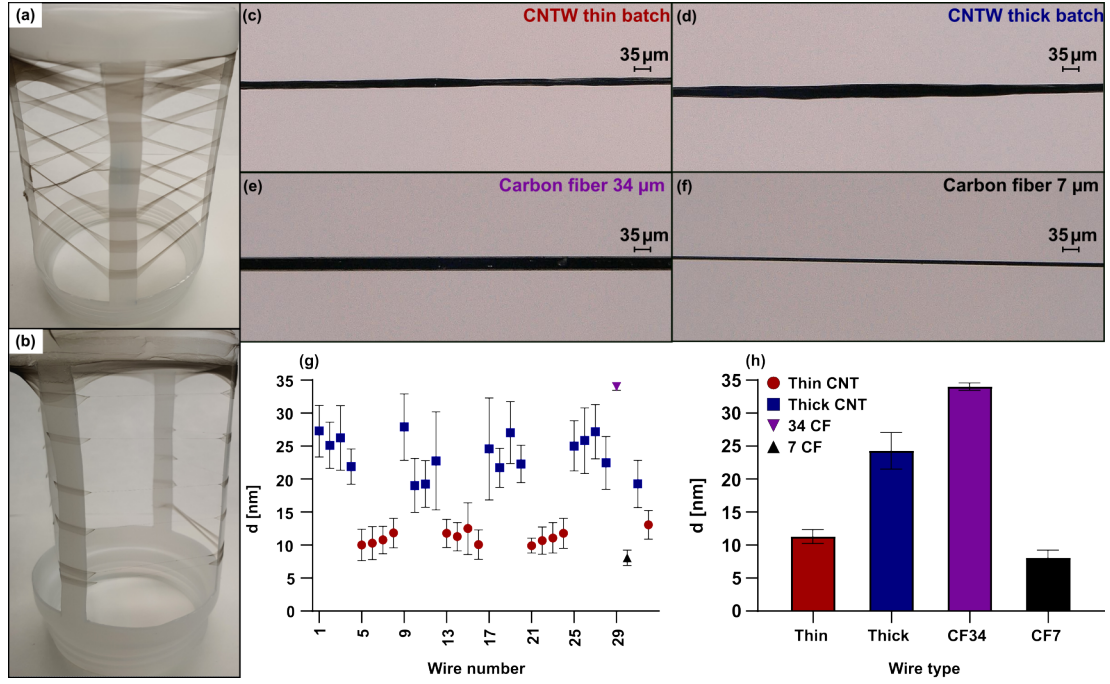


Figure 3.10: Sample procurement: (a) from filaments; (b) to fibers after densification with solvent (from Ref [183]). Optical microscope pictures: (c) of a typical thin CNT wire; (d) of a typical thick CNT wire; (e) a carbon fiber of 34 μm currently used in the wire-scanners; (f) a carbon fiber of 7 μm used in wire-scanners of PSB; (g) average diameter and standard deviation of each sample installed on the grid with their associated number; (h) average diameter for each batch.

Mechanical properties

The mechanical properties of the pristine wires have been tested on samples from the same batch of fabrication. The specific strength is slightly higher for the thin batch than for the thick batch with an average value of 1.19 ± 0.21 N.tex⁻¹ against 1.10 ± 0.09 N.tex⁻¹. The specific modulus is also higher for small diameters, with an average value of 89.1 ± 9.3 N.tex⁻¹ against 78.8 ± 10.7 N.tex⁻¹ for large diameters. At the opposite, the strain to failure for the thin batch is smaller with 3.96 ± 0.51 % against 6.74 ± 1.78 % for the thick batch.

Considering a density of 0.8 g.cm⁻³ for both wires, which is an assumption, the strength is 1488 MPa and 1375 MPa for the thin and thick wires respectively and 111 GPa and 99 GPa for the Young's modulus.

3.2.6 Grid preparation

The electrical layout of the PCB was designed in-house to fit the specifications in term of geometry and available space. The printed-circuit on the grids was designed with the Altium software³. The tracks are 0.2 mm wide and 20 μm thick in copper (Fig. 3.11). The wires are then one by one stretched by hand, positioned

³ <https://www.altium.com/>

on the frame and fixed by tape. They were glued with a conductive quick curing bi-component epoxy adhesive Epotecn E207 precisely dropped with a needle on the copper track.

The assembly of the grid was done in several steps, starting with the mounting of the screen. Then comes the tricky step with the soldering of the wires onto the PCB grid (connecting holes). We finalize the assembly of the polarization plates and the covering plate. The wires have pins at the other extremity, plugged on a feedthrough. This feedthrough is connected to the extremity of the instrument through a SubD50 connector. We added a custom connector to pass from a SubD connector to MDM connector, required for the connection in the tunnel.

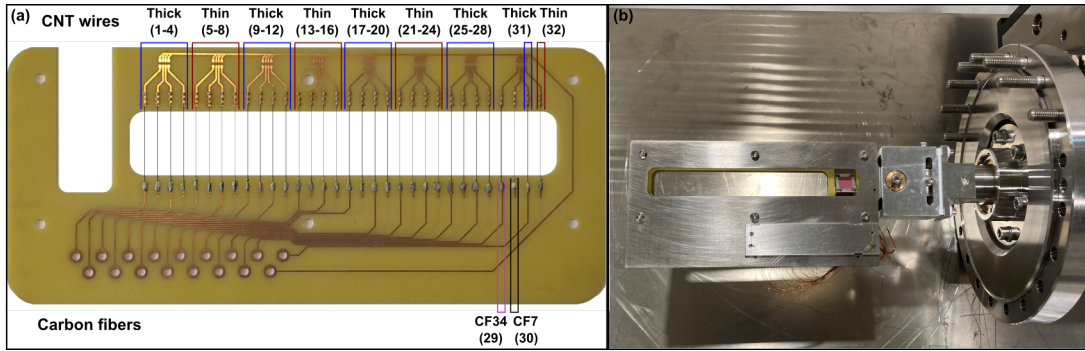


Figure 3.11: (a) grid platine with CNT wires and carbon fibers samples layout; (b) integration of the grid with the polarisation plates and the BTV screen on the main arm of the SEM-grid.

3.3 PRE-TEST SIMULATIONS

3.3.1 Choice of parameters for the FLUKA Monte-Carlo code

The FLUKA code is a general purpose Monte Carlo code for the interaction and transport of hadrons, heavy ions, and electromagnetic particles from few keV (or thermal energies for neutrons) to cosmic ray energies, in arbitrary materials. It is built and maintained with the aim of including the best possible physical models in terms of completeness and precision. In this "microscopic" approach, each step has sound physical basis. This kind of algorithm leads to faster results than compiling particle production data at single interaction level [323].

FLUKA has a graphical interface FLAIR. The whole model is built through this interface and the different parameters are written in a text file (Fig. 3.12).

Pre-test simulations were required to validate the project proposal to the Hi-RadMat board and to anticipate the thermal behavior of the wires. The assessment of the energy deposited into the wires was performed with FLUKA. Energy trans-

3.3. Pre-test simulations

```

1 TITLE
2 SPS exit at 440 GeV
3 * Set the defaults for precision simulations
4 DEFAULTS
5 * Define the beam characteristics
6 BEAM -440. 0.0 1E-6 1E-3 0.0 0.0 -1. PROTON
7 DETECT 0.0 1E-6 1E-3
8 DETECT 0.0 1E-6 1E-3
9 SCORE ENERGY DOSE
10 USRIN 8. ENERGY -21. @LASTREG 400. 10000.EDREG
11 USRIN 1. 0.0 -10000. 1. 1. &
12 USRIN 10. ENERGY -23. 25. 25. 105.Bts
13 USRIN -25. DOSE -20. 500. 500. 500. &
14 USRIN 12. DOSE -25. wire1
15 USRIN wire1
16 USRIN 12. ENERGY -26. wire1
17 USRIN wire1
18 USRIN 12. PROTON -27. wire1
19 *USRIN wire1
20 *USRIN 12. DPA-SCO -34. wire1
21 *USRIN wire1
22 *USRTRACK -1. ENERGY -35. wire1.90066E-6 500.EWT &
23 *USRTRACK 500. 1E-6
24 *USRTRACK -1. ENERGY -35. scint 198. 500.EsT &
25 *USRCOLL 500. 1E-6
26 USRYIELD 123. PROTON -45. wire1 tank_in LETelt
27 USRYIELD 0.5 0.1 100. 440. 3101. &
28
29 RANDOMIZ
30 STEPSIZE 0.000001 1. wire1
31 FLUKAFIX 0.01
32 *IONTRANS HEAVYION CNT_Fe
33 MULSOFT 1. 2000.GLOBAL
34 DELTARAY 1E-6 CNT_Fe
35 PHYSICS 2. EM-DISSO
36 *PHYSICS 3003. 1000. EVAPORAT
37 *PHYSICS DMTHRES
38 *PHYSICS COALESCE
39 *PHYSICS 1.
40 * Define the beam position
41 * Set the number of primary histories to be simulated in the run
42 START 10000000 COMNAME
43 GEOBEGIN 1.
44 0
45 RPP BLK -55. 55. -55. 55. -55. 110.
46 RPP ROOM -50. 50. -50. 50. -50. 105.
47 RCC TUBEL_EX 0.0 0.0 -50. 0.0 0.0 155. 5.
48 RCC TUBEL_IN 0.0 0.0 -50. 0.0 0.0 155. 4.8
49 RCC TANK_E -22.05 0.0 0.0 44.1 0.0 0.0 10.2
50 RCC TANK_I -21.85 0.0 0.0 43.7 0.0 0.0 10.
51 RCC WIRE30 0.0 -1. 0.0 0.0 2. 0.0 0.00055
52 RPP SCINT -21.5 -10.5 -18.5 -0.5 100. 101.
53 END
54 blk_hole 5 +BLK -ROOM
55 airroom 5 +ROOM-TUBEL_EX- (+TANK_E) -SCINT
56 wire1 5 +WIRE30
57 tank_ex 5 +TANK_E-TANK_I-TUBEL_EX-TUBEL_IN
58 tank_in 5 +TANK_I-WIRE30
59 tubel_ex 5 +TUBEL_EX -TUBEL_IN-TANK_I
60 tubel_in 5 TUBEL_IN-TANK_I
61 scint 5 +SCINT
62 END
63 GEOEND
64 MATERIAL 24. 7.18 DEBOG CHROMIUM
65 * Stainless-Steel (typical)
66 * Stainless-Steel is produced with Cr content ranging from
67 * 4 - 19 Atomic Percent, and with C content from 0 - 4 percent.
68 MATERIAL 8.
69 COMPOUND 18. CHROMIUM 74. IRON 8. NICKELStainles
70 * 570 Polyvinyltoluene
71 *
72 MATERIAL 10.3 CARBON
73 COMPOUND 10. HYDROGEN 9. CARBON
74 MATERIAL 1.7
75 COMPOUND 1. CARBON
76 * air tube
77 *
78 MATERIAL 1.18E-8 AIR_TANK
79 COMPOUND -1.248E-4 CARBON -0.755267 NITROGEN -0.231781 OXYGENAIR_TANK
80 COMPOUND -0.012827 ARGON
81 * CNT_Fe
82 *
83 MATERIAL 0.8 CNT_Fe
84 COMPOUND -0.9 CARBON -0.1 IRON
85 MATERIAL 6. CNT_Fe
86 COMPOUND -100. CARBON
87 ASSIGNMA Stainles tank_ex
88 ASSIGNMA AIR_TANK tank_in
89 ASSIGNMA AIR_TANK tubel_in
90 * ..1...2...3...4...5...6...7...
91 ASSIGNMA BLCKHOLE blk_hole
92 ASSIGNMA AIR airroom
93 ASSIGNMA Stainles tubel_ex
94 *ASSIGNMA glassyC win_in win_out
95 ASSIGNMA Polyvinyl scint
96 ASSIGNMA CNT_Fe wire1
97 PART-THR -1E-06 ELECTRON POSITRON 0.0
98 *PART-THR -1E-07 ELECTRON POSITRON 0.0
99 EMF
100 STOP

```

Figure 3.12: Typical input file for the Fluka model.

fer from protons to the material at such a momentum ($440 \text{ GeV}\cdot\text{c}^{-1}$) cannot be described only with the Bethe-Bloch formula (Eq. (1.40)), but needs to consider high energy secondary electrons ($>1 \text{ keV}$), namely delta electrons.

The choice of the parameters, the binning and the scoring is important for the calculation time. In our case, a simulation could require 48 hours with a spatial analysis plot on the whole model and enough primary particles to get robust statistics. By removing this output, the time of calculations is around 4 hours, and the size of the output folder is around 4.5 Go.

3.3.1.0.1 Physics input parameters

The momentum was set at $440 \text{ GeV}\cdot\text{c}^{-1}$. The number of primary particles, representing the number of stories for the Monte-Carlo simulations, was implemented at $5 \cdot 10^6$ per core, with 8 running cores and 5 cycles leading to a total number of stories of $2 \cdot 10^9$.

The choice of the beam shape was initially subject of debate. As the code simulate a single proton story, the deposited energy given in the scoring is the average value of all the simulations. A Gaussian shape will tend to scatter the results. To show this effect, we performed simulations for the same target (pure $34 \mu\text{m}$ carbon fiber of $2.1 \text{ g}\cdot\text{cm}^{-3}$), only changing the beam shape and size. Beam sizes of 0.25 mm and 0.50 mm correspond to FWHM (Eq. (1.35)) of 0.059 and 0.118 cm (cm is the unit of length in the code). The results showed a decrease by 95% of the average deposited energy in the wire between a gaussian beam size of 0.25 mm and a pencil beam with a radius null (8351 eV and 357 eV respectively) and 98% between a gaussian beam with characteristic size of 0.50 mm and annular with a radius null (180 eV). The problem comes from the fact that with a beam much larger in

section than the diameter of the braid, most of the protons will not interact with the braid, therefore depositing nothing as energy. This will contribute to lowering the average of the energy deposit very strongly.

3.3.1.0.2 Geometry

The geometry built for all the simulations is basic. We first define a black box which is the limit where the particles are tracked. This black box, called black hole in the software, is a cube of $110 \times 110 \times 110$ cm centered around the wire. Inside this box, we define the environment, composed of air. The tank is represented by a cylinder of dimension ($d20 \times l44$ (x)) in centimeter with a wall thickness of 4 mm and two pipes of diameter 10 cm and length ≈ 6 cm are connected at each side of the tank in the beam direction (z). The tank is in stainless steel and closed at the entry and exit of the beam with two glassy carbon windows. The wire of variable material and diameter has a length of 2 cm and is installed in the middle of the tank (Fig. 3.13). The pressure in the tank was implemented by the adjusting the air density.

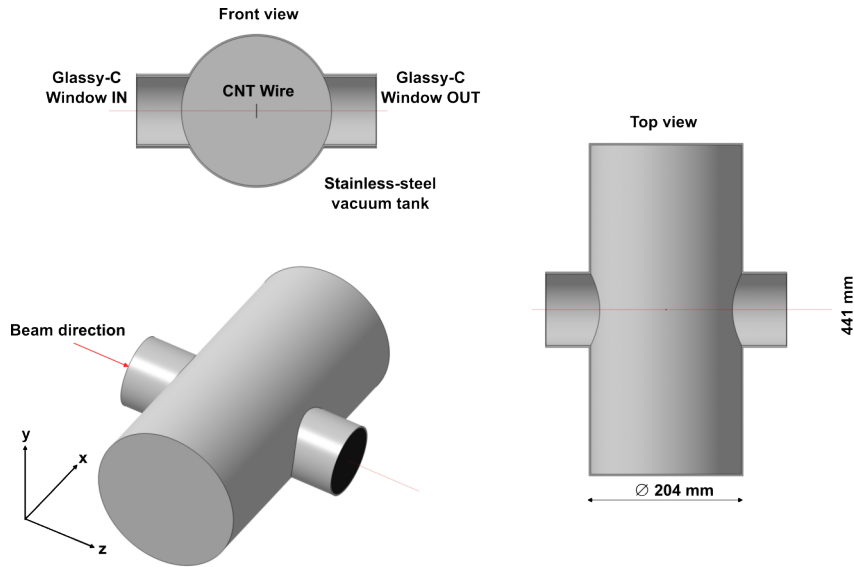


Figure 3.13: Geometry of the model for the FLUKA simulations, composed of two tubes (in/out), a tank and the wire in the middle.

3.3.2 Vacuum level

The secondary electron emission is a sensitive measurement due to the ionization of the air during the passage of a high-energy, high-intensity beam, leading to the high probability of secondary particles to hit the wires of the SEM-grid. The number of the electrons may be calculated to evaluate the necessary vacuum level to avoid the perturbation of the measurement due to parasite electrons colliding with the samples.

The maximum allowed gas pressure for HiRadMat SEM grids is estimated from the number of residual gas particles that are inelastically scattered by the 440 GeV.c⁻¹ proton beam that could hit a SEM wire. Residual gas is best approximated by nitrogen (N₂), that is assumed to have a cross-section 102 times higher than Neon (Ne) (based on measured fluorescence cross-section data [324]).

The overall number of Ne atoms that are inelastically scattered per beam is:

$$N_{isw(Ne)} = N_p \sigma_{p-Ne} \rho_{Ne} p_i \quad (3.2)$$

where N_p is the number of protons in the beam, ρ_{Ne} is the density of the gas, σ_{p-Ne} the cross-section for proton-Ne inelastic scattering ($\sigma_{p-Ne} = 245$ mbarn) and p_i is the probability that a scattered particle (particle from the environment hit by a proton) hits the wire. Such a probability is calculated from scattering data measured from the LHC Beam Gas Vertex instrument for $l_{path} = 150$ mm before a vertical wire of 30 μ m of diameter for a beam with horizontal and vertical beam size of 0.5 mm. The calculation is done via a Monte-Carlo simulation and is $p_i = 6.06.10^{-4}$.cm.

Considering a Ne gas pressure of $P_{Ne} = 1$ mbar, the gas density ρ_{at-Ne} is derived from the pressure as follows:

$$\rho_{at-Ne} = \frac{P_{Ne} \mathcal{N}_A}{\mathcal{R}T} \quad (3.3)$$

where $\mathcal{N}_A$ is the Avogadro number and $\mathcal{R}$ the ideal gas constant.

This gives $\rho_{Ne} = 2.47.10^{16}$ particles.cm⁻³ at $T = 293.15$ K.

For a beam of 288 bunches, consisting in $N_p = 3.46.10^{13}$ protons, the number of scattered particles per beam that hit the wire is $N_{isw} = 127$.

Considering that the probability for a proton of the beam to hit the wire is $1.85.10^{-2}$, and the probability to emit a secondary electron is of the order of 10^{-2} per proton, the emission of secondary electrons per beam is expected to be of the order of 10^9 electrons. We conclude therefore that a 1 mbar pressure has no significant effect on the SEM-grid. To ensure that no parasite particles hit the wires and distort the measurement, two polarisation plates was installed to catch these particles before reaching the wires.

To corroborate this result, we performed FLUKA simulations to get the fluence of electrons after the passage of a 0.25 mm beam at 440 GeV.c⁻¹ in the tank at different vacuum level: the atmospheric pressure, 1 mbar and 10^{-2} mbar, corresponding to air density of $1.2.10^{-3}$ g.cm⁻³, $1.18.10^{-6}$ g.cm⁻³ and $1.18.10^{-8}$ g.cm⁻³. We observe a fluence of 1.47 ± 0.05 electrons per incident protons for $P = P_{atm}$, 1.47 ± 0.05 for $P = 1$ mbar and 1.20 ± 0.04 for $P = 10^{-2}$ mbar. There is a slight difference between the atmospheric pressure and 1 mbar in the cone of projection (Fig. 3.14), but get a variation of 19 % of the fluence for a decrease of the pressure by a factor 10^5 . This indicates that the vacuum level has finally a poor impact on the production of secondary electrons.

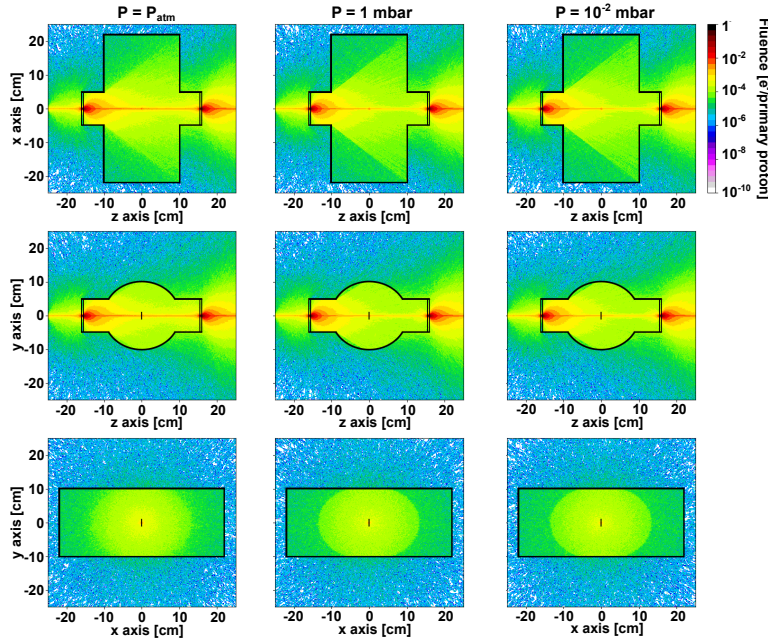


Figure 3.14: Evolution of electron fluence with the pressure for $P = P_{atm}$, $P = 1$ mbar and $P = 10^{-2}$ mbar

3.3.3 Energy deposition and stopping power

The composition of the CNT wires was a problem, due to the presence of iron atoms coming from the decomposition of the ferrocene catalytic particles. Hence, to simulate the deposited energy and the production of secondary electrons, we had to consider a proportion of iron (10 % in weight) in the wire. The effective atomic number is then described by the empirical relation [325]

$$Z_{\text{eff}} = \frac{\sum_{i=1}^{n_{\text{comp}}} \left(\frac{f_i Z_i}{M_i} \right)}{\sum_{i=1}^{n_{\text{comp}}} \left(\frac{f_i}{M_i} \right)} \quad (3.4)$$

with f_i , Z_i and M_i the fraction, the atomic number and the molar mass of the i -th element.

For our samples, the new effective atomic number is then $Z_{\text{eff}} = 6.467$ ⁴.

We now compare the behavior of four samples: 2 CNT wire tested in HiRadMat with diameters of 11 and 24 μm with a density of 0.8 g.cm^{-3} (value supplied by IMDEA) and 2 carbon fibers of 7 and 34 μm with densities of 1.7 and 2.1 g.cm^{-3} respectively.

⁴ Considering two elements: carbon and iron, with $f_C = 0.9$, $Z_C = 6$, $M_C = 12.01 \text{ g.mol}^{-1}$ and $f_{Fe} = 0.1$, $Z_{Fe} = 26$, $M_{Fe} = 55.85 \text{ g.mol}^{-1}$

The deposited energy is obtained in two ways by using FLUKA estimators. The first one gives the probability for an incident proton to deposit a certain energy in the wire. We chose a minimum energy of 1 keV (minimum energy for FLUKA) and a maximum of 1 MeV (the probability is near zero for higher energies) (Fig. 3.15.a). By multiplying the probability of the event by the width of the channel and summing over the spectrum, we obtain the total energy deposited in the wire. The choice of the number of bins is crucial. The higher the number of bins, the more precise the results. We decided to use 2048 bins. We notice different shapes for the curves. The peak shifts towards higher energies with increasing diameter. It is observed that for small diameters (7 and 11 μm), the peak is reached immediately, which means that the energy deposited is maximal at low energies. For CF34, a plateau is observed up to about 10 keV before increasing to its maximum and then decreasing again. It is observed that the shapes of the curves during the decrease phase are substantially the same. The figure. 3.15.b shows the evolution of the total response in terms of the deposited energy in the four samples and the average track length. We observe that the total response for the energy is the largest for the CF7 and the lowest for the CF34. Conversely, the track length is maximal for the CF34 and minimal for the CF7.

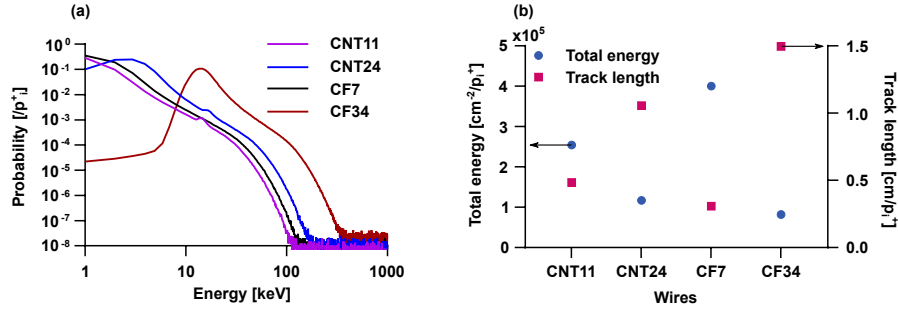


Figure 3.15: (a) Deposited energy per primary proton in CNT of 11 and 24 μm and CF of 7 and 34 μm in diameter at $440 \text{ GeV} \cdot \text{cm}^{-1}$ for energies range from 1 keV to 1 MeV; (b) Total response of the deposited energy in the wire considering the volume of each sample and track length.

The second method which is also the most accurate and the one we will use later, is the use of the estimator USRBIN which scores directly the energy deposited in a described regular mesh. Results for both methods are synthesized in the table. 3.2.

We observe the largest variation for the CNT11 with an increase of 44 % between the spectrum and the estimator and the smallest variation for the CNT24 and CF34 with an increase of only 3 % and 4 % respectively. The largest variation we obtain for the smallest deposited energy could come from the combination of a small diameter and a very light density, leading to weaker statistical robustness, especially since we saw on the table. 3.2.a that the lower cut-off at 1 keV was probably too high for these samples. At the opposite, the large diameter and density of the CF34 allows a good reproducibility of the results. The value which

Table 3.2: Simulated energy deposition simulation for four different samples corresponding to various densities.

	d [μm]	ρ [g.cm ⁻³]	E_{dep} (spectrum) [eV]	E_{dep} (usrbin) [eV]	Variation [%]
CNT	11	0.8	848	1228 (± 0.04 %)	+44%
CNT	24	0.8	2665	2754 (± 0.03 %)	+3%
CF	7	1.7	1354	1622 (± 0.03 %)	+18%
CF	34	2.1	9893	10393 (± 0.02 %)	+4%

is considered true for the rest of the study is the energy given by the USRBIN scoring. This method also gives an excellent statistical error, with a variation on the average of the order of 1 eV.

The deposited energy is the basic parameter which allows the calculation of the stopping power and then the Secondary Electron Yield (SEY) and finally the temperature increase during a pulse. The stopping power calculated from the energy deposition can be expressed in MeV.cm⁻¹ with

$$\left\langle \frac{dE}{dx} \right\rangle = \frac{E_{dep}}{d} \quad (3.5)$$

or the specific stopping power can be expressed in MeV.cm².g⁻¹ with

$$\left\langle \frac{dE}{dx} \right\rangle = \frac{E_{dep}}{d\rho} \quad (3.6)$$

These quantities normalize the energy released by an incident particle in a material. They give quite similar results, indicating that the beam deposit globally the same energy per proton in these four wires (Table. 3.3).

A 3D illustration of the deposited energy in the tank as well as the projection of the dose is shown in the figure. 3.16.

3.3.4 Secondary Electron Yield (SEY) estimation

The SEY is the number of secondary electron emitted per incident particles. Its computation require various parameters (Eq. (1.43)), such as the characteristic length L_s (Eq. (1.44)), which corresponds to the thickness of the material from which on the low energy electrons can escape depending on the density of the material, the molar mass and the effective atomic number. The SEY also depends on the stopping power (dE/dx (Eq. (3.5))).

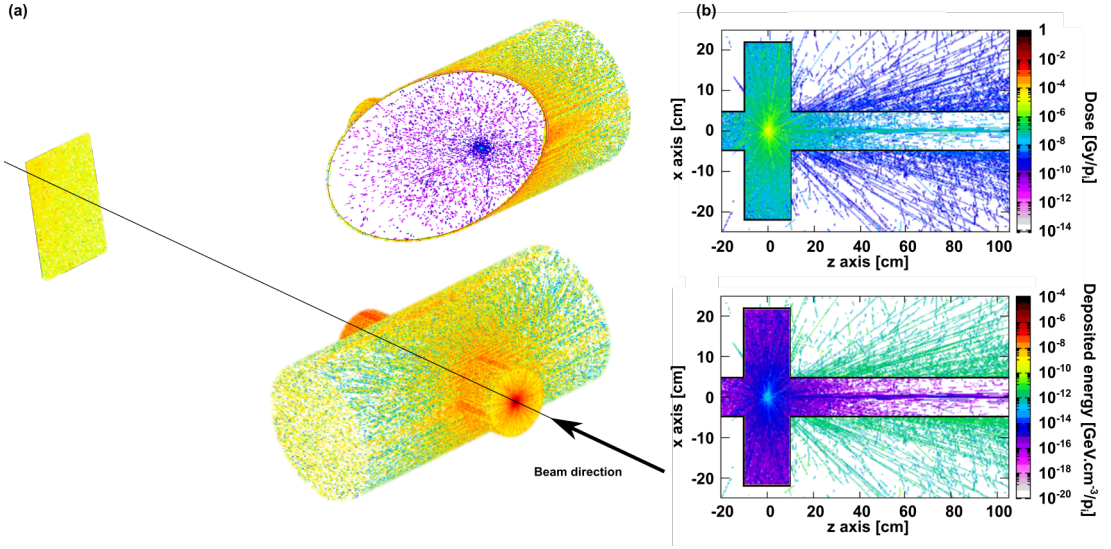


Figure 3.16: (a) Representation of the energy deposited in the tank and scintillator on the 3D model; (b) Dose and deposited energy in the tank in a slice of -0.2 mm / +0.2 mm on y axis.

The atomic density (ρ_{at}), corresponding to the number of atoms per cube centimeter can be calculated with

$$\rho_{at} = \frac{\rho \mathcal{N}_A}{M} \quad (3.7)$$

with ρ the bulk density, $\mathcal{N}_A$ the Avogadro's constant and M the molar mass of the material. The effective molar mass of one mole the CNT containing iron is expressed as

$$M_{CNT} = p_C M_C + p_{Fe} M_{Fe} \quad (3.8)$$

with p_C and p_{Fe} the proportion of carbon and iron respectively, M_C and M_{Fe} the molar mass of carbon (12.01 g.mol⁻¹) and iron (55.85 g.mol⁻¹) respectively, leading to $M_{CNT} = 16.39$ g.mol⁻¹.

From the deposited energy and the parameters described above, the SEY, expressed in number of electrons produced per square centimeters per primary proton, can be calculated with the [equation. \(1.43\)](#). Results are given in the [table. 3.3](#).

Since the atomic density depends on the composition and on the bulk density of the material, we get the same atomic density for both CNT (considering the same ratio of iron) which is 2.5 times less than for the CF7 with $2.9386.10^{22}$ and $8.5234.10^{22}$ at.cm⁻³ respectively. The CF34 has the highest atomic density with $1.0529.10^{23}$ at.cm⁻³.

The characteristic length, also depending on the composition of the material, is similar for both CNT and is 4.96 nm. For both carbon fibers, L_s drops off to 1.75 and 1.42 nm for 7 and 34 μ m respectively.

The SEY can now be computed thanks to the [equation. \(1.43\)](#). Results are shown in the [table. 3.3](#). Carbon nanotubes wires have a larger SEY than carbon fibers,

owing to the lower density and the higher probability for an electron to escape. The secondary electron emission (SEE) can be then obtained by multiplying the SEY by the total number of incident protons and the surface of interest.

Table 3.3: SEY parameters (atomic density, characteristic length and stopping power) and SEY calculation.

	ρ_{at} [at.cm ⁻³]	L_s [nm]	dE/dx		SEY
			[MeV.cm ⁻¹]	[MeV.cm ² .g ⁻¹]	[e ⁻ .cm ⁻² /p _i ⁺]
CNT11	2.939.10 ²²	4.96	1.117	1.398	5.542.10 ⁻³
CNT24	2.939.10 ²²	4.96	1.151	1.439	5.714.10 ⁻³
CF7	8.523.10 ²²	1.75	2.317	1.363	4.065.10 ⁻³
CF34	1.053.10 ²³	1.42	3.057	1.456	4.342.10 ⁻³

We can observe the effect of diameter at constant density by comparing the two CNT wires. For a 118 % increase in diameter, the SEY changes by only 3 %. Another interesting point to underline is that an increase in the density of the wire produces a decrease of the SEY. In addition, the increase in density decreases the SEY by decreasing the characteristic length. The electrons enable of escaping from the wire are contained in a smaller volume, and are therefore in less quantity. The increase in diameter by a factor of 3 (CF34 VS CNT11) does not compensate for the decrease in density by 60 %.

This is explained by the fact that secondary electrons are emitted from the surface, which means the surface at an atomic level. That is why even for variations of diameters in the order of some micrometers, the secondary electron production does not change very much. Moreover, it is important to notice that FLUKA does not consider secondary electrons with energies lower than 1 keV for the SE production, whereas most of them have an energy between 10 eV and some hundreds of eV.

Ideally, for operational SEM-grids as well as for test instruments in HiRadMat, to get a large number of secondary electron emitted, we should have wires with large diameters and low density. But a compromise should be found to get a good enough resolution in terms of number of wires and layout. However, this information is not crucial for the wire-scanners since the measurement method is based on the capture of all secondary particles.

3.3.5 Secondary particle shower

During the interaction between the beam and the wire, a secondary particle shower is created by spallation⁵. In wire-scanners, all these particles are intercepted by the scintillator. We modeled a scintillator as a regular parallelepiped of dimensions (11 cm (x) $\times$ 18 cm (y) $\times$ 1 cm (z)), located 10 cm and 0.5 cm away from the beam axis on x and y respectively, and a meter downstream from the

⁵ Spallation is a nuclear reaction in which an atomic nucleus is struck by an incident particle of high energy. Upon impact, the core decomposes producing jets of lighter particles

wire. We scored the deposited energy and the fluence⁶ of all particles through the scintillator for the four conditions. We scored the fluence of the main secondary particles exiting the surface of the wire and interacting with the scintillator for the four different samples (Fig. 3.17). Most of the produced particles are neutrons, electrons, positrons and pions. Other exotic particles are also created, but to a lesser extent.

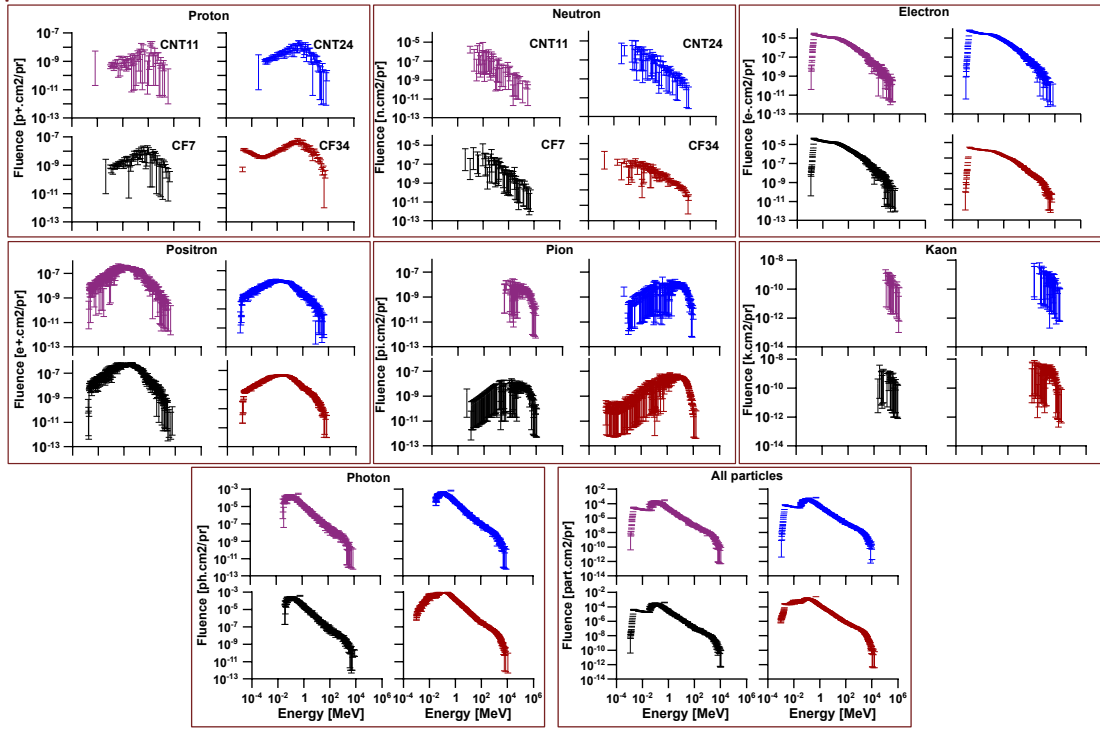


Figure 3.17: Fluence in the scintillator of main particles (proton, neutron, electron, positron, charged pion, charged kaon and photon) simulated by FLUKA, created from the beam wire interaction at 440 GeV.c^{-1} for an energy range from 1 keV to 450 GeV.

The distribution of the fluence is always the same, whatever the particles considered, with the highest fluence for the CF34 and the lowest fluence for the CNT11. The fluence of the CF7 is between the CNT11 and the CNT24. The most numerous produced particles are photons and then electrons with positrons and charged pions in quite similar quantities (Fig. 3.18.a).

Since we scored the tracking of particles inside the wire, the production of the secondary particles comes from the interaction between the protons of the beam and the first atoms of the wire, leading to few secondary particles. On the other hand, we observe a balanced distribution of secondary particles in the scintillator (Fig. 3.18.b), coming from the distance between the scintillator and the wire and the interaction between the secondary particles and the steel wall of the vacuum tube.

⁶ Number of tracked particles per area unit.

The energy deposited in the scintillator globally follows the same distribution as the fluence, except for neutrons which deposit much less energy than the other particles (Fig. 3.18.c). Finally, we can say that whatever the sample, the signal produced in the scintillator has the same order of magnitude. The question of producing enough secondary particles that deposit energy in the scintillator is therefore not a restriction on the type of wire that we could use.

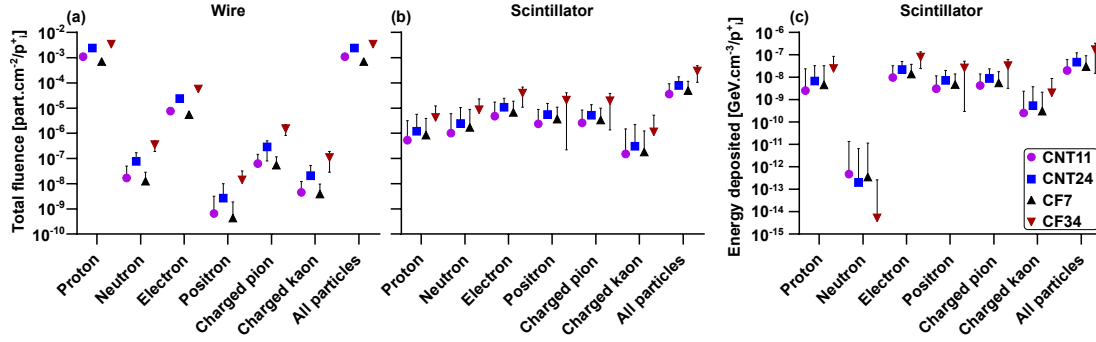


Figure 3.18: Total fluence response for protons, neutrons, electrons, positrons charged pions and kaons and photons: (a) in the wire; (b) in the scintillator; (c) energy deposited in the scintillator by the secondary particles.

By considering this model and this geometry, we observe that the ratio between the energy deposited in the scintillator and the energy deposited in the wire are quite close for all the samples, with an average of 1.73 ± 0.09 % (Table. 3.4). As regards the dose⁷, a marked increase with the density is observed from 1.29 % for CNT11 to 3.40 % for CF34.

The new photomultipliers installed on the new fast wire-scanners automatically select the appropriate amplifiers among four depending on the intensity in the scintillator. Since there are no major differences between these four wires in terms of energy, despite the variation of diameters, density and compositions, this means that each of these wires could be used in the wire-scanners if we only consider the signal point. Let us now consider the question of the rise of temperature. If there is enough signal but the wire cannot hold the increase of the temperature this is a problem. Moreover, during a scan with a wire-scanner, the beam is circulating and interacts more frequently with the wire, creating more secondary particles.

Sapinski modeled the interaction of a 450 GeV and a 7 TeV beam with a carbon wire on varying density [51]. These simulations showed that for an incident proton at 450 GeV gave a high fluence of electrons at low energy (< 10 MeV) and a larger presence of gamma, proton, positrons and pions at higher energies (up to 10^6 MeV). The spectrum is very different for an incident beam of 7 TeV. Electrons are present at low energies (10^{-3} - 10 MeV) with the same profile and a fluence ten times larger. Gamma are also present at low energies and follow the same bell

⁷ The dose is defined as the energy deposited per mass unit of the medium crossed, expressed in Gy.

Table 3.4: Total energy deposited and dose in the wire and the scintillator.

	Energy [GeV.cm ⁻³ /p _i ⁺]		Dose [Gy/p _i ⁺]		Ratio [%]	
	Wire	Scinti	Wire	Scinti	Energy	Dose
CNT11	1.23.10 ⁻⁶	2.04.10 ⁻⁸	1.53.10 ⁻⁶	1.98.10 ⁻⁸	1.66	1.29
CNT24	2.76.10 ⁻⁶	4.79.10 ⁻⁸	3.45.10 ⁻⁶	4.65.10 ⁻⁸	1.73	1.35
CF7	1.62.10 ⁻⁶	3.02.10 ⁻⁸	9.54.10 ⁻⁷	2.93.10 ⁻⁸	1.86	3.07
CF34	1.04.10 ⁻⁵	1.74.10 ⁻⁰⁷	4.95.10 ⁻⁶	1.68.10 ⁻⁷	1.67	3.40

curve than the pions between 50 and 10⁷ MeV. Protons, positrons, neutrons and pions appear around 1 MeV and are quite constant in fluence up to 10⁷ MeV.

Sapinski's results seem to show that the beam energy (between 450 GeV and 7 TeV) has no major influence on the total deposited energy (7640 eV and 7460 eV) for a wire diameter of 30 µm and a density of 2 g.cm⁻³. However, the change in diameter has a direct impact on the deposited energy with 6804 eV and 2690 eV for wires of 30 and 10 µm respectively, a density of 1.8 g.cm⁻³ and a beam of 7 TeV. This shows a decrease by a factor 2.5 for the energy against a decrease by a factor 3 for the diameter.

3.3.6 Interest of a scintillator

The installation of a scintillator downstream of the experiment was considered, to match as much as possible wire-scanner conditions in operation. Contrary to the SEM-grid which measures the number of secondary electron emission, the scintillator does not select specific particles. The beam-wire interaction creates a secondary particle shower, of all particles (protons, electrons, neutrons, pions, kaons, neutrino, photons etc ...). Each particle hitting the scintillator creates photons amplified by the photomultiplier. In the accelerator, the beam only interacts with the wire since it travels in vacuum line (contrary to our experiment where the beam crosses the windows of the tanks) . The light produced in the scintillator then comes from this interaction.

In HiRadMat, the problematic is different. As HiRadMat is at the extraction of the SPS, the beam has to be stopped. To do so, a dump specially designed to absorb the beam is located 10 m after the end of the line. It is composed of a core of carbon wrapped in copper and shielded with iron and concrete blocks.

Even though the dump is designed to absorb the beam, when the beam collides with the dump, there is a production of back-scattered particles. The question was the interest of installing a scintillator if the signal coming from the dump is much higher than the signal coming from the wire.

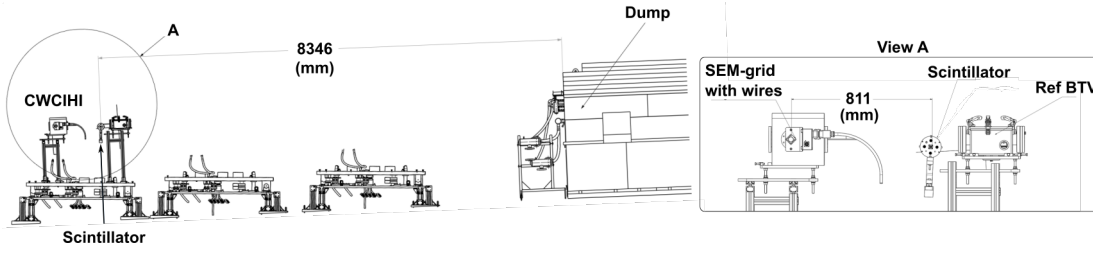


Figure 3.19: First layout of the CWCIHI experiment in HiRadMat, with installation on the first table (A) upstream to the BTV of reference, located ~ 8 m from the dump. The samples are at a distance of 80 cm from the scintillator.

Simulations were performed with FLUKA to get the energy deposited in the scintillator from the dump and from the wire (material carbon and density 2.1 g.cm^{-3}), considering a position of the scintillator of 9 m upstream to the dump and 80 cm downstream to the wire.

From the USRBIN scoring, results showed that the energy deposited in the scintillator by the back-scattered particles from the dump was $66.5 \cdot 10^{-9} (\pm 4.8) \% \text{ GeV}$ per primary proton and $12.4 \cdot 10^{-3} (\pm 1.1) \% \text{ GeV}$ per interacting primary proton (since all the protons of the beam do not interact with the wire). Considering a beam of 288 bunches (N_b) with a nominal intensity of $1.2 \cdot 10^{11}$ proton per bunch (N_{pb}), the number of interactions with the dump is defined by

$$N_{i\text{-dump}} = N_b N_{pb} \quad (3.9)$$

The total energy deposited in the scintillator from the dump is then $E_{\text{tot-dump}} = E_{\text{dep}} N_{i\text{-dump}}$ corresponding to $23 \cdot 10^5 \text{ GeV}$.

For the wire, since all protons from the beam do not interact with the wire, we get the number of interactions with

$$N_{i\text{-wire}} = N_b N_{p-b} f_{wb} p_i \quad (3.10)$$

with f_{wb} is the fraction of overlap of the wire centered on the gaussian beam, and equal to $\approx 1.6\%$ for a $20 \mu\text{m}$ wire and a $\sigma_b = 0.50 \text{ mm}$. p_i is the probability of inelastic collision defined as

$$p_i = 1 - \exp\left(-\frac{d_{av}}{\lambda_i}\right) \quad (3.11)$$

with λ_i the mean free path of inelastic interactions, depending on the material and defined as

$$\lambda_i = \frac{M}{\rho \mathcal{N}_A \sigma_i} \quad (3.12)$$

where M and ρ are the molar mass and density of the wire respectively, $\mathcal{N}_A$ the Avogadro's constant and σ_i the inelastic cross-section for proton on carbon at 440 GeV, and equal to 216 mbarn ($216 \cdot 10^{-27} \text{ cm}^2$).

3.3. Pre-test simulations

This leads for the deposited energy on the scintillator from the wire to a total energy of $2 \cdot 10^5$ GeV.

The signal over noise ratio is therefore 2/23 (Fig. 3.21).

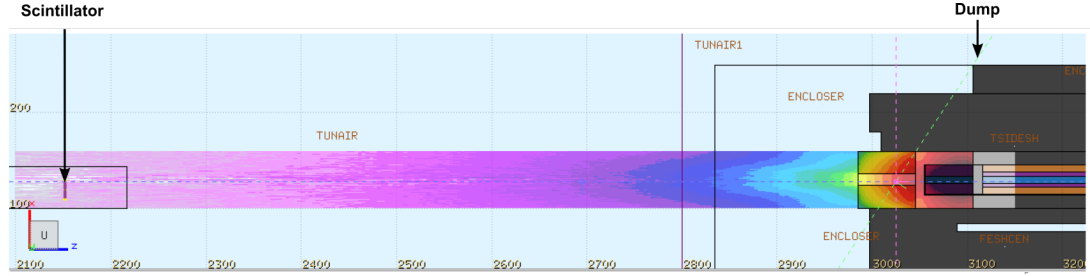


Figure 3.20: Fluence of the back-scattered particles from the dump during irradiation test.

The fact that the noise is 12 times larger than the signal we want to measure is an issue which could have been addressed by installing successive shielding layers between the dump and the scintillator. But this solution implies other issues in terms of logistics, installation and radio-protection considerations.

Another solution would have been to use a single bunch and discretize in time particles from the wires and particles coming from the dump. Considering the geometry, it has been estimated that the secondary particles fly for 3 ns before hitting the scintillator. Considering the initial time when the beam hits the wire, the back-scattered particles arrive on the scintillator 57 ns later (30 ns from the wire to the dump and 27 ns from the dump to the scintillator). The electronics should have been fast enough to measure this difference. Unfortunately, we did not have this kind of electronics quickly available.

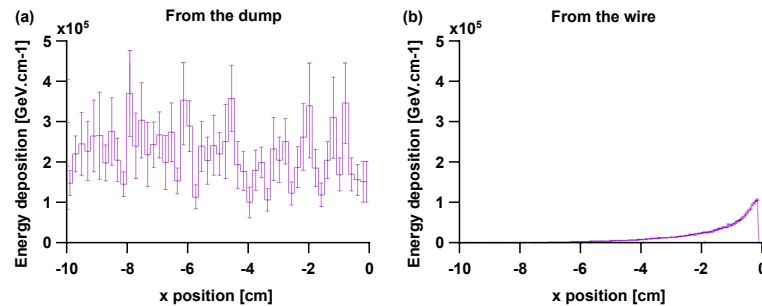


Figure 3.21: Energy deposited in the scintillator: (a) from the dump; (b) from a $20 \mu\text{m}$ wire of 1.4 g.cm^{-3} .

Considering these different parameters, we made the choice not to install a scintillator and focus on the SEM-grid measurement.

3.3.7 Thermal simulations and temperature estimations

The calculation of the temperature is a central piece of this study. We are looking for a material with high mechanical characteristics, but so far, the cause of failures in operation is the overheating.

In order to estimate the maximum temperature reached by the samples during the tests at HiRadMat, we use a python calculation code developed in-house. This calculation is based on a power balance equation [51]

$$\begin{aligned}
 E_{\text{dep}} \frac{\Delta N_{\text{hits}}}{\Delta t} = & \rho V c_p(T) \frac{\Delta T}{\Delta t} \\
 & - A_{\text{rad}} \varepsilon_{\text{th}} \sigma_B (T^4 - T_{\text{amb}}^4) - k_{\text{th}}(T) S \frac{\Delta T}{\Delta y} \\
 & - A_{\text{rad}} \left(\Phi + \frac{2k_B T}{q_e} J_{\text{th}}(y) + C(y) \Delta R J_{\text{th-tot}}^2 \right) \\
 & - H_{\text{sub}} \frac{\Delta n}{\Delta t}
 \end{aligned} \tag{3.13}$$

where the various quantities are described below the [equation. \(1.52\)](#).

The input parameters of the beam used are: the energy deposited by an incident proton (estimated with the Fluka simulations for each sample ([Table. 3.2](#)), the distribution of particles in the transverse plane (Gaussian shape), the beam size (0.25 mm), the duration of the pulse ($7.95 \cdot 10^{-6}$ s) and the total intensity of the beam (nominal intensity of $3.4 \cdot 10^{13}$). On the wire side, we can vary the diameter (7, 11, 24 and 34 μm), the density (1.7, 0.8, 0.8, 2.1 $\text{g} \cdot \text{cm}^{-3}$), the atomic number and molar mass, to take into account the iron inclusions in the CNT wires (6 and 6.467 and 12.01 and 16.39 for carbon fiber and CNT respectively). In addition, it is also possible to define the thermal properties (conductivity and specific heat capacity) as a function of temperature ([Fig. 3.22](#)). Since we do not have data for the variations of these properties at high temperature for the CNTs wires, it is assumed for these simulations that the properties are similar to those of carbon fibers. Introducing the thermal properties of iron, we obtain the specific heat capacity and thermal conductivity as a function of the temperature with

$$c_p(T)_{\text{CNT}} = 0.9 * c_p(T)_C + 0.1 * c_p(T)_{Fe} \tag{3.14}$$

and

$$k_{th}(T)_{\text{CNT}} = 0.9 * k_{th}(T)_C + 0.1 * k_{th}(T)_{Fe} \tag{3.15}$$

The evolution of the temperature as a function of time over the duration of the pulse (7.95 μs) shows a linear increase during the pulse ([Fig. 3.23](#)). We first fixed the thermal parameters to the standard value. We first focus on a beam size of 0.25 mm, and we predict an increase of the temperature up to 3189 K for the CNT24, only 8 % more than for the CF7 which has the lowest temperature increase ([Fig. 3.23.a](#)). The temperature of 1810 K, melting temperature of iron,

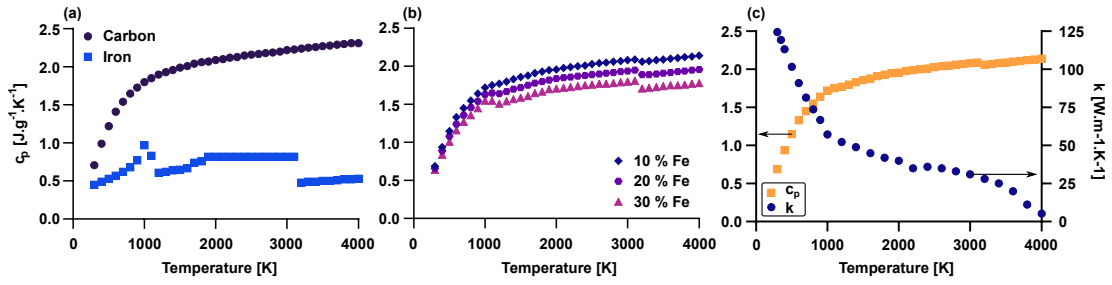


Figure 3.22: Evolution of the specific heat capacity [$\text{J.kg}^{-1}.\text{K}^{-1}$]: (a) of pure carbon (dark blue circle) and pure iron (light blue square); (b) a compound material composed of 10% in weight of iron (dark blue diamond), 20 % of iron (purple hexagon) and 30 % of iron (pink triangle), based on the *NIST-JANAF Thermochemical Tables*, the *table of physical and chemical constant* and the *Handbook of physical quantities*; (c) specific heat capacity (c_p) and thermal conductivity (k_{th}) for CNT wires containing 10 % in weight of iron as a function of the temperature.

is reached in 5 μs for the four samples. The maximal temperature is reached for the CF34 with 3190 K at the end of the pulse, only 8 % more than for the CF7 (Table. 3.5). During the cooling down, the temperature of the CNT11, CNT24 and CF7 decrease in a similar way. The time during which the temperature of the wire is above the melting temperature of iron is less than 4 ms., whereas for the CF34, it lasts 12.5 ms. The ambient temperature is reached after a second for the CNT11, CNT24 and CF7, whereas for the CF34, it is still at 465 K.

Applying variable thermal parameters, we observe a large decrease of the maximal temperature, reaching only 1595 K at the maximum (for the CF24). Surprisingly, the temperature of the CF34 after one second is 590 K, higher by 27 % from the previous simulation (Fig. 3.23.b). This seems to indicate that the variation of the thermal properties with the temperature plays a major role in the thermal behavior of the wire. This leads to the conclusion of the necessary precise knowledge of these properties.

For a beam size of 0.50 mm, the maximum temperatures drops to 734 K for the CNT24 and 697 K for the CF7 (Fig. 3.23.b). This shows that multiplying the beam size by two, the temperature decreases by 54 %.

The temperatures obtained indicate a large variation in the results if we consider variable or constant thermal parameters. Accurate knowledge of these parameters is therefore crucial to accurately estimate the temperatures reached during irradiation and the consequences in terms of degradation. The iron thermal parameters penalize the CNT samples both in the temperature rise and in the cooling profile.

If we refer to the results of the cases of a beam of 0.25 mm and 0.50 mm with variable parameters (Table. 3.5), the maximum temperatures do not reach in any case for any of the samples neither the temperature of sublimation of carbon, nor the melting point of iron. So there should be no degradation of the wires in that scenario.

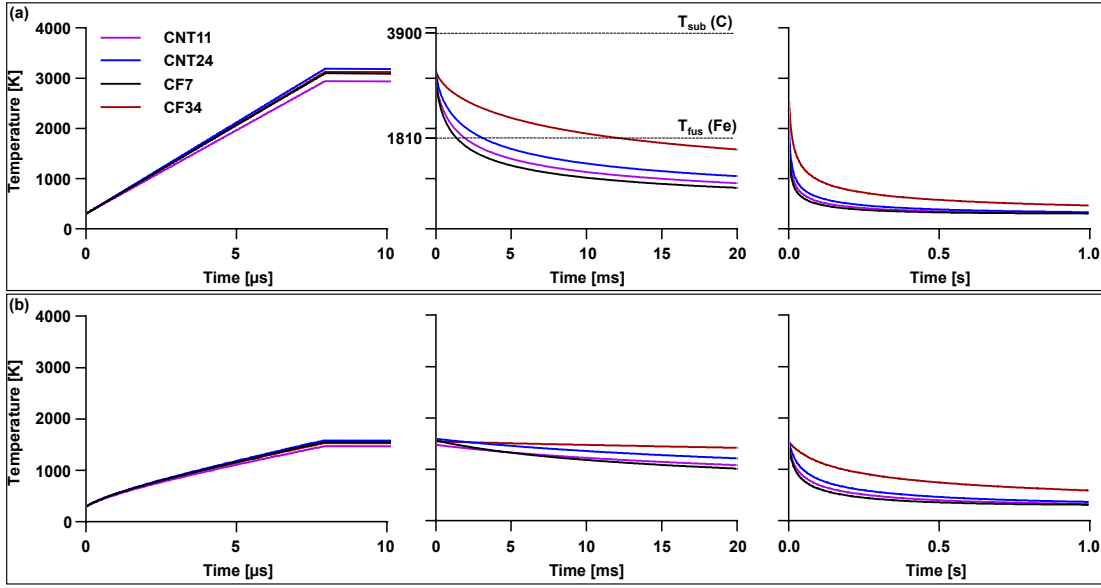


Figure 3.23: Evolution of the temperature as a function of time, for different time scale (μs , ms and s) for a beam size of 0.25 mm, with the consideration: (a) of thermal parameters fixed at the standard temperature (300 K); (b) thermal parameters function of the temperature between 300 and 4000 K.

In addition, the delays between each pulse being ~ 40 s (SPS cycle for our HiRad-Mat tests), the samples have ample time to cool and regain their initial temperature, not initiating a second pulse with a temperature greater than 300 K.

These results are therefore encouraging as regards the lifetime of the samples during the whole experiment.

Table 3.5: Maximal temperature for various conditions of thermal parameters and beam size for the four samples.

Parameters	σ_b [mm]	Max temperature [K]			
		CNT11	CNT24	CF7	CF34
No variation	0.25	3099	3190	2944	3127
Variation	0.25	1562	1594	1477	1542
Variation	0.50	723	734	697	718

3.4 RESULTS

3.4.1 Beam-wire interaction

3.4.1.1 Number of particles crossing the samples

Since various parameters are changing in our experiment (beam size, beam position, intensity, apparent diameter of the wires), the number of interactions varies. To study the evolution of the emission of secondary electrons during irradiation and the mechanical properties to failure as a function of the dose, a normalization is carried out making it possible to overcome the various variable parameters.

However, it should be noted that given the great irregularity of the samples in terms of geometry, the true volume of the wire, and therefore the quantity of material, interacting with the beam cannot be precisely known for all the samples. That is why the results obtained should be considered carefully.

The beam is assumed to have a Gaussian distribution in the horizontal and vertical transverse plans. The number of protons which interacts with the wire is defined by

$$N_{p-w} = I_{\text{BCT}} \frac{1}{2\pi\sigma_x\sigma_y} \int_{-d/2}^{+d/2} \int_{-l/2}^{+l/2} \exp\left(-\frac{1}{2}\left(\left(\frac{x-\mu}{\sigma_h}\right)^2 + \left(\frac{y-\mu}{\sigma_v}\right)^2\right)\right) dx dy \quad (3.16)$$

with I_{BCT} the intensity of the full beam measured by the Beam Current Transformer instrument, d and l the diameter and the length of the wire respectively, σ_h and σ_v the dimension of the beam in the horizontal and vertical plans respectively.

Since the length of the wire is much larger than the beam size, we assume that all the protons distributed on the vertical gaussian go through the wire. We can then reduce the analysis to a single gaussian in the horizontal plane.

The horizontal section (in the plane (x, y)) of the beam is then defined by the function

$$g(x) = \frac{1}{\sigma_h\sqrt{2\pi}} \exp\left(-\frac{1}{2}\left(\frac{x-\mu}{\sigma_h}\right)^2\right) \quad (3.17)$$

The total number of protons crossing the wire N_{p-w} during a pulse is defined as

$$N_{p-w} = I_{\text{BCT}} \int_{-d/2}^{d/2} g(x) dx \quad (3.18)$$

Using the change of variable and defining $x' = \frac{x-\mu}{\sqrt{2}\sigma_x}$, we obtain

$$N_{p-w} = I_{\text{BCT}} \frac{1}{\sqrt{\pi}} \int_{-a}^b \exp(-x'^2) dx' \quad (3.19)$$

with $a = \frac{\frac{d}{2}+\mu}{\sigma_h\sqrt{2}}$ and $b = \frac{\frac{d}{2}-\mu}{\sigma_h\sqrt{2}}$

Splitting the integral, we can express this formula in terms of the error function

$$\text{erf}(X) = \frac{2}{\sqrt{\pi}} \int_0^X \exp(-x^2) dx \quad (3.20)$$

we get for the intensity in the wire

$$N_{p-w} = I_{\text{BCT}} \frac{1}{\sqrt{\pi}} \left[\int_{-a}^0 \exp(-x''^2) dx'' + \int_0^b \exp(-x''^2) dx'' \right] \quad (3.21)$$

Doing a last change of variable with $x'' = -x'$

$$\int_{-a}^0 \exp(-x''^2) dx' = \int_0^a \exp(-x''^2) dx'' \quad (3.22)$$

Finally, the number of protons crossing the wire can be expressed as

$$N_{p-w} = I_{\text{BCT}} \frac{1}{2} \left[\text{erf}\left(\frac{\frac{d_w}{2} - \mu}{\sigma_x \sqrt{2}}\right) + \text{erf}\left(\frac{\frac{d_w}{2} + \mu}{\sigma_x \sqrt{2}}\right) \right] \quad (3.23)$$

However, all incident protons do not interact with the wire. The number of inelastic-collisions is defined by

$$N_{ic} = N_{p-w} p_i \quad (3.24)$$

where p_i is the probability for an interaction, defined in the [equation. \(3.11\)](#).

We can estimate the dose deposited by the beam in the wire, assuming a deposited energy of 1230 eV and 2747 eV for thin and thick wires respectively ([Table. 3.2](#)). We also assume that the vertical distance where all the energy is deposited is $6\sigma_v$. The dose is the parameter which quantifies the energy per mass. Firstly considering the deposited energy calculated with Fluka for the various samples and secondly considering the theoretical mass of perfectly cylindrical wire of specific diameter and density, the dose can be expressed as

$$D = \frac{E_{dep-tot}}{m_{w-i}} \quad (3.25)$$

with $E_{dep-tot}$ is the total energy (J) deposited by the protons interacting with the wire and m the mass of the wire (kg) where the energy is deposited. By expliciting the terms, the dose is

$$D = \frac{4N_{ic}E_{dep}}{6\rho\pi d^2\sigma_v} \quad (3.26)$$

since the volume $V = 6\sigma_v \frac{\pi d^2}{4}$.

3.4.1.2 Determination of the parameters

The beam parameters vary at each new cycle. To get the number of particles going through the samples, actual values of each parameter for the different sequences were considered.

The real total beam intensity (I_{BCT}) (theoretically: $288 \text{ bunches} \times 1.2 \cdot 10^{11}$ protons) was provided by a Beam Current Transformer (BCT) located upstream, in the SPS.

The beam size and the beam position were precisely measured with the BTV of reference for each pulse using a digital camera located 5 m away of the screen in the technical tunnel. This BTV has a high resolution camera of 800×500 pixels with a pixel size of 0.036 mm. The relative position between the wire and the beam can be known by the difference of the beam position between the reference BTV and the local BTV. The local BTV is only used to this purpose and cannot be used to get the absolute beam size. The combination of the screen material, the analogue camera, and the location of the camera give saturated images. This gives large uncertainties on the standard deviation of the gaussian fit, but the mean position is reliable.

With a single bunch beam, we are able to get the images on the two screens. This is a necessary step to set the center of the image using the references on the sides of the screens (Fig. 3.24.a.b).

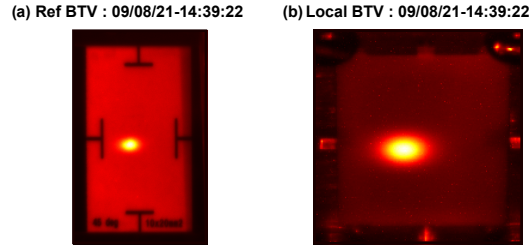


Figure 3.24: Images of the screens used in the (a) BTV of reference; (b) In-situ BTV, illuminated by the beam. The slits on the edges allows the centering of the beam.

The calibration was performed by selecting two pictures of the same beam on both BTV for both days of the experiment (11/08/21 for the 0.25 mm beam and 13/08/21 for the 0.50 mm beam) (Fig. 3.25.a.d.g.h). We observe a clear difference between the quality of the two images, with an image saturated with noise in the background for the local BTV. We plotted the average value for all the columns of the image giving the horizontal projection and the average on all the rows for the vertical projection. For the reference BTV we take the raw signal, which gives the mean (μ) and the standard deviation (σ) of the gaussian fit (Fig. 3.25.e.i). Considering the high level of noise of the local BTV, we normalized the raw signal by taking the average value of the signal between -6 mm and -4 mm and +4 mm and +6 mm. This normalization has an impact on σ , but also on μ . This difference is larger for the noisier image (local BTV) and is 11 % for μ , for $\sigma_b = 0.25$ mm in the

horizontal plane and shifted from 0.658 mm for $\sigma_b = 0.5$ mm. This normalization is then not negligible. The high r -squared indicates a good fit curve representation of the data (Fig. 3.25.f.j).

The center, the standard deviation and the r -squared (r^2) of the gaussian for both BTV are summarized in the table. 3.6.

For the calibration of the first test run, the horizontal projection shows a center at 0.24 mm for the BTV of reference and 0.08 mm for our local BTV. This leads to a difference of 0.16 mm, which will be considered for the rest of the experiment for the beam size of 0.25 mm. For the second run, the difference is 0.09 mm.

Table 3.6: Parameters of the gaussian fit (center μ , standard deviation σ and r -squared r^2) for the horizontal and vertical projection of the BTV of reference and the local BTV for both dates of the experiments.

		Horizontal				Vertical			
		Ref		Local		Ref		Local	
		Raw	Norm	Raw	Norm	Raw	Norm	Raw	Norm
$\sigma_b = 0.25$ mm									
μ	[mm]	0.24	0.24	0.07	0.08	0.06	0.06	-0.37	-0.37
σ	[mm]	0.31	0.30	1.03	0.56	0.25	0.24	0.53	0.39
r^2	-	0.991	0.99	0.42	0.97	0.99	1.00	0.55	0.97
$\sigma_b = 0.50$ mm									
μ	[mm]	0.27	0.27	-0.48	0.18	0.24	0.24	-0.02	-0.22
σ	[mm]	0.65	0.62	4.67	0.76	0.48	0.47	5.05	0.52
r^2	-	0.99	1.00	0.35	0.89	0.99	1.00	0.22	0.89

The real relative horizontal position between the beam and the wire is then supposed to be

$$\mu_{r-0.25} = \mu_{ref-BTV} - 0.16$$

$$\mu_{r-0.50} = \mu_{ref-BTV} - 0.09$$

The experiment was split up in two runs, the first one with a required beam size of 0.25 mm and the second one with a required beam size of 0.50 mm. The tests having being performed during different days, the intensity increased from $(2.76 \pm 0.017) \cdot 10^{13}$ protons to $(2.97 \pm 0.017) \cdot 10^{13}$ protons in average (Fig. 3.26), but shows a good consistency in intensity and in beam size with the increase of the number of pulse. The variation of the beam size for the first run is less than 5 % and except for the three first pulses, the beam size is constant for the second run. However, we easily have access to the real beam sizes and centers thanks to the images of the BTV, hence for the rest of the study, we analyze each pulse with the real data (*cf*: Annex A.7).

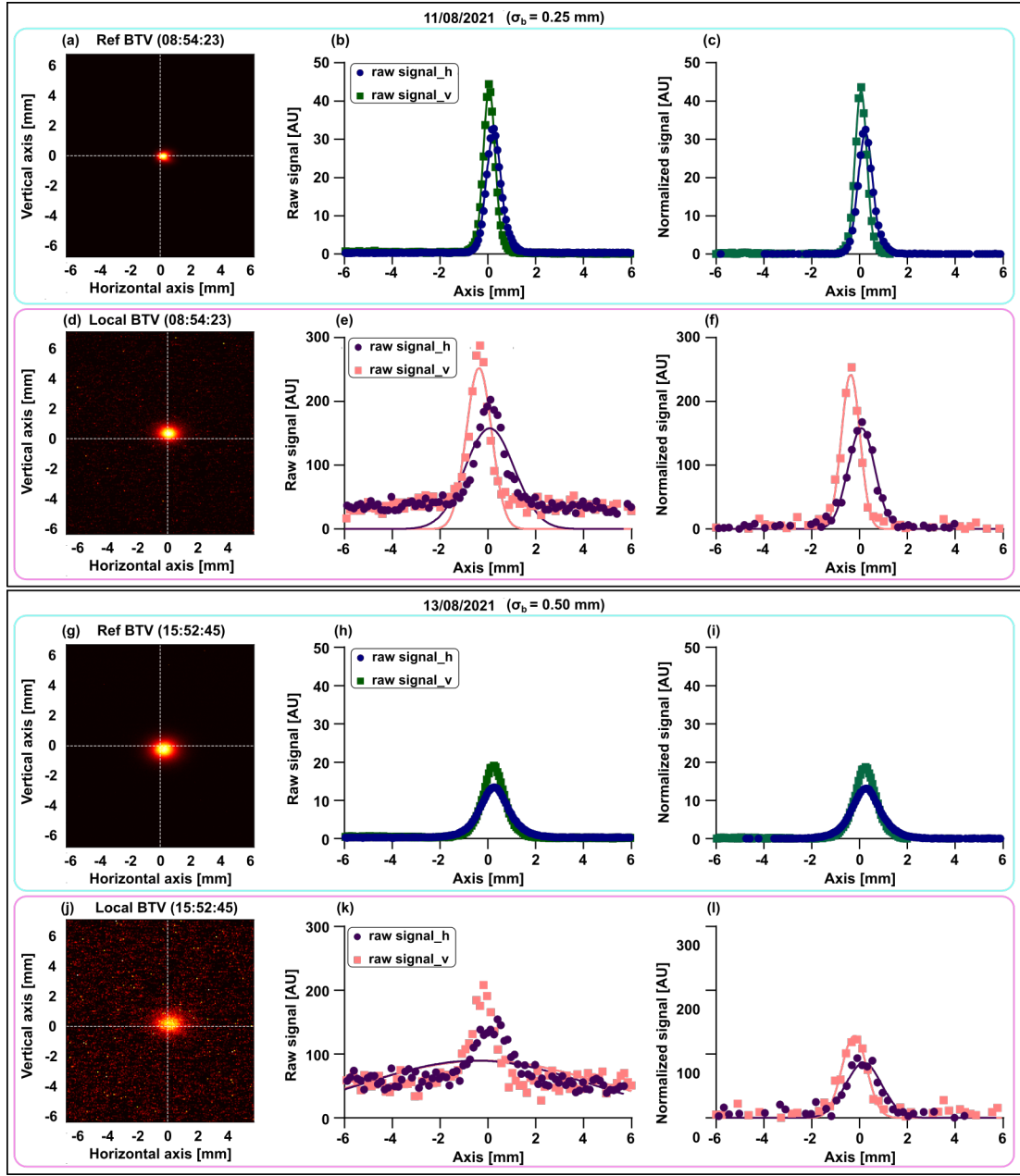


Figure 3.25: Alignment calibration: (a,d,g,j) Images of the reference screen (a,g) and the in-situ screen (d,j) with the beam spot for the first run (11/08/2021- $\sigma_b = 0.25$ mm) and second run (11/08/2021- $\sigma_b = 0.25$ mm) respectively ; (b,e,h,k) Raw signal of the horizontal (circle) and vertical (square) projections and gaussian fit for the first and second run respectively; (c,f,i,k) Normalized signal of the horizontal (circle) and vertical (square) projections and gaussian fit for the first and second run.

For a given condition, the number of protons going through the wire depends on the diameter of the wire, its position relative to the beam position and the beam size (Fig. 3.27). Each plot indicates the relative position of the wire to the beam on the horizontal axis, for both conditions of beam size (0.25 and 0.50 mm) and type

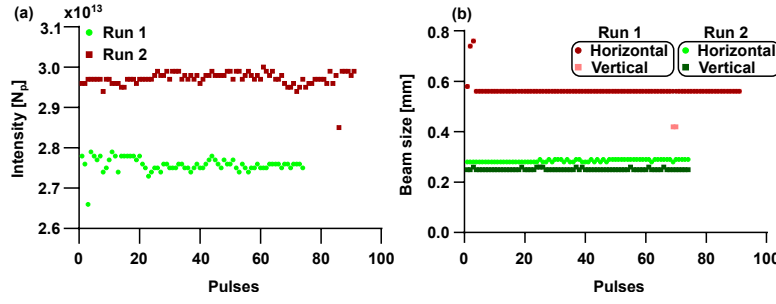


Figure 3.26: (a) Beam intensity measured by the BCT for the first run (green) and the second run (red); (b) Horizontal and vertical beam size (circle and square) beam size analyzed from the BTV for the first run (green) and the second run (red).

of wire (thin and thick). The fraction of the gaussian seen by the wire represents 1.5 % and 3.4 % for the conditions 0.25 μ m thin and 0.25 μ m thick respectively, leading to an average number of protons of $4.31 \cdot 10^{11}$ and $9.38 \cdot 10^{11}$ respectively. For the large beam size, the fraction of the gaussian is 0.7 % and 1.5 % and the intensities crossing the wires are $2.09 \cdot 10^{11}$ and $4.46 \cdot 10^{11}$ respectively.

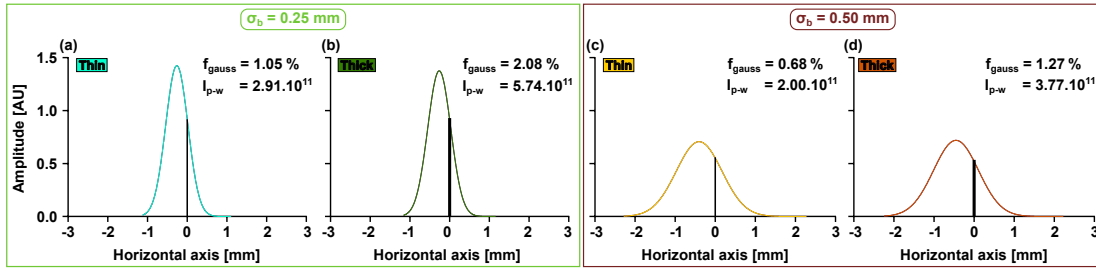


Figure 3.27: Fraction of the wire crossed by the beam for the following conditions: (a) average diameter of 11 μ m for the thin batch, considering an average beam size of 0.29 mm, a beam center at -0.26 mm and an average pulse intensity of $2.76 \cdot 10^{13}$; (b) average diameter of 24 μ m for the thick batch, considering an average beam size of 0.28 mm, a beam center at -0.26 mm and an average pulse intensity of $2.75 \cdot 10^{13}$; (c) average diameter of 11 μ m, average beam size of 0.57 mm, beam center at -0.40 mm and an average pulse intensity of $2.97 \cdot 10^{13}$; (d) average diameter of 24 μ m, average beam size of 0.56 mm, a beam center at -0.45 mm and an average pulse intensity of $2.97 \cdot 10^{13}$.

In this first part, we described the method allowing to evaluate the number of protons of the beam which interact with the wire. We started with a theoretical analysis by considering the beam as perfectly Gaussian in both directions of the transverse plane.

By making the non-negligible assumption that the samples are perfectly cylindrical, we have defined the dose deposited in the wires. To be able to calculate the dose deposited for each sample, we analyzed the real sizes and positions of the beam by comparing the images of the same beam on two aligned BTVs (one reference for the HiRadMat line and one local, positioned on our experience). After a normalization to obtain a clean signal, we were able to deduce the difference between horizontal centering allowing the calculation of the real position of the

Table 3.7: Average horizontal beam size (σ_h), center (μ_h) and pulse intensity ($\bar{I}_{pulse}$) and standard deviation for both theoretical beam size (0.25 mm and 0.50 mm) and both wire type (thin, thick) conditions.

	σ_b [mm]	μ [mm]	$\bar{I}_{pulse}$ [N_p]
$\sigma_b = 0.25$ mm			
Thin	0.29 ± 0.01	-0.26 ± 0.02	$(2.76 \pm 0.012) \cdot 10^{13}$
Thick	0.28 ± 0.01	-0.26 ± 0.03	$(2.75 \pm 0.021) \cdot 10^{13}$
$\sigma_b = 0.50$ mm			
Thin	0.57 ± 0.04	-0.40 ± 0.16	$(2.97 \pm 0.023) \cdot 10^{13}$
Thick	0.56 ± 0.00	-0.45 ± 0.06	$(2.97 \pm 0.012) \cdot 10^{13}$

wire compared to the beam. This information, combined with the knowledge of the real intensity of each pulse, and of the apparent diameter of each wire gives the number of protons crossing the wire.

All subsequent calculations are based on these results.

3.4.2 Signal during irradiation

3.4.2.1 Data selection

Among the raw signals, we observed outliers for some tests. To verify and remove them safely, we sort in ascending order the data and calculate the discriminate function r , for each condition. For $14 \leq n_{samples} \leq 25$, we have

$$r_{22} = \frac{x_3 - x_1}{x_{n-2} - x_1} \quad (3.27)$$

with x_1 the value of the suspect sample.

We had this outliers for 2 wires. On the sample 15 from the first run, thin diameter, we get a raw signal of 374⁸. For the analysis, we consider all samples of the same conditions (samples 8,13,14) leading to the sorted list of: 374, 1512, 1631, 1664, 1704, 1704, 1793, 1887, 2109, 2110, 2315, 2339, 2378, 2430, 2451, 2506, 2506, 2511. The r function for the sample 15 is then 0.59. The Dixon table [326] gives for a test with 18 samples a maximum ratio of 0.36. The value of 374 can then be removed.

The sample 19 from the second run, thick diameter, we observed two outlier values between two normal shots: 413 and 316. Adding the sample 20 we obtain the list: 316, 413, 1850, 1957, 2062, 2255, 2318, 2438, 2512, 2653, 2669, 2885. For

⁸ The signal produced by the SEM-grid and read on the interface during the measurement is a number of counts of ADC (Analog to Digital Converter) and not directly the number of secondary electrons.

$11 \leq n_{samples} \leq 13$, we have

$$r_{21} = \frac{x_3 - x_1}{x_{n-1} - x_1} \quad (3.28)$$

and for 12 samples, the ratio has to be less than 0.42. In our case, we obtain ratios of 0.65 and 0.68 for the values 316 and 413 respectively. However, all the other parameters for these particular pulses are normal. We may suspect an electronic issue.

These values of signal can thus be removed for our signal analysis, but we still need to consider the intensity crossed by the wires.

3.4.2.2 Data analysis

As the normalization takes into consideration the number of particles going through the wires, we can calculate it for each condition with the knowledge of the intensity, the diameter of the wire, the center position and the size of the beam (*cf Annex table. A.6*). From the energy deposition obtained with the Fluka simulations, we calculated the stopping power (*Table. 3.3*). The secondary electron yield (SEY) can then be defined by the *equation. (1.43)*

$$SEY = 0.01L_s \frac{dE}{dx} \bigg|_{el} \left[1 + \frac{1}{1 + 5.4 \cdot 10^{-6} \left(\frac{E_k}{m_p} \right)} \right] \quad (3.29)$$

The secondary electron emission is a surface effect. When an incident particle crosses matter, it has a certain probability to create secondary electron of low energy which can escape from the surface.

The *figure. 3.28* represents the average raw signal for all the pulses seen by the wire, recovered by the SEM-grid, for each wire (red circles for thin wires and blue squares for thick wires) as a function of the sample diameter, for both runs (, filled for the beam size of 0.25 mm and empty for the beam size of 0.50 mm).

Each condition (beam size - type of wire) will be denominated as R1t and R1T for the first run with the small beam size with thin and thick wires respectively, and R2t and R2T for the second run with the large beam size with thin and thick wires.

For the four conditions, we observe homogeneous signals, with an average raw signal for R1t of $(-2.11 \pm 0.34) \cdot 10^3$, $(-2.42 \pm 0.41) \cdot 10^3$ for R1T, $(-2.02 \pm 0.36) \cdot 10^3$ for R2t and $(-2.48 \pm 0.42) \cdot 10^3$ for R2T. The average signal is more consistent for large beam size than for small beam size (*Fig. 3.28*). Surprisingly, after several tries on both carbon fibers of 7 and 34 μm , we did not get any signal, that is why they do not appear in the analysis.

These results show no clear influence of the diameter on the raw signal. To take into account the various parameters assumed (beam size, beam position, beam intensity, wire diameter), we normalize by the number of interaction calculated with

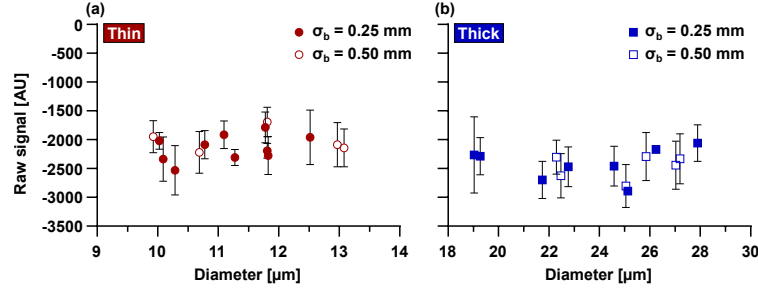


Figure 3.28: Raw signal measured by the SEM-grid as a function of the measured apparent diameter: (a) for thin wires (red); (b) for thick wires (blue), and both runs (filled symbols for the first run ($\sigma_b = 0.25$ mm) and empty symbols for the second run ($\sigma_b = 0.50$ mm)).

the equation. (3.24). The normalized signal for the secondary electron emission measurement is then

$$\|s\| = \frac{\text{Signal}_{SEM}}{N_{ic}} \quad (3.30)$$

This normalization, which considers the real intensity for each pulse, the beam position and the real diameter clearly distinguish both groups for each type of wires. The signal decreases linearly with the increase of the diameter (Fig. 3.29). The slopes for both runs of the thin wires are sensitively equally (-0.29 and -0.27 respectively). The high r squared (0.83 and 0.75) indicate a good quality of this fit. For thick wires, we also observe two groups corresponding to the different runs.

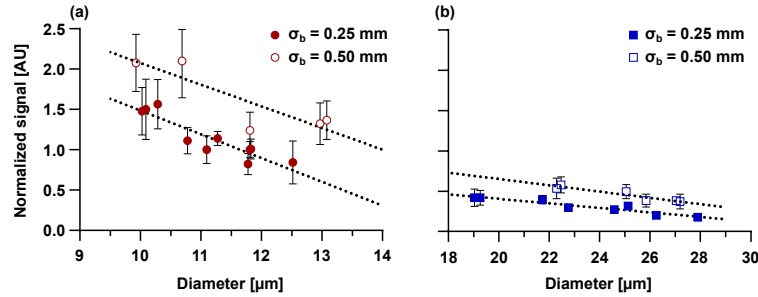


Figure 3.29: Normalized signal measured by the SEM-grid for both runs and both wire types: (a) thin wires; (b) thick wires.

The observation of a decrease in the normalized signal with increasing diameter is quite counter-intuitive. The electrons able to escape from the wire and thus contributing to the SEY are contained in a surface defined by

$$S_{es} = \pi \left(\frac{d}{2} \right)^2 - \pi \left(L_s - \left(\frac{d}{2} \right) \right)^2 \quad (3.31)$$

with d the diameter of the wire and L_s the characteristic length (Eq. (1.44)). This surface is therefore expressed as

$$S_{es} = \pi L_s (d - L_s) \quad (3.32)$$

Hence we should have a linear evolution of the normalized signal with a positive slope of 15.57 taking $L_s = 4.96 \text{ }\mu\text{m}$, to be compared with the negative slopes of the [figure. 3.29](#).

This highlights the limits of the assumptions made first on the perfect cylindricity of the samples. There is most likely a very high variability in the geometry of the samples relative to each other and second the density of 0.8 g.cm^{-3} is probably not exact (but unfortunately impossible to measure).

We varied the number of pulses in order to gradually increase the dose deposited both in a sample by successive pulses, and between samples. The evolution of the normalized signal as a function of the cumulative dose for a sample shows two different behaviors between the thin wires and the thick wires. For thin wires, a fairly random evolution is observed without a clear trend, with a majority of samples producing a zigzag signal. At its highest, the signal variation can reach 93 % ([Fig. 3.30.a](#)). The average normalized signal is plotted against the average dose for thin wires in the [figure. 3.30.b](#). We observe large variations between the samples, with an average value of the normalized signal of 1.32 ± 0.40 . For thick wires, the signal is more constant with the increase of the cumulative dose and the variation between samples is smaller than for the thin wires ([figure. 3.30.c](#)). The average signal is 0.37 ± 0.12 , with a range between the maximum and the minimum value of 0.40.

For each sample, we do not observe obvious regular increase of the signal with the increase of the integrated intensity, leading to the supposition that there is no modification of the structure. As the secondary electron emission is a surface effect, we assume that the samples are not modified by the beam.

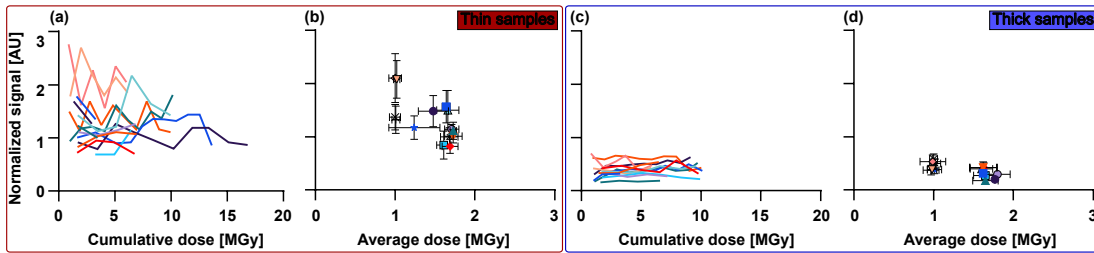


Figure 3.30: Evolution of the normalized signal with the cumulative dose: (a) for thin and (b) for thick wires. Average normalized signal against average dose for each sample: (c) of thin wires and (d) of thick wires.

Theoretically, if the thin and thick wires would have the exact same density and a perfect regular cross-section, the normalized signal should be the same, which is clearly not the case.

At the start of the experiment, we checked and measured the apparent "diameter" given by the 2D projection by optical microscope. After the surprising results of secondary electron emission, in particular for thin wires, we carried out electron microscope observations, including cross-sections and 3D observation. The results

obtained show for thin wires an apparent diameter of 13 μm (Fig. 3.31.a). The 3D view shows a foil structure with a width of 13 μm corresponding to the diameter seen in 2D projection, but a height of 42 μm and a thickness of 450 nm (Fig. 3.31.b). For thick wires we observe an apparent diameter of 28 μm (Fig. 3.31.c) and the cross section gives indications on the precise shape of the wire (at the place of the cut) (Fig. 3.31 .d).

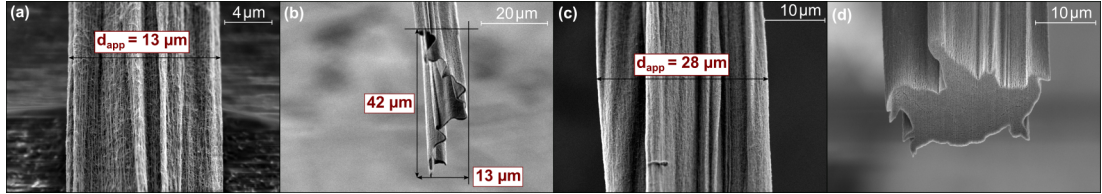


Figure 3.31: SEM micrographs of samples of reference: (a) for a thin wire on top view; (b) 3D view and cross-section of the same thin wire; (c) top view of a thick wire; (d) 3D view and cross-section of the same thick wire.

These observations make it possible to give a hypothesis on the non-reproducibility and the large variability of the results of thin wires. First of all, the amount of matter interacting with the beam could vary greatly from one sample to another and secondly the fact of having a small thickness could allow the emission of a greater number of secondary electrons than in the case of a perfectly circular section. For thick wires, the section seems to be more homogeneous although far from being perfectly circular. These variations in geometry could explain the variations of the signal responses but overall the quantity of material is independent of the apparent diameter (diameters we measured).

3.4.3 Post-irradiation analysis

3.4.3.1 Geometry of the wires

After the irradiation test, the samples were collected for observation and mechanical testing. All the wires seem to have been impacted, more or less significantly. We observe a local deformation, at the precise location of the impact of the beam (Fig. 3.32-top pictures) and degradation (Fig. 3.33).

To study the deformation of each wire, the image was binarized using the ImageJ free software. The binarized images were imported on Python and treated with a in-house code. We calculated the number of black pixels per column along the wire axis, represented by the black curve (Fig. 3.32). The red curve is a correction of the black curve minus the mean diameter of the non-irradiated sections of the wires. The purple curves are the gaussian fit of the red curves.

We observe four characteristic kind of modification of the wire surface (the individual profiles can be found in the *Annexes Fig. A.4 and Fig. A.5*:

- 1- A few CNTs are separated from the surface, with minor variation of the profile (Fig. 3.32.a).
- 2- A deformation appears, in the shape of a Gaussian profile, but the surface seems intact (Fig. 3.32.b).
- 3- The degradation is initiated on the beginning of the deformation (Fig. 3.32.c).
- 4- The degradation propagates along the deformation (Fig. 3.32.d).

By degradation we mean rapid variations of the superficial structure, with sharp discontinuities of the profile. We basically observe the first kind for w1 (wire tested under low intensity), w5 and w6 (low dose < 3.5 MGy), w27 (10 pulses / 9.73 MGy) and w31 (10 pulses / 10.15 MGy). The second kind is observed for only 2 wires : w3 (very low dose : 1.73 MGy) and w32 (10 pulses / 9.32 MGy). The most frequent level in our experiment is the number three, with an initiation of the degradation on the side of the deformation. The kind of full degradation appeared obviously for w24 (10 pulses / 11.97 MGy).

These results are surprising and the explanation is not obvious. An hypothesis would be that the real geometry of the wire as well as the local distribution of the residual catalytic particles of iron plays a role in the mechanism of degradation.

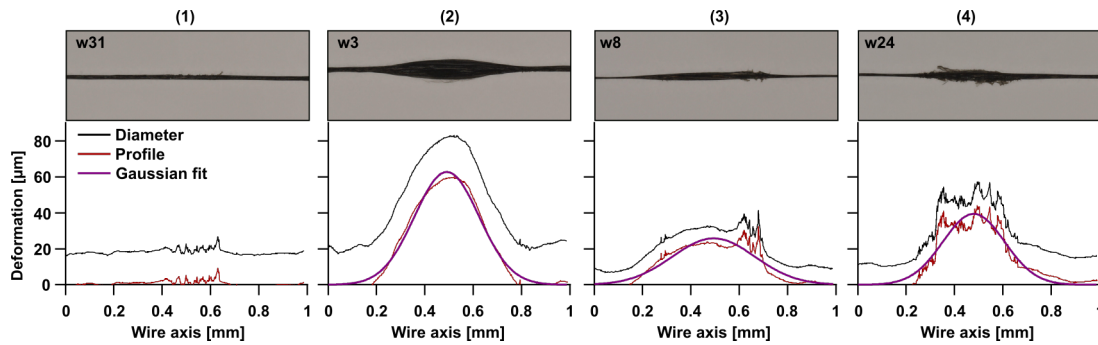


Figure 3.32: Kinds of degradation for the CNT wires during irradiation: (1) CNTs are separated from the surface with no deformation; (2) Deformation of the wire under the form of a Gaussian shape; (3) initiation of the degradation on the side of the deformation; (4) Propagation of the degradation along the deformation.

After obtaining the gaussian fit for each sample, we plot the amplitude and the standard deviation (σ_{fit}) as a function of the total dose in the samples. Concerning the amplitude of the Gaussian, we observe for the thin wires a tendency to increase with the increase of the dose deposited in the wire with a slope of the linear regression quite similar for the thin and thick wires (Fig. 3.34.a). As for the standard deviation, it seems to increase slightly for thin wires and almost constant for thick wires (Fig. 3.34.b). We clearly observe differences between thin and thick wires with an average amplitude for thick wires larger by a factor 2.9 than for the thin wires, and a standard deviation for thick wires 25 % larger than for thin wires (Fig. 3.34.c). However, these linear tendencies have to be carefully considered as the r -squared is very low (<0.5).

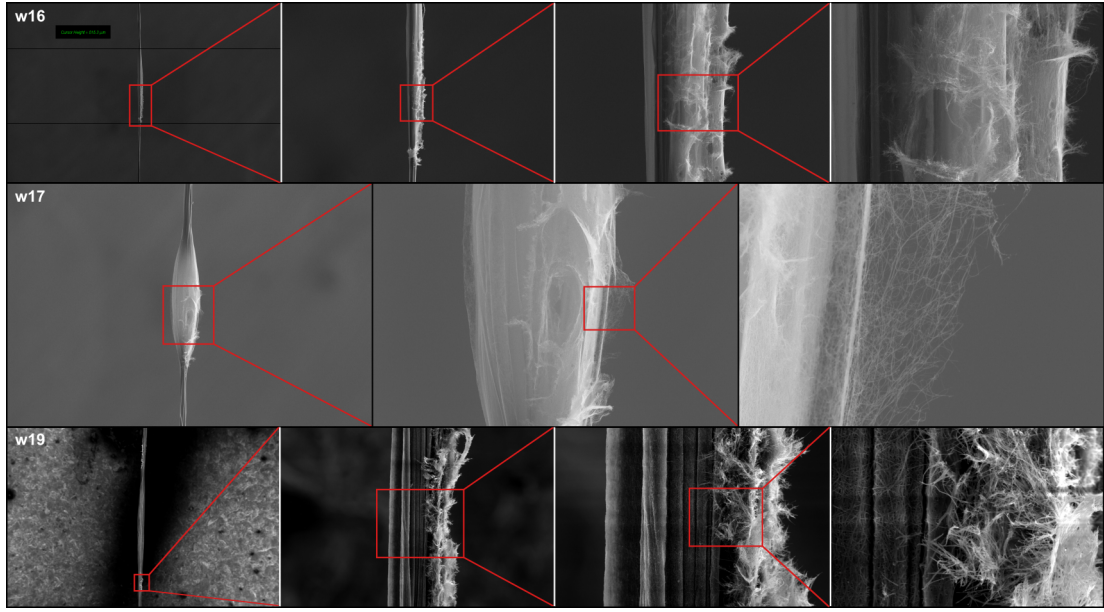


Figure 3.33: SEM pictures with progressive zoom after irradiation of the samples: w16 (from x60 to x3k), w17 (from x100 to x3k) and w19 (from x60 to x10k).

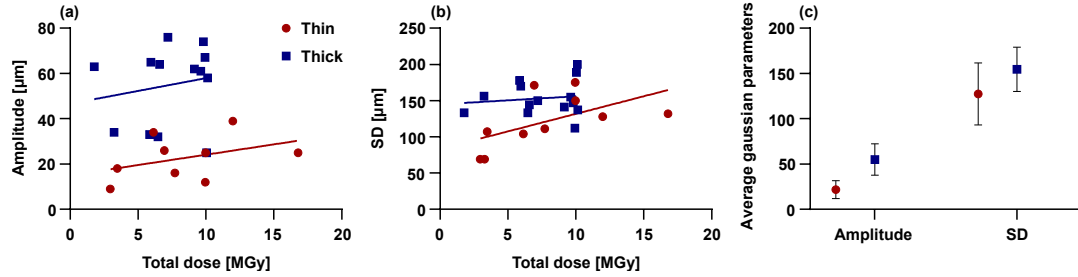


Figure 3.34: Parameters of the fit for thin and thick wires as a function of the dose deposited in the wire: (a) Amplitude of the Gaussian fit; (b) Standard deviation (SD) of the Gaussian fit; (c) Average value for the amplitude and the SD.

The wires deform in different stages. First of all, if the structure and geometry allow it, the wire deforms locally where the wire has been in interaction with the beam. It bulges and takes a Gaussian shape. Secondly, one of the outer edges of the deformation is weakened and damage appears. The parts of the wire that break first must have predispositions to degradation. These predispositions can be of a structural nature: local defects and/or greater or lesser presence of iron. The most probable explanation for these deformations would be that the temperature reached during the test exceeds the transition temperature of iron. The residual particles would melt and migrate guided by the temperature gradient. The particles agglomerate and fit between the tubes and bundles to form larger structures. During their cooling, these particles push outwards the various sub-elements (tube, bundles, fibers) which break under excessive stress. This type of deformation has already been observed after irradiation by laser and white light flash camera [327].

From these profiles and these parameters, we analyzed the ratio between the increase of the surface defined as the area under the gaussian fit and the original apparent surface, as a function of the dose deposited in the wires. To do so, we assume that the wires have a constant diameter. The initial apparent surface is then defined as $S_{a-i} = dl_w$ with d and l_w the diameter and the length of the wire respectively. The increase of the surface of the deformed wire is defined as

$$S_g = a \int_{-\infty}^{+\infty} \exp\left(-\frac{1}{2} \frac{(x - \mu)^2}{\sigma_b^2}\right) dx = a\sigma_b\sqrt{2\pi} \quad (3.33)$$

The ratio between the initial apparent area and the additional apparent area after irradiation as a function of the total dose deposited in the wires is plotted in the figure. 3.35. A tendency to increase with increasing dose is observed for both types of wires. Note that the variations are large and that the r -squared of the linear fits are very low with 0.2 and 0.1 respectively). The average ratios are 1.1 ± 0.8 and 1.9 ± 0.8 for thin and thick wires respectively, indicating that thick wires could have a possible greater propensity to deform than thin wires.

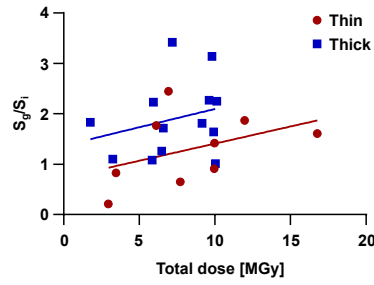


Figure 3.35: Ratio of the apparent surface increase with the initial surface as a function of the total dose deposited in the wire.

These results are difficult to interpret and find a logical connection with the dose is not easy. We could have expected either an increase of the ratio meaning that a higher dose in the wire breaks bonds between tubes and bundles and free fibers or a decrease probably to to ablation and vaporization of material. For thick wires, there is a slight increase if we make a linear fit, but the low r -squared indicates a poor robustness of the results.

3.4.3.2 Structure analysis

In order to study the possible structural modifications after the irradiation, we made an analysis by Raman spectroscopy. Analysis by Raman spectroscopy is a non-destructive method that allows the characterization of the chemical and molecular composition of the external structure of the material studied. Each material with a specific structure has its own characteristic spectrum. When characterizing Raman spectra of CNTs, it is important to study the similarities and differences of Raman spectra among graphene-related structures [328].

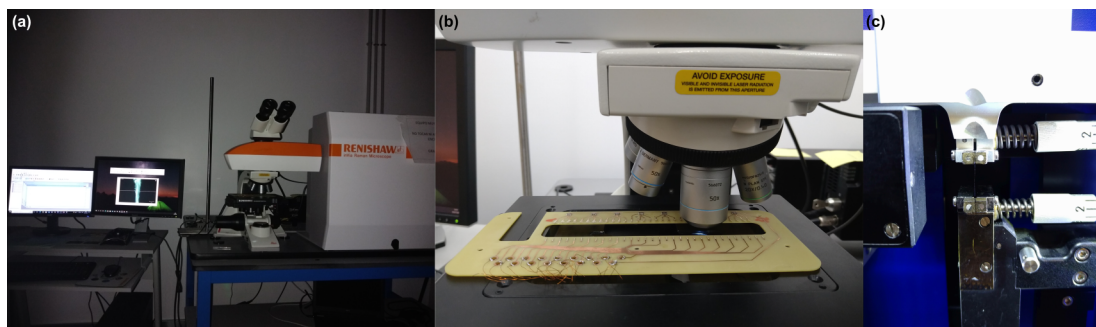


Figure 3.36: Machines for post-irradiation tests: (a) Renishaw inVia Raman Microscope with a 532 nm wavelength laser; (b) Samples mounted on the grid under observation; (c) Textechno Favimat with load cell of 1 N used for the tensile tests.

The characterization was done with a Renishaw⁹ inVia™ Raman microscope with an 532 nm wavelength laser. We performed analysis on the irradiated wires in three distinct zones: end of the wire (10 mm from the impact zone), beginning of the deformation and center of the deformation, for thin and thick samples. The spectra obtained for irradiated wires are characteristic spectra of CNT wires with three characteristic peaks at 1350, 1560 and 2700 cm^{-1} for the D, G and 2D band respectively and two secondary peaks at 2440 and 2940 cm^{-1} (Fig. 3.37.a). The D peak is attributed to the sp^2 atom ring breathing mode while the G peak corresponds to the bond stretching in rings and chains. The ratio between the D peak intensity and the G peak evaluates the disorder in carbon system.

We normalized the spectrum to get the maximal intensity at 1 for the G peak. To characterize the structure quality of the CNTs, we look at the $I_D/I_G = I_D$ ratio for the three positions (Fig. 3.37.b). We observe for the thin wires a decrease of the peak intensity of 40 % between the far and damage center position, with values of 0.2019 and 0.1424 respectively. For thick diameters, the decrease is only 16 % with values of 0.2508 and 0.2161.

The decrease of the I_D/I_G ratio indicates a change in the structure of the CNTs. It has been shown that the change of ratio intensity is related to the pyrolysis and the graphitization state. First occurs pyrolysis ($470 \text{ K} \leq T \leq 1270 \text{ K}$) inducing an increase in the ratio I_D/I_G with increasing temperature. This is followed by the graphitization regime during which the I_D/I_G ratio decreases with increasing temperature [329, 330].

These observations could therefore indicate that the temperatures reached locally allows the graphitization of the structure. Local defects could be removed and/or used to create covalent bonds between CNTs and between bundles. This would explain that for all the irradiated wires, the specific strength tends to in-

⁹ <https://www.renishaw.com>

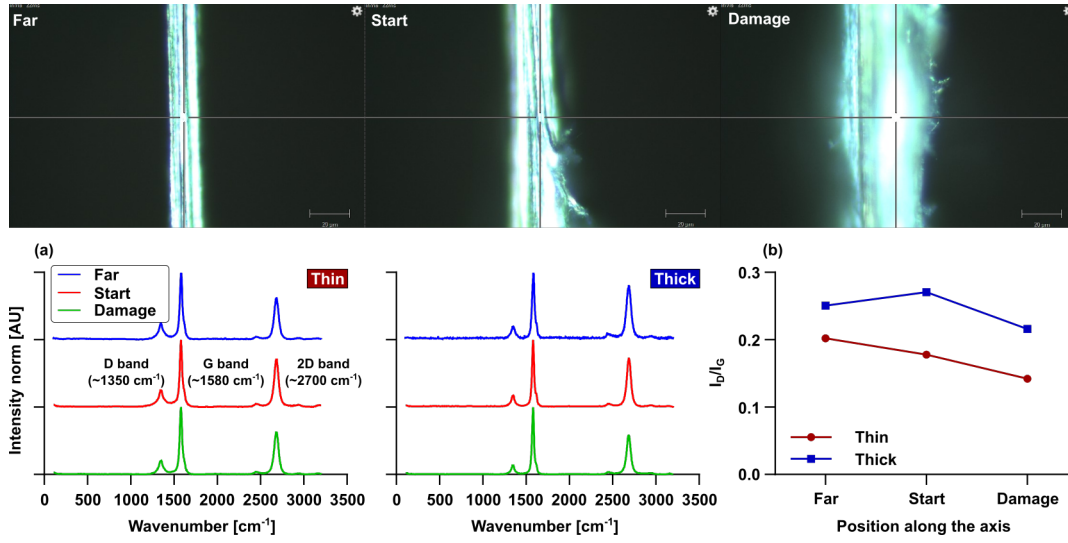


Figure 3.37: Pictures of the wires under the Raman laser for the three locations (far, start of the deformation, middle of the deformation). (a) Raman spectra for thin and thick wires for three different position along the axis: analysis area far from the beam spot (10 mm) (blue line), beginning of the deformation (red line) and middle of the deformation (green line); (b) Evolution of the I_D/I_G ratio for the three locations along the axis of the sample.

crease with the deposited dose, but that overall the mechanical properties are degraded compared to a pristine wire by the creation of local areas of fragility, as we shall see in the next topic.

3.4.3.3 Mechanical analysis

Since the final aim of this project was to study the possible use of this type of wires for beam instrumentation, and more particularly in wire-scanners, we tested the mechanical response in tension after irradiation. It has been shown [270] that gamma ray irradiation between 100 and 700 kGy improves the mechanical properties of CNT wires by about 20 % compared to a pristine wire. This seems to come from the creation of strong covalent bonds between individual CNTs and between bundles.

Our concern was to assess if the CNT wires will have the same response to a proton irradiation at 440 GeV.c⁻¹ and an intensity in the range of 2.7.10¹³.

We tested remaining samples in tension using a Textechno¹⁰ Favimat machine with a load cell of 1 N (Fig. 3.36.c). The tensile speed was 0.2 mm.min⁻¹. The specific strength - strain curves for the CNT wire, thin and thick are plotted in the figure. 3.38.a,b. We also tested the only 34 μm carbon fiber (Fig. 3.38.c). Unfortunately, the 7 μm carbon fiber broke during the installation on the tensile machine.

¹⁰ <https://www.textechno.com>

Despite some variations in the results on the specific strength, specific modulus and strain to failure, both type of wire groups are clear and we can recognize the type of wire with the tensile curve. By comparison with the diameters we measured, everything fits well, except for the sample w16. It was measured at 10 μm (thin batch), but present a curve similar to the thick wires with a clear plastic deformation part and a load to failure closer to the thick wires. We decide not to consider this sample in the analysis of the average.

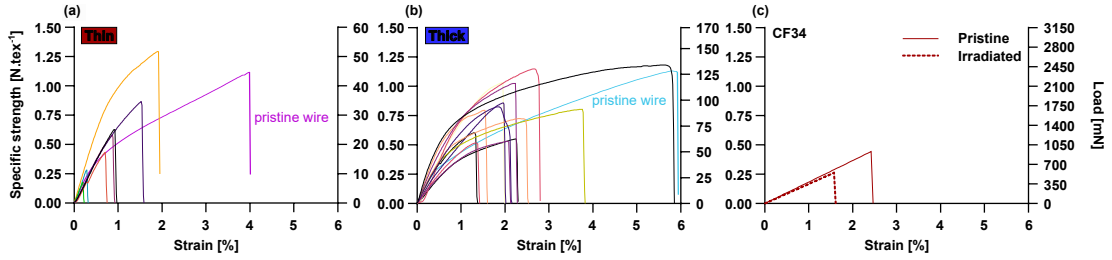


Figure 3.38: Raw tensile curves after irradiation for: (a) Thin wires and (b) Thick wires, with typical curves of pristine samples (purple for the thin wire and turquoise for the thick wire); (c) Carbon fiber of 34 μm .

First, we clearly observe a difference between both types of wires, thin and thick, with an average strain to failure twice as high for thick yarns as for thin wires with a value of 2.48 ± 1.20 % against 1.13 ± 0.70 %, average maximum breaking forces of 19.87 ± 10.02 mN and 92.41 ± 26.04 mN respectively. Considering linear densities of 0.04 g.tex^{-1} for thin wires and 0.114 g.tex^{-1} for thick yarns, the specific strengths are therefore worth on average $0.50 \pm 0.25 \text{ N.tex}^{-1}$ and $0.89 \pm 0.29 \text{ N.tex}^{-1}$ respectively. The specific modulus is higher by 9 % for thick wires than for thin wires.

Concerning the sample w16, we measured a load to failure of 51.8 mN, which is more than twice the average value for the thin samples. If we consider it as a thin sample, the specific strength would be 1.29 N.tex^{-1} and the specific modulus 126 N.tex^{-1} , which is much higher than what we found for the thin samples. If we consider it as a thick wire, the specific strength and the specific modulus are 0.45 and 44 N.tex^{-1} respectively. These values are lower than what we found for the thick batch. An explanation would be that this wire is a wire of "transition" between both batches with a very irregular and anisotropic a cross-section larger than the thin wires and smaller than the thick wires (Table. 3.8).

The carbon fiber shows a typical linear increase before the failure, without plastic deformation.

Between the pristine wires and the irradiated wires, we observe a decrease by 71 % and 63 % for the strain to failure for the thin and the thick wires respectively. The specific strength decreases by 58 % for the thin wires and 19 % for the thick wires after the irradiation. Interestingly, the irradiation does not seem to impact the specific modulus for the thick wires with a variation of only 1 %, which is

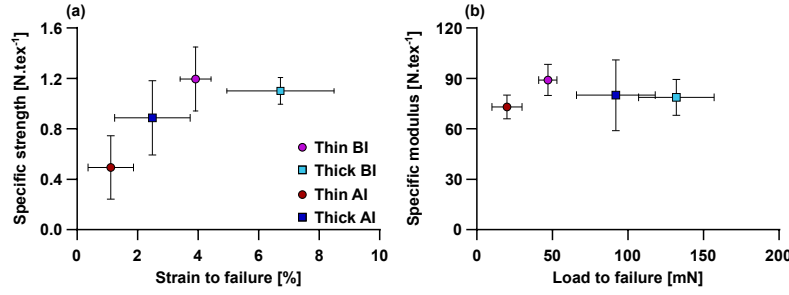


Figure 3.39: (a) Average specific strength; (b) Average specific modulus for thin (round) and thick (square) wires, before and after irradiation.

completely in the error area, whereas for the thin wires, there is a decrease by 18 %. The carbon fiber shows a decrease of the specific strength by 33 % after the irradiation and 26 % for the strain to failure.

Table 3.8: Maximal strain to failure, load to failure, specific strength (SS) and specific modulus (SM) for both thin and thick wires. Same parameters for the sample w16 with the assumption of being a thin or a thick wire.

	ϵ_{max} [%]	F_{max} [mN]	SS [N.tex ⁻¹]	SM [N.tex ⁻¹]
Thin	1.13 ± 0.70	20 ± 10	0.50 ± 0.25	73 ± 7
Thick	2.48 ± 1.20	92 ± 26	0.89 ± 0.29	80 ± 21
CF34	1.78	622	0.29	27
w16 (thin)	1.95	51.79	1.29	126
w16 (thick)	1.95	51.79	0.45	44

Now we consider the mechanical properties for each wire as a function of the dose deposited (Fig. 3.40.a.b.c) and as a function of the increase of surface ratio (Fig. 3.40.a.b.c). For both cases, the results do not show clear tendencies.

In the same way as for the analysis of secondary electrons as a function of the deposited dose, the results are unfortunately not conclusive to draw any conclusion on the impact of irradiation on the mechanical properties of CNT wires. Failing to provide a clear and definitive answer on this point, we have highlighted a determining factor in this type of test and analysis: the geometry of the sample.

In this part, we focused on the geometrical, structural state and the modifications of the mechanical properties of CNT wires after irradiation. A clear observation is that the irradiation has a direct impact on the shape of the sample, with local deformation at the location of the impact zone. This deformation is more or less significant depending on the samples. Different types of degradation were observed, ranging from a few CNTs detached from the wire to a major deformation with rupture of the continuity of the wire along this deformation. Unfortunately, the correlation between deposited dose and relative surface increase is not at all obvious.

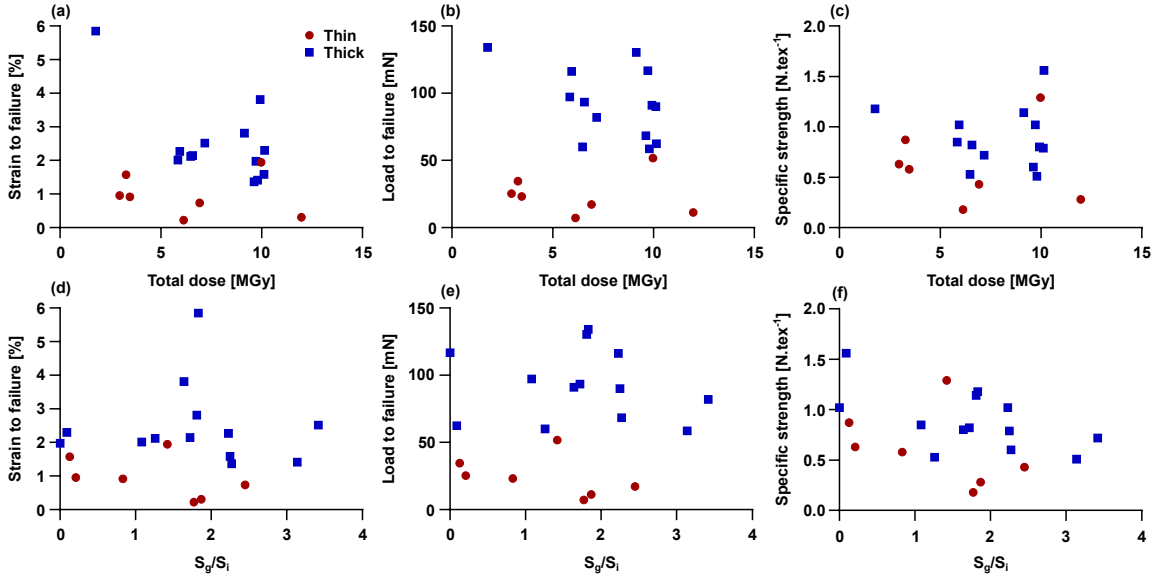


Figure 3.40: Mechanical properties of CNT wires after irradiation as a function of the total dose and the increase of surface ratio: (a,d) Strain to failure; (b,e) Load to failure; (c,f) specific strength.

To understand where these deformations come from, we performed Raman analyzes at different points of the wire. The results showed no obvious differences, indicating that the structural integrity was not altered. Finally, we carried out tensile tests which revealed a reduction in mechanical properties, both in terms of strain to failure, but also for specific strength. But unfortunately, in the same way as for the study of the emission of secondary electrons, the link between the evolution of these properties and the dose deposited in the wire is not conclusive.

3.5 CONCLUSION & GUIDELINES

The objective of the experiment in HiRadMat was to characterize the physical and mechanical properties (secondary electron emission) of CNTY produced by FFCVD after irradiation. 30 CNT wires and 2 carbon fibers of 7 and 34 μm were mounted on a grid.

Models of particle/matter interactions were simulated using Fluka software in order to obtain information on the energy deposition but also on the type of secondary particles emitted for the 4 types of sample: CNT wires: thin (11 μm) and thick (24 μm), carbon fiber: 7 μm and 34 μm . The result was that the secondary particles hitting the scintillator would preferentially be electrons, with average deposited energy given in the [table. 3.4](#).

In order to optimize the number of protons crossing the wire, we combined two BTV, instruments allowing the direct visualization of the beams, as well as the precise knowledge of their center and size. It was shown that despite careful alignment, the wires were slightly offset from the center of the bundle. The analyzes showed that to obtain Gaussian fits with high r -squared, a normalization by the mean value of the background noise was necessary.

Regarding the SEY, the analysis of the signal normalized by the number of protons crossing the wire decreases greatly as a function of the diameter of the wire for thin wires and is almost flat for thick wires. Moreover, the evolution of this normalized signal as a function of the cumulative dose for each sample showed for the thin wires a tendency to remain flat, despite large possible variations within the same sample and between samples. For thick threads, the tendency towards constancy is much clearer and the variations much lower.

We started the post-irradiation analysis by observing the deformations presented by the majority of the samples. We have deduced four phases of in the evolution of the degradation: 1-a few CNTs/bundles are torn from the surface, 2-the wire deforms following a Gaussian profile, 3-the wire begins to degrade more significantly by starting on one side of the deformation, 4- the degradation spreads to the entire deformation with clear fiber breaks and tearing into sheets.

We conclude that these CNT wires (FCCVD method from IMDEA) can not be used in the wire scanners in that state, without additional purification step or better feeding to get more cylindrical cross-section, such as those we used in our mechanical tests described in the [chapter. 2](#). The deformations caused by irradiation would modify the homogeneity of the signal during the scan. We were also able to show the irradiation by protons does not improve the mechanical properties of these CNT wires.

Unfortunately, due to variations in the initial wires, as well as technical difficulties, we were not able to compare the different conditions as was planned in the initial experimental design.

In order to be able to start the HiRadMat experience again by eliminating the unknowns that I faced, I would like to leave some “recommendations” here.

The structure of the wires being the central parameter during the analysis, it is essential to know it perfectly. With this in mind, I recommend the use of purified samples for which the greatest number of parameters are known and controlled (method of manufacturing, type of CNT, diameter, density, remaining impurities (Fe) and mechanical properties). If possible, the samples used should come from the same initial reel. The installation of wires from several suppliers (Hitachi Zosen¹¹, DexMat¹², IMDEA¹³) would allow an excellent comparison. Thus, the pre-irradiation observations may be significant. The diameter and the homogeneity of the structure must be checked under the electron microscope both with 2D projection views and cross-sections via the FIB to check the degree of homogeneity

¹¹ <https://www.hitachizosen.co.jp/english>

¹² <https://dexmat.com>

¹³ <https://materials.imdea.org>

of the wire.

The tensioning of the wires during the installation and fixing of the wires on the grid must be ensured by means of specific tools (not yet developed at CERN, but commercial femtotoools products could be a possibility¹⁴) in order to place them precisely. Each sample should be electrically tested to ensure that the results from each sample will be comparable.

In order to optimize the alignment between the beam and the samples, a calibration must be carried out between the center of the screen of the in-situ BTV, and the center of each wire. During this calibration, if the instrument used is a SEM-grid of the same type as that used in CWCIIH, a return to the home position is necessary after each position overrun (problem of mechanical slack in the endless screw).

During the experiment, the use of the reference BTV is necessary to control the precise position and size of the beam. It is also strongly recommended to have the Python data analysis code (left in EDMS, data storage server at CERN) opened in order to check the stored data in real time and ensure that the data is correctly recorded.

¹⁴ <https://www.femtotoools.com>

General conclusion

CONCLUSION

This low-density material project for beam instrumentation was born from the fact that currently, interceptive instruments such as wire-scanners cannot be used at the maximum intensities available in the SPS and the LHC. Upcoming projects such as HL-LHC and FCC will further increase in intensity and energy, and decrease in beam size, leading to even higher beam densities.

Many studies have been conducted on the choice of materials for wire-scanners, and their thermal and mechanical resistance during a scan. The final choice fell on two types of carbon fiber, with a diameter of 7 and 34 μm . However, two problems arise. The first is that the yarns used were purchased 50 years ago, from a company that no longer exists, and the stock is running out. The second is that with the current wires, at top energy, it is only possible to scan a beam at 0.3 % of its maximum intensity. If it is true that today there are very high resistance and high thermal conductivity carbon fiber, which would meet the many of the specifications, particularly in terms of mechanical deformation and heat evacuation during the scan, these wires have mostly diameters of about 5 nm. However, such small diameters are difficult to handle, and given the current design of the instrument, their installation would be very complicated.

This is why we decided to investigate carbon nanotubes, a particular structure of carbon, which, at the nanoscopic scale, has properties far superior to all the materials currently available. Given their diameter of a few nanometers along with maximum lengths of some micrometers, it is clear that for the moment there is no question of using an individual tube directly. However, in recent years, the technologies for assembling these nanotubes into fibers, yarns, wires and mats have been developing rapidly.

While many studies have been conducted on the influence of various nano and microscopic parameters the mechanical resistance on these micro-assemblies, fewer studies have been conducted on the characterization of thermal properties and very little on the responses to irradiation.

This thesis work, particular, because at the crossroads of several fields of mechanics: materials, design and simulations, and several fields of physics: physics of particles, physics of accelerator aimed to study the behavior of such material under the conditions of irradiation of the SPS and the LHC which was never been before. We set up an experiment on the HiRadMat line in order to determine a physical property (emission of secondary electrons) and the mechanical properties of fracture toughness after irradiation by a high-energy and high-intensity proton beam, combined with low beam sizes. This can only be done at CERN.

In order to verify the possibility of installing CNT wires on wire-scanners in the same way as the carbon fibers currently used, we first studied the influence of copper-coating on the mechanical properties of CNT wires. diameter 10 μm , 20 μm and 30 μm , supplied by Hitachi Zosen (Japan).

A preliminary analysis of the internal structure of these wires was carried out using electron microscopy (SEM) and gallium ion beam (FIB) imaging. Image processing revealed a higher packing factor for the 20 μm diameter than for the 30 μm diameter. A cross section at the level of the copper plating was also carried out in order to observe the interface between the copper and the carbon. We very clearly observe a depth of copper penetration inside the inter-bundle interstices.

We conducted a test campaign using two different types of assembly, the first a pristine wire glued to a paper frame and clamped between two plates, the second copper-plated at its ends. Tensile tests have shown consistent and repeatable results for small diameters and huge variations for 20 μm and 30 μm copper wires. In order to take into account the differences in the effective length of the wires on the mechanical properties obtained, we used a Weibull method to be able to compare the results of the two types of assembly with an operational wire. Based on the results obtained, we were able to derive the failure probabilities for the case of an operational wire, for a pre-stress defined as the pre-load of 30 g applied to a 34 μm wire. The results show a clear degradation of the mechanical properties for the copper wires, and therefore a greater failure probability.

We also studied the influence of the loading speed on the mechanical properties, which do not really show a clear change in the results, despite very different fracture facies.

Subsequently, we looked at the impact of the scan speed on the resolution of the measurement, which showed a limitation of 4.5 m^{-1} for the SPS and 0.6 m.s^{-1} for the LHC. The wire-scanners cannot therefore be used at their nominal speed for these accelerators. Considering the results obtained previously on the properties of the wires, we have modeled the deformation of the wire during a scan, initially by considering a stationary state, and in a second time with a velocity profile which varies according to time to closer to the actual profile. The results show deformations at maximum speed which exceed the required specifications. However, the dynamic analysis has shown that the actual velocity profile used at CERN allows the wire to deform according to the adiabatic regime, ie it follows the instantaneous stationary regime at all times. This makes it possible to achieve an almost zero level of vibration.

Finally, it was shown that in the case of a pristine wire, the amplitude of the maximum deformation of the worst wire remains lower than the amplitude of the maximum deformation of the best copper wire.

It is therefore quite clear that it would ideally be necessary to seek to install wires without copper plating to increase the pre-stress and reduce the deformations.

For this experiment, we used wires provided by IMDEA (Madrid), and synthesized by FCCVD method. We took electron microscope images before the test to estimate the diameter of each sample. We were surprised by the significant

irregularity of the diameter along the wire and the variations between each sample. Since this manufacturing method produces samples that are not repeatable in their structure and geometry, and that it was not possible to cut and observe each sample planned for the experiment, we had to make many assumptions and approximations. We considered 2 batches of CNT wires, the first composed of thin wires and the second of thick wires, with a diameter of 11 and 24 μm on average. Moreover we considered that the wires were perfectly cylindrical. We estimated a volume density of 0.8 g.cm^{-3} for the two types of wires and a mixed composition of 90% carbon and 10% iron by mass.

Before running the experiment, we performed simulations to get an expectation on the results. For particle/matter interaction simulations, we used Fluka software, a code based on a Monte-Carlo method. This software does not allow to model 1D elements such as CNTs. We built a simplified model of the experiment, and varied the diameter, density and composition of the wire to compare the different responses of the wires available. However, the specific structure of the CNTs is not considered, which could have an impact on the emission of secondary electrons as well as on the signal generated in the scintillator, due to a possible "channeling effect" of the electrons in the tubes.

Thanks to the information of the energy deposited in the wires, given by the results of the simulations, we were able to calculate different physical characteristics specific to our samples, with the assumptions of a perfect circular wire and the same behavior for a CNT wire than a carbon fiber. The results showed that the secondary emission yield was effectively the same for the four samples, around $5.416.10^{-3} \text{ e}^{-}.\text{cm}^2/\text{p}_i^{+}$. The necessity of having a high secondary electron emission yield requires using wires with large diameters and low density.

In order to get closer to the reality of wire-scanners in the accelerators, we built a second model by adding an empty line along the entire beam path and a scintillator. We observed the fluence spectrum of the particles entering the scintillator, allowing the reconstruction of the profile during a scan. Electrons are the most predominant in terms of energy.

As the main cause of wire breakage during a scan is the sudden and local increase in temperature, we performed thermal simulations using the data provided by the results of Fluka simulations. To do this, we used a Python code, developed in-house. This very complete code makes it possible to simulate the increase in temperature by taking into account many parameters, and especially by being able to vary the thermal properties according to the temperature. The results show that the maximal temperature reached by the wires widely depend firstly on the thermal properties and secondly on the beam size. The non-consideration of the evolution of the thermal properties with the temperature overestimate the result by a factor 2, with temperatures around 3000 K. However, the results should be taken with caution principally because we had to extrapolate the temperature dependences.

Secondly, we developed an experiment to test these CNT wires on a specific experimental line for this type of test: HiRadMat. Helped by a small team of mechanical

and electronic technicians, as well as two master's trainees, we designed, assembled and integrated the experiment. The crucial point and real challenge of this experiment was to ensure the alignment of the beam and the samples. To do this, we combined the use of different instruments. This makes it possible to know at each pulse the relative position of the wire in the beam, and therefore the fraction of the Gaussian profile seen by the wire.

We performed in-situ measurements of the emission of secondary electrons for two sizes of beams and the two types of wires. The analysis of the results of the signals normalized by the dose deposited during the pulse as a function of the cumulative dose deposited during all the pulses does not show any drastic change. Given the results of the analysis of the 3D microscopic images, where we were able to observe the real geometry of the reference wires (non-irradiated wires), we can provide explanations for the large dispersion in the signals between samples of the same conditions.

The irradiation generated more or less significant structural modifications depending on the samples, represented by a deformation at the point of impact with the beam. Some samples show localized degradation on one side with assemblies of CNTs that detach from the wire. The analysis of the deformations seems to indicate a direct impact of the dose deposited with an increase in the deformation during the increase in the dose, whatever the type of wire. Surprisingly, Raman analyzes showed no structural changes in terms of CNTs. The decrease in the I_D/I_G ratio after irradiation indicates that the wires have fewer defects in term of CNT structure. These deformations, not observed on the carbon fibers, could be a consequence of the residual catalytic particles putting the various sub-elements of the yarn under pressure.

Finally, the samples were tested for tensile strength. Two different behaviors appear between thin and thick wires, with a more pronounced plastic zone for thick wires. The analysis of the maximum breaking loads as a function of the total dose received by each sample does not show any clear evolution for the thin wires, whereas a downward trend for an increase in the dose seems appear for the thick wires. The strain to failure remains generally constant regardless of the dose received.

The objective of this project was to study the possibility of replacing the carbon fibers currently used by CNT yarns. Our experience at HiRadMat has clearly shown the limits of wires produced by FCCVD with deformations from the first pulse. Even if the production of secondary electrons does not seem affected by these deformations, the use of such a wire is not possible for wire-scanners, without significant improvement of its characterization such as purity and cylindricity.

PERSPECTIVES

The replacement of carbon fibers for particle beam instrumentation, in particular the measurement of the transverse profile, seems to be inevitable. However, the materials currently available on the market, whose geometric specifications match our requirements, are far from the optimum properties of the individual CNTs. If the idea is to persist in this way, there will be several areas of study to deepen. First of all, we will continue to work in close collaboration with the various manufacturing players (IMDEA, Bath University) who control the entire process. A working group was set up to develop the ideal material. To do so, the requirements and specifications are given by CERN. Based on these specifications, a group will be in charge of particle/matter interaction simulations as well as thermal responses (Chrono-environment, Université de Franche-Comté). On this occasion, it could be interesting to push research using molecular dynamic models of bundles of CNTs in order to take into account the particular structure of CNTs wires as well as their bonds inside a wire and see their effect on the mechanical properties of the bundles. It will probably take several iterations before having a satisfactory answer, but the conditions of use of these wires are neither trivial nor common. Given the preliminary results, purification post-treatments seem essential. Another non-negotiable issue for the use of a new material is the reproducibility of the wires. It must be possible to be certain that from one wire to another, the physical, chemical, mechanical, thermal and geometric characteristics are the same, within the specified tolerances.

Initially, the experimental design was to attain wire failure. Unfortunately, we were limited by the number of protons available during the experiment, and we were not able to attain failure. We were thus not able to fully study the mechanical properties of the wires, as we are missing the characterization of resistance to irradiation.

The second point to seriously consider is the use of an individual nanotube. If at the moment it seems complicated, it would seem that this path is the best considering all the parameters. However, this would imply major changes in the logistics, implementation and integration of this nanometric object on our instruments. It would then be necessary to review all the installation and handling procedures. Current tools will have to be adapted and new ones will have to be developed. Going from the microscopic scale to the nanoscale involves a profound paradigm shift, but such nano-assembly studies are currently developed (*eg*: FEMTO-ST).

The characterization of these wires will be a major challenge in the future. Given the exceptional working conditions at CERN, and more particularly in the accelerators, each intervention is an ordeal and requires a lot of resources. External qualification upstream of the operational phases is necessary. In this context, the development of a test bench for the characterization of thermal properties (specific heat capacity, thermal conductivity and radiosity) at high temperature should be considered. We started this project with the submission of a proposal for a European grant EU-Attract (2018). Methods have been developed for the

characterization of micrometric carbon fibers at high temperature on the one hand [331, 332, 333, 334], and for CNT wires at room temperature on the other [335, 336]. The idea would be to have a single, modular facility, allowing the testing, characterization and qualification of various micrometric wires, where the use of the femto-tools could be useful.

As seen previously, the copper coating of the wires reduces their mechanical properties. A line of research should also focus on three points. 1- improvement of the process in order to have a final product with a repeatable quality over time (final diameter constant coppering, controlled roughness, hardness). This could be done through an intermediate material allowing better adhesion between the different materials. 2- change of the material allowing brazing on the copper inserts of the forks of the instrument. 3- change of the wire fixing system on the forks, which would protect against any structural and behavioral modification of the wire. Some tests were carried out in front of the beam at the SPS (2017 and 2022), with linear wire-scanners and 30 μm CNT wires. The last wire was installed without copper plating, and fixed to the forks by pinching. Early results have shown a signal indicating that copper plating is not necessary.

I hope this work to be the basis for extensive and complementary studies that will first allow a deeper understanding of the behavior of these carbon nanotube wires under very high energy irradiation.

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Appendix A

Annexes

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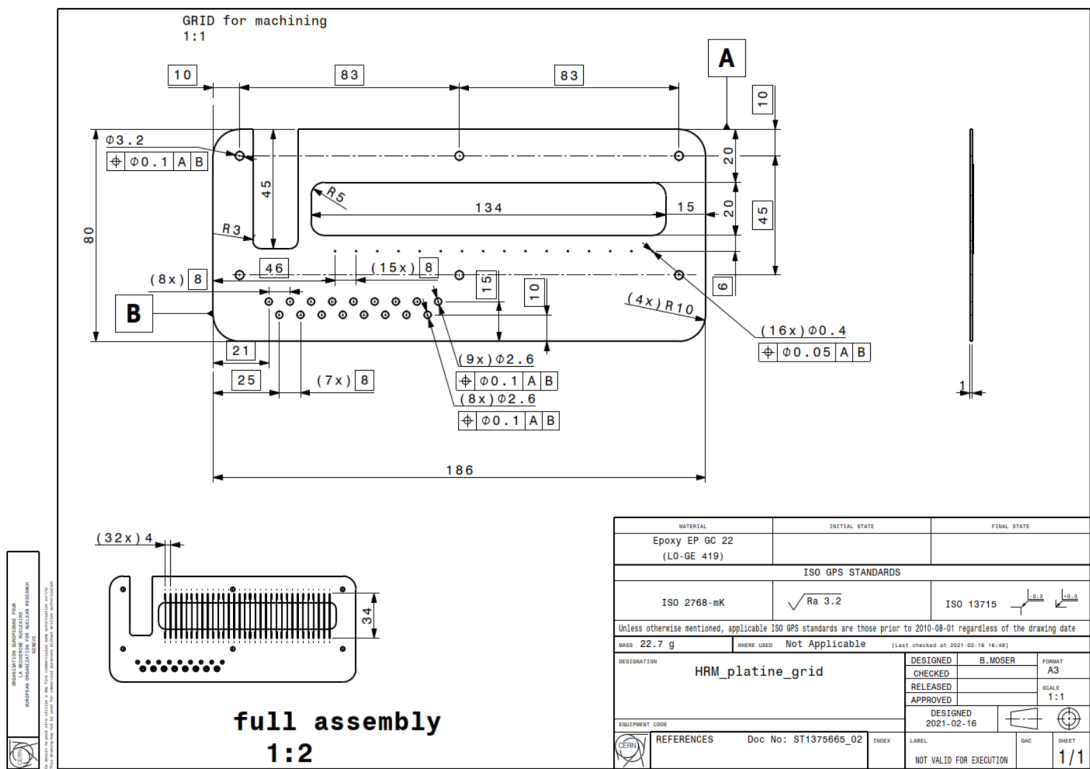


Figure A.2: Drawing of the main grid

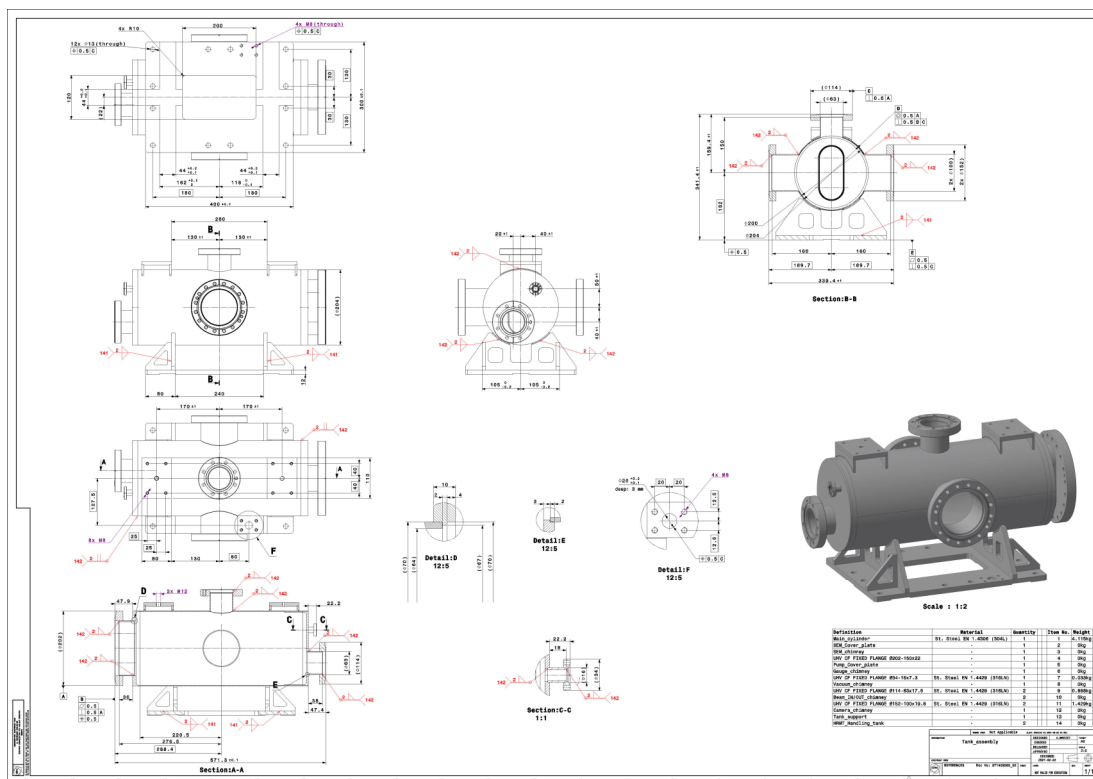


Figure A.3: Drawing of the vacuum tank assembly

A.2 TIMESTAMPS FOR THE SEM/BCT/BTV DATA

Considering a time range the 11th of August, between 11:54:00 and 13:19:00 UTC (13:54 and 15:19 local time zone), we have as follow:

Considering a time range the 13th of August, between 16:30:00 and 18:17:00 UTC (18:30 and 20:17 local time zone), we have as follow:

Table A.1: Timestamp and reference number for the tests with the beam size 0.25 mm (1/2)

Pulse	Nb	BTV	Nb	BCT	Nb	SEM	Wire
N1	1	11:54:52	0	11:54:30	1	11:55:08	w3
N1	2	11:55:30	1	11:55:08	2	11:55:46	w4
N2	3	11:56:09	2	11:55:46	3	11:56:25	w4
N1	4	11:56:47	3	11:56:25	4	11:57:03	w5
N2	5	11:57:25	4	11:57:03	5	11:57:42	w5
N1	6	11:58:04	5	11:57:42	6	11:58:20	w6
N2	7	11:58:42	6	11:58:20	7	11:58:58	w6
N1	8	11:59:21	7	11:58:58	8	11:59:37	w7
N2	9	11:59:59	8	11:59:37	9	12:00:15	w7
N1	10	12:00:37	9	12:00:15	10	12:00:54	w8
N2	14	12:04:28	13	12:04:06	16	12:04:44	w8
N3	15	12:05:06	14	12:04:44	17	12:05:22	w8
N4	19	12:10:13	18	12:09:51	25	12:10:30	w8
N1	20	12:10:52	19	12:10:30	26	12:11:08	w9
N2	21	12:11:30	20	12:11:08	27	12:11:46	w9
N3	22	12:12:09	21	12:11:46	28	12:12:25	w9
N4	24	12:13:25	23	12:13:03	30	12:13:42	w9
N1	25	12:14:04	24	12:13:42	31	12:14:20	w10
N2	26	12:14:42	25	12:14:20	32	12:14:58	w10
N3	27	12:15:21	26	12:14:58	33	12:15:37	w10
N4	28	12:15:59	27	12:15:37	34	12:16:15	w10
N1	32	12:28:09	31	12:27:46	53	12:28:25	w11
N2	34	12:29:25	33	12:29:03	55	12:29:42	w11
N3	36	12:30:42	35	12:30:20	57	12:30:58	w11
N4	38	12:31:59	37	12:31:37	59	12:32:15	w11
N5	39	12:32:37	38	12:32:15	60	12:32:54	w11
N6	42	12:34:33	41	12:34:10	63	12:34:49	w11
N1	43	12:35:11	42	12:34:49	64	12:35:27	w12
N2	44	12:35:49	43	12:35:27	65	12:36:06	w12
N3	45	12:36:28	44	12:36:06	66	12:36:44	w12
N4	46	12:37:06	45	12:36:44	67	12:37:22	w12
N1	47	12:37:45	46	12:37:22	68	12:38:01	w13
N2	48	12:38:23	47	12:38:01	69	12:38:39	w13
N3	49	12:39:01	48	12:38:39	70	12:39:18	w13
N4	50	12:39:40	49	12:39:18	71	12:39:56	w13
N1	53	12:41:35	52	12:41:13	74	12:41:51	w14
N2	54	12:42:13	53	12:41:51	75	12:42:30	w14

Table A.2: Timestamp and reference number for the tests with the beam size 0.25 mm (2/2)

Pulse	Nb	BTV	Nb	BCT	Nb	SEM	Wire
N3	56	12:43:30	55	12:43:08	77	12:43:46	w14
N4	57	12:44:09	56	12:43:46	78	12:44:25	w14
N1	58	12:44:47	57	12:44:25	79	12:45:03	w15
N2	59	12:45:25	58	12:45:03	80	12:45:42	w15
N3	60	12:46:04	59	12:45:42	81	12:46:20	w15
N4	61	12:46:42	60	12:46:20	82	12:46:58	w15
N1	62	12:47:21	61	12:46:58	83	12:47:37	w16
N2	63	12:47:59	62	12:47:37	84	12:48:15	w16
N3	64	12:48:37	63	12:48:15	85	12:48:54	w16
N4	65	12:49:16	64	12:48:54	86	12:49:32	w16
N5	66	12:49:54	65	12:49:32	87	12:50:10	w16
N6	67	12:50:33	66	12:50:10	88	12:50:49	w16
N1	68	12:51:11	67	12:50:49	89	12:51:27	w17
N2	69	12:51:49	68	12:51:27	90	12:52:06	w17
N3	74	13:03:21	73	13:02:58	108	13:03:37	w17
N4	75	13:03:59	74	13:03:37	109	13:04:15	w17
N5	76	13:04:37	75	13:04:15	110	13:04:54	w17
N6	77	13:05:16	76	13:04:54	111	13:05:32	w17
N1	78	13:05:54	77	13:05:32	112	13:06:10	w18
N2	80	13:07:11	79	13:06:49	114	13:07:27	w18
N3	81	13:07:49	80	13:07:27	115	13:08:06	w18
N4	82	13:08:28	81	13:08:06	116	13:08:44	w18
N5	83	13:09:06	82	13:08:44	117	13:09:22	w18
N6	84	13:09:45	83	13:09:22	118	13:10:01	w18
N1	85	13:10:23	84	13:10:01	119	13:10:39	w23
N2	86	13:11:01	85	13:10:39	120	13:11:18	w23
N3	87	13:11:40	86	13:11:18	121	13:11:56	w23
N4	88	13:12:18	87	13:11:56	122	13:12:34	w23
N5	89	13:12:57	88	13:12:34	123	13:13:13	w23
N6	90	13:13:35	89	13:13:13	124	13:13:51	w23
N7	91	13:14:13	90	13:13:51	125	13:14:30	w23
N8	92	13:14:52	91	13:14:30	126	13:15:08	w23
N9	93	13:15:30	92	13:15:08	127	13:15:46	w23
N10	94	13:16:09	93	13:15:46	128	13:16:25	w23
N1	95	13:16:47	94	13:16:25	129	13:17:03	w24
N2	96	13:17:25	95	13:17:03	130	13:17:42	w24
N3	97	13:18:04	96	13:17:42	131	13:18:20	w24

Table A.3: Timestamp and reference number for the tests with the beam size 0.5 mm (1/3)

Pulse	Nb	BTV	Nb	BCT-SEM	Wire
N1	0	16:30:35	1	16:31:06	w24
N2	1	16:31:28	2	16:31:58	w24
N3	3	16:32:21	3	16:32:51	w24
N4	4	16:33:13	4	16:33:44	w24
N5	5	16:34:06	5	16:34:37	w24
N6	6	16:34:59	6	16:35:30	w24
N7	7	16:35:52	7	16:36:22	w24
N8	8	16:36:45	8	16:37:15	w24
N1	9	16:37:37	9	16:38:08	w25
N2	11	16:39:23	11	16:39:54	w25
N3	12	16:40:16	12	16:40:46	w25
N4	13	16:41:09	13	16:41:39	w25
N5	14	16:42:01	14	16:42:32	w25
N6	15	16:42:54	15	16:43:25	w25
N7	16	16:43:47	16	16:44:18	w25
N8	17	16:44:40	17	16:45:10	w25
N9	18	16:45:33	18	16:46:03	w25
N1	23	16:49:57	23	16:50:27	w26
N2	24	16:50:49	24	16:51:20	w26
N3	25	16:51:42	25	16:52:13	w26
N4	26	16:52:35	26	16:53:06	w26
N5	27	16:53:28	27	16:53:58	w26
N6	28	16:54:21	28	16:54:51	w26
N7	29	16:55:13	29	16:55:44	w26
N8	30	16:56:06	30	16:56:37	w26
N9	31	16:56:59	31	16:57:30	w26
N10	32	16:57:52	32	16:58:22	w26
N1	44	17:08:25	44	17:08:56	w27
N2	45	17:09:18	45	17:09:49	w27
N3	46	17:10:11	46	17:10:42	w27
N4	47	17:11:04	47	17:11:34	w27
N5	49	17:12:49	49	17:13:20	w27
N6	51	17:14:35	51	17:15:06	w27
N7	52	17:15:28	52	17:15:58	w27
N8	53	17:16:21	53	17:16:51	w27
N9	54	17:17:13	54	17:17:44	w27
N10	55	17:18:06	55	17:18:37	w27
N1	56	17:18:59	56	17:19:30	w28
N2	58	17:20:45	58	17:21:15	w28
N3	59	17:21:37	59	17:22:08	w28

Table A.4: Timestamp and reference number for the tests with the beam size 0.5 mm (2/3)

Pulse	Nb	BTV	Nb	BCT-SEM	Wire
N4	61	17:23:23	61	17:23:54	w28
N5	62	17:24:16	62	17:24:46	w28
N6	63	17:25:09	63	17:25:39	w28
N7	64	17:26:01	64	17:26:32	w28
N8	65	17:26:54	65	17:27:25	w28
N9	67	17:28:40	67	17:29:10	w28
N10	68	17:29:33	68	17:30:03	w28
N1	69	17:30:25	69	17:30:56	w31
N2	70	17:31:18	70	17:31:49	w31
N3	71	17:32:11	71	17:32:42	w31
N4	72	17:33:04	72	17:33:34	w31
N5	74	17:34:53	74	17:35:27	w31
N6	75	17:35:49	75	17:36:24	w31
N7	76	17:36:46	76	17:37:20	w31
N8	77	17:37:42	77	17:38:16	w31
N9	78	17:38:39	78	17:39:13	w31
N10	79	17:39:35	79	17:40:09	w31
N1	80	17:40:31	80	17:41:06	w32
N2	81	17:41:28	81	17:42:02	w32
N3	82	17:42:24	82	17:42:58	w32
N4	83	17:43:21	83	17:43:55	w32
N5	84	17:44:17	84	17:44:51	w32
N6	85	17:45:13	85	17:45:48	w32
N7	86	17:46:10	86	17:46:44	w32
N8	87	17:47:06	87	17:47:40	w32
N9	88	17:48:03	88	17:48:37	w32
N10	89	17:48:59	89	17:49:33	w32
N1	92	17:51:48	92	17:52:22	w19
N2	93	17:52:45	93	17:53:19	w19
N3	94	17:53:41	94	17:54:15	w19
N4	95	17:54:37	95	17:55:12	w19
N5	96	17:55:34	96	17:56:08	w19
N6	97	17:56:30	97	17:57:04	w19
N1	98	17:57:27	98	17:58:01	w20
N2	99	17:58:23	99	17:58:57	w20
N3	100	17:59:19	100	17:59:54	w20
N4	101	18:00:16	101	18:00:50	w20
N5	102	18:01:12	102	18:01:46	w20
N6	103	18:02:09	103	18:02:43	w20

Table A.5: Timestamp and reference number for the tests with the beam size 0.5 mm
(3/3)

Pulse	Nb	BTV	Nb	BCT-SEM	Wire
N1	104	18:03:05	104	18:03:39	w21
N2	105	18:04:01	105	18:04:36	w21
N3	106	18:04:58	106	18:05:32	w21
N4	107	18:05:54	107	18:06:28	w21
N5	108	18:06:51	108	18:07:25	w21
N6	109	18:07:47	109	18:08:21	w21
N1	110	18:08:43	110	18:09:18	w22
N2	112	18:09:40	112	18:11:10	w22
N3	113	18:11:33	113	18:12:07	w22
N4	115	18:13:25	115	18:14:00	w22
N5	116	18:14:22	116	18:14:56	w22
N6	117	18:15:18	117	18:15:52	w22

Table A.6: Synthesis of the data for thin wires and beam size of 0.25

Wire -	Raw Signal	I_b [N _{prot}]	μ_h [mm]	σ_h [mm]	σ_v [mm]	f_g [%]	Dose [MGy]	Dose total [MGy]
w5	-2124	$2.79 \cdot 10^{13}$	-0.321	0.28	0.25	0.74	26.10	57.54
w5	-1920	$2.78 \cdot 10^{13}$	-0.271	0.28	0.25	0.89	31.43	
w6	-2834	$2.77 \cdot 10^{13}$	-0.271	0.28	0.25	0.92	31.27	63.84
w6	-2226	$2.78 \cdot 10^{13}$	-0.261	0.28	0.25	0.95	32.57	
w7	-2260	$2.74 \cdot 10^{13}$	-0.251	0.28	0.25	1.03	33.10	67.33
w7	-1908	$2.75 \cdot 10^{13}$	-0.241	0.28	0.25	1.06	34.23	
w8	-1793	$2.77 \cdot 10^{13}$	-0.261	0.28	0.25	1.09	32.27	134.38
w8	-2451	$2.79 \cdot 10^{13}$	-0.231	0.28	0.25	1.20	35.65	
w8	-2339	$2.78 \cdot 10^{13}$	-0.271	0.28	0.25	1.06	31.34	
w8	-2511	$2.74 \cdot 10^{13}$	-0.231	0.28	0.25	1.20	35.11	
w13	-1512	$2.75 \cdot 10^{13}$	-0.261	0.29	0.25	1.08	31.82	131.20
w13	-2110	$2.76 \cdot 10^{13}$	-0.251	0.28	0.25	1.12	33.23	
w13	-1887	$2.76 \cdot 10^{13}$	-0.271	0.29	0.25	1.05	30.95	
w13	-1631	$2.75 \cdot 10^{13}$	-0.231	0.28	0.25	1.19	35.20	
w14	-2315	$2.75 \cdot 10^{13}$	-0.241	0.28	0.25	1.11	34.21	130.49
w14	-2109	$2.74 \cdot 10^{13}$	-0.251	0.29	0.26	1.07	31.53	
w14	-2430	$2.75 \cdot 10^{13}$	-0.231	0.29	0.25	1.13	34.86	
w14	-2378	$2.76 \cdot 10^{13}$	-0.271	0.28	0.26	1.01	29.90	
w15	-	$2.75 \cdot 10^{13}$	-0.271	0.28	0.25	1.12	30.94	124.73
w15	-1664	$2.75 \cdot 10^{13}$	-0.261	0.28	0.25	1.16	31.93	
w15	-1704	$2.76 \cdot 10^{13}$	-0.251	0.29	0.25	1.18	32.88	
w15	-2506	$2.77 \cdot 10^{13}$	-0.291	0.28	0.25	1.04	28.98	
w16	-2331	$2.78 \cdot 10^{13}$	-0.251	0.29	0.25	0.95	33.35	194.24
w16	-1903	$2.77 \cdot 10^{13}$	-0.241	0.28	0.25	0.99	34.54	
w16	-1900	$2.76 \cdot 10^{13}$	-0.261	0.29	0.25	0.93	32.10	
w16	-2847	$2.75 \cdot 10^{13}$	-0.311	0.28	0.25	0.78	26.87	
w16	-2652	$2.77 \cdot 10^{13}$	-0.251	0.29	0.25	0.95	33.25	
w16	-2374	$2.76 \cdot 10^{13}$	-0.241	0.29	0.25	0.98	34.12	
w23	-1803	$2.75 \cdot 10^{13}$	-0.251	0.28	0.25	1.06	33.15	325.55
w23	-1572	$2.76 \cdot 10^{13}$	-0.241	0.29	0.25	1.08	33.98	
w23	-2297	$2.76 \cdot 10^{13}$	-0.271	0.29	0.25	0.99	31.05	
w23	-2104	$2.76 \cdot 10^{13}$	-0.261	0.29	0.25	1.02	32.04	
w23	-1873	$2.76 \cdot 10^{13}$	-0.251	0.29	0.26	1.05	31.70	
w23	-1698	$2.75 \cdot 10^{13}$	-0.221	0.29	0.25	1.14	35.79	
w23	-2244	$2.76 \cdot 10^{13}$	-0.261	0.29	0.25	1.02	32.05	
w23	-1956	$2.76 \cdot 10^{13}$	-0.301	0.28	0.25	0.89	27.95	
w23	-1896	$2.75 \cdot 10^{13}$	-0.231	0.29	0.25	1.11	34.86	
w23	-1701	$2.76 \cdot 10^{13}$	-0.251	0.29	0.25	1.05	32.98	
w24	-2159	$2.76 \cdot 10^{13}$	-0.261	0.29	0.25	1.08	32.00	98.72
w24	-2333	$2.76 \cdot 10^{13}$	-0.261	0.29	0.25	1.08	31.93	
w24	-2077	$2.75 \cdot 10^{13}$	-0.231	0.29	0.25	1.18	34.79	

Table A.7: Synthetis of the data for thick wires and beam size of 0.25

Wire -	Raw Signal	I_b [N _{prot}]	μ_h [mm]	σ_h [mm]	σ_v [mm]	f_g [%]	Dose [MGy]	Dose total [MGy]
w3	-2171	$2.78 \cdot 10^{13}$	-0.241	0.28	0.25	2.58	32.97	32.97
w4	-2935	$2.76 \cdot 10^{13}$	-0.261	0.28	0.25	2.32	30.84	59.47
w4	-2850	$2.66 \cdot 10^{13}$	-0.261	0.28	0.26	2.32	28.63	
w9	-1796	$2.78 \cdot 10^{13}$	-0.271	0.28	0.25	2.49	29.83	122.08
w9	-2078	$2.78 \cdot 10^{13}$	-0.281	0.28	0.25	2.40	28.73	
w9	-1864	$2.78 \cdot 10^{13}$	-0.281	0.28	0.25	2.40	28.70	
w9	-2498	$2.78 \cdot 10^{13}$	-0.221	0.28	0.25	2.91	34.82	
w10	-1580	$2.78 \cdot 10^{13}$	-0.301	0.28	0.25	1.52	27.39	121.81
w10	-1811	$2.77 \cdot 10^{13}$	-0.261	0.28	0.26	1.76	30.32	
w10	-2922	$2.78 \cdot 10^{13}$	-0.231	0.28	0.25	1.93	34.78	
w10	-2732	$2.76 \cdot 10^{13}$	-0.281	0.28	0.25	1.64	29.32	
w11	-2405	$2.75 \cdot 10^{13}$	-0.251	0.28	0.25	1.84	32.33	182.68
w11	-2615	$2.73 \cdot 10^{13}$	-0.261	0.28	0.25	1.78	31.02	
w11	-2218	$2.74 \cdot 10^{13}$	-0.251	0.28	0.26	1.84	30.93	
w11	-1827	$2.75 \cdot 10^{13}$	-0.281	0.29	0.26	1.66	28.02	
w11	-2635	$2.75 \cdot 10^{13}$	-0.311	0.28	0.26	1.48	25.08	
w11	-2033	$2.74 \cdot 10^{13}$	-0.221	0.28	0.25	2.01	35.30	135.16
w12	-2973	$2.76 \cdot 10^{13}$	-0.211	0.29	0.25	2.40	35.48	
w12	-2375	$2.76 \cdot 10^{13}$	-0.201	0.28	0.25	2.51	37.09	
w12	-2379	$2.75 \cdot 10^{13}$	-0.261	0.29	0.25	2.09	30.81	
w12	-2161	$2.75 \cdot 10^{13}$	-0.251	0.29	0.25	2.15	31.78	
w17	-2376	$2.76 \cdot 10^{13}$	-0.241	0.29	0.25	0.98	32.60	184.55
w17	-2933	$2.77 \cdot 10^{13}$	-0.211	0.29	0.25	2.39	35.42	
w17	-2678	$2.75 \cdot 10^{13}$	-0.291	0.29	0.25	2.59	27.78	
w17	-2519	$2.74 \cdot 10^{13}$	-0.281	0.29	0.25	2.04	28.62	
w17	-2313	$2.76 \cdot 10^{13}$	-0.251	0.29	0.25	2.11	31.66	
w17	-1921	$2.75 \cdot 10^{13}$	-0.271	0.29	0.26	2.32	28.47	180.46
w18	-2587	$2.74 \cdot 10^{13}$	-0.251	0.29	0.25	2.19	31.78	
w18	-2534	$2.75 \cdot 10^{13}$	-0.251	0.29	0.25	2.06	31.79	
w18	-2305	$2.75 \cdot 10^{13}$	-0.271	0.29	0.25	2.06	29.89	
w18	-3212	$2.76 \cdot 10^{13}$	-0.261	0.29	0.25	1.93	30.99	
w18	-2943	$2.75 \cdot 10^{13}$	-0.251	0.29	0.25	1.99	31.86	180.46
w18	-2587	$2.75 \cdot 10^{13}$	-0.321	0.29	0.26	2.06	24.15	

Table A.8: Synthetis of the data for thin wires and beam size of 0.50

Wire -	Raw Signal	I_b [N _{prot}]	μ_h [mm]	σ_h [mm]	σ_v [mm]	f_g [%]	Dose [MGy]	Dose total [MGy]
w24	-1583	$2.96 \cdot 10^{13}$	-0.042	0.58	0.58	1.18	11.04	69.07
w24	-1608	$2.96 \cdot 10^{13}$	0.138	0.74	0.74	0.81	6.69	
w24	-1678	$2.97 \cdot 10^{13}$	0.268	0.76	0.76	0.63	6.08	
w24	-1765	$2.97 \cdot 10^{13}$	-0.442	0.56	0.56	0.58	8.73	
w24	-1812	$2.97 \cdot 10^{13}$	-0.402	0.56	0.56	0.62	9.20	
w24	-1933	$2.97 \cdot 10^{13}$	-0.422	0.56	0.56	0.65	8.98	
w24	-1983	$2.97 \cdot 10^{13}$	-0.402	0.56	0.56	0.63	9.22	
w24	-1170	$2.94 \cdot 10^{13}$	-0.402	0.56	0.56	0.65	9.13	
w31	-1456	$2.98 \cdot 10^{13}$	-0.452	0.56	0.56	1.19	8.60	87.53
w31	-1781	$2.99 \cdot 10^{13}$	-0.472	0.56	0.56	0.67	8.38	
w31	-1969	$2.99 \cdot 10^{13}$	-0.422	0.56	0.56	0.65	8.99	
w31	-1939	$2.97 \cdot 10^{13}$	-0.372	0.56	0.56	0.70	9.53	
w31	-2550	$2.99 \cdot 10^{13}$	-0.442	0.56	0.56	0.74	8.75	
w31	-2188	$2.99 \cdot 10^{13}$	-0.412	0.56	0.56	0.68	9.12	
w31	-1820	$2.97 \cdot 10^{13}$	-0.442	0.56	0.56	0.70	8.71	
w31	-2425	$2.98 \cdot 10^{13}$	-0.422	0.56	0.56	0.68	8.97	
w31	-2007	$2.99 \cdot 10^{13}$	-0.492	0.56	0.56	0.70	8.12	85.80
w31	-2720	$2.98 \cdot 10^{13}$	-0.472	0.56	0.56	0.63	8.35	
w32	-2257	$2.97 \cdot 10^{13}$	-0.482	0.56	0.56	0.65	8.20	
w32	-1676	$2.98 \cdot 10^{13}$	-0.492	0.56	0.56	0.64	8.10	
w32	-2345	$2.97 \cdot 10^{13}$	-0.532	0.56	0.56	0.63	7.57	
w32	-2004	$3.00 \cdot 10^{13}$	-0.442	0.56	0.56	0.59	8.78	
w32	-2689	$2.99 \cdot 10^{13}$	-0.412	0.56	0.56	0.68	9.12	
w32	-2276	$2.98 \cdot 10^{13}$	-0.422	0.56	0.56	0.71	8.97	
w32	-1861	$2.99 \cdot 10^{13}$	-0.442	0.56	0.56	0.70	8.76	53.32
w32	-2511	$2.97 \cdot 10^{13}$	-0.492	0.56	0.56	0.68	8.08	
w32	-2059	$2.97 \cdot 10^{13}$	-0.362	0.56	0.56	0.63	9.63	
w32	-1754	$2.98 \cdot 10^{13}$	-0.452	0.56	0.56	0.76	8.60	
w21	-1694	$2.97 \cdot 10^{13}$	-0.422	0.56	0.56	1.30	9.02	
w21	-2366	$2.97 \cdot 10^{13}$	-0.482	0.56	0.56	0.53	8.29	
w21	-2087	$2.96 \cdot 10^{13}$	-0.422	0.56	0.56	0.49	9.00	
w21	-1687	$2.99 \cdot 10^{13}$	-0.442	0.56	0.56	0.53	8.84	
w21	-2089	$2.96 \cdot 10^{13}$	-0.402	0.56	0.56	0.52	9.24	52.21
w21	-1753	$2.98 \cdot 10^{13}$	-0.432	0.56	0.56	0.55	8.94	
w22	-2645	$2.85 \cdot 10^{13}$	-0.492	0.56	0.56	0.53	7.81	
w22	-1892	$2.99 \cdot 10^{13}$	-0.382	0.56	0.56	0.52	9.55	
w22	-2522	$2.99 \cdot 10^{13}$	-0.422	0.56	0.56	0.60	9.05	
w22	-1758	$2.99 \cdot 10^{13}$	-0.412	0.56	0.56	0.57	9.19	
w22	-2427	$2.98 \cdot 10^{13}$	-0.472	0.56	0.56	0.58	8.41	
w22	-2059	$2.99 \cdot 10^{13}$	-0.492	0.56	0.56	0.53	8.19	

Table A.9: Synthetis of the data for thick wires and beam size of 0.50 (1/2)

Wire -	Raw Signal	I_b [N _{prot}]	μ_h [mm]	σ_h [mm]	σ_v [mm]	f_g [%]	Dose [MGy]	Dose total [MGy]
w25	-1886	$2.97 \cdot 10^{13}$	-0.392	0.56	0.56	0.65	8.94	
w25	-2627	$2.97 \cdot 10^{13}$	-0.442	0.56	0.56	1.40	8.37	
w25	-2890	$2.96 \cdot 10^{13}$	-0.362	0.56	0.56	1.31	9.25	
w25	-2862	$2.96 \cdot 10^{13}$	-0.412	0.56	0.56	1.45	8.71	
w25	-2896	$2.96 \cdot 10^{13}$	-0.442	0.56	0.56	1.36	8.36	76.31
w25	-2933	$2.95 \cdot 10^{13}$	-0.402	0.56	0.56	1.31	8.80	
w25	-2908	$2.95 \cdot 10^{13}$	-0.502	0.56	0.56	1.38	7.62	
w25	-3046	$2.97 \cdot 10^{13}$	-0.412	0.56	0.56	1.19	8.74	
w25	-3170	$2.97 \cdot 10^{13}$	-0.512	0.56	0.56	1.36	7.52	
w26	-1147	$2.97 \cdot 10^{13}$	-0.432	0.56	0.56	1.17	8.49	
w26	-2229	$2.96 \cdot 10^{13}$	-0.422	0.56	0.56	1.37	8.56	
w26	-2304	$2.97 \cdot 10^{13}$	-0.412	0.56	0.56	1.39	8.70	
w26	-2335	$2.97 \cdot 10^{13}$	-0.462	0.56	0.56	1.40	8.11	
w26	-2355	$2.97 \cdot 10^{13}$	-0.452	0.56	0.56	1.31	8.25	84.28
w26	-2462	$2.97 \cdot 10^{13}$	-0.372	0.56	0.56	1.33	9.15	
w26	-2386	$2.97 \cdot 10^{13}$	-0.542	0.56	0.56	1.48	7.15	
w26	-2522	$2.98 \cdot 10^{13}$	-0.392	0.56	0.56	1.15	8.97	
w26	-2604	$2.99 \cdot 10^{13}$	-0.532	0.56	0.56	1.44	7.31	
w26	-2551	$2.98 \cdot 10^{13}$	-0.332	0.56	0.56	1.17	9.59	
w27	-1243	$2.98 \cdot 10^{13}$	-0.372	0.56	0.56	1.54	9.15	
w27	-2108	$2.99 \cdot 10^{13}$	-0.412	0.56	0.56	1.55	8.73	
w27	-2189	$2.97 \cdot 10^{13}$	-0.562	0.56	0.56	1.48	6.88	
w27	-2349	$2.99 \cdot 10^{13}$	-0.422	0.56	0.56	1.17	8.61	
w27	-2381	$2.99 \cdot 10^{13}$	-0.472	0.56	0.56	1.46	8.01	80.72
w27	-2438	$2.99 \cdot 10^{13}$	-0.502	0.56	0.56	1.36	7.67	
w27	-2576	$2.97 \cdot 10^{13}$	-0.522	0.56	0.56	1.30	7.37	
w27	-2599	$2.98 \cdot 10^{13}$	-0.392	0.56	0.56	1.25	8.94	
w27	-2627	$2.97 \cdot 10^{13}$	-0.422	0.56	0.56	1.52	8.57	
w27	-2778	$2.98 \cdot 10^{13}$	-0.572	0.56	0.56	1.46	6.78	
w28	-2623	$2.97 \cdot 10^{13}$	-0.492	0.56	0.56	1.15	7.85	
w28	-2741	$2.98 \cdot 10^{13}$	-0.432	0.56	0.56	1.09	8.59	
w28	-2688	$2.96 \cdot 10^{13}$	-0.512	0.56	0.56	1.19	7.56	
w28	-2827	$2.97 \cdot 10^{13}$	-0.462	0.56	0.56	1.05	8.22	
w28	-2828	$2.98 \cdot 10^{13}$	-0.422	0.56	0.56	1.14	8.72	84.39
w28	-2802	$2.98 \cdot 10^{13}$	-0.422	0.56	0.56	1.21	8.72	
w28	-2963	$2.99 \cdot 10^{13}$	-0.452	0.56	0.56	1.21	8.37	
w28	-2954	$2.98 \cdot 10^{13}$	-0.432	0.56	0.56	1.16	8.58	
w28	-1860	$2.98 \cdot 10^{13}$	-0.382	0.56	0.56	1.19	9.17	
w28	-1986	$2.98 \cdot 10^{13}$	-0.432	0.56	0.56	1.27	8.59	

Table A.10: Synthetis of the data for thick wires and beam size of 0.50 (2/2)

Wire -	Raw Signal	I_b [N _{prot}]	μ_h [mm]	σ_h [mm]	σ_v [mm]	f_g [%]	Dose [MGy]	Dose total [MGy]
w19	-2669	$2.96 \cdot 10^{13}$	-0.452	0.56	0.56	0.67	8.17	53.95
w19	-	$2.95 \cdot 10^{13}$	-0.402	0.56	0.42	1.49	11.63	
w19	-	$2.95 \cdot 10^{13}$	-0.512	0.56	0.42	1.27	9.93	
w19	-1957	$2.96 \cdot 10^{13}$	-0.482	0.56	0.56	1.39	7.81	
w19	-2255	$2.94 \cdot 10^{13}$	-0.482	0.56	0.56	1.33	7.79	
w19	-2885	$2.95 \cdot 10^{13}$	-0.412	0.56	0.56	1.33	8.61	
w20	-2512	$2.97 \cdot 10^{13}$	-0.582	0.56	0.56	1.47	6.73	49.91
w20	-2062	$2.95 \cdot 10^{13}$	-0.412	0.56	0.56	0.93	8.76	
w20	-2653	$2.96 \cdot 10^{13}$	-0.392	0.56	0.56	1.21	9.01	
w20	-2318	$2.96 \cdot 10^{13}$	-0.592	0.56	0.56	1.24	6.58	
w20	-1850	$2.97 \cdot 10^{13}$	-0.362	0.56	0.56	0.91	9.37	
w20	-2438	$2.97 \cdot 10^{13}$	-0.352	0.56	0.56	1.29	9.47	

A.3 WIRE DEFORMATION



Figure A.4: Wires after irradiation with rea measured beam size (σ), number of pulses (N_p) and number of interactions (N_i).

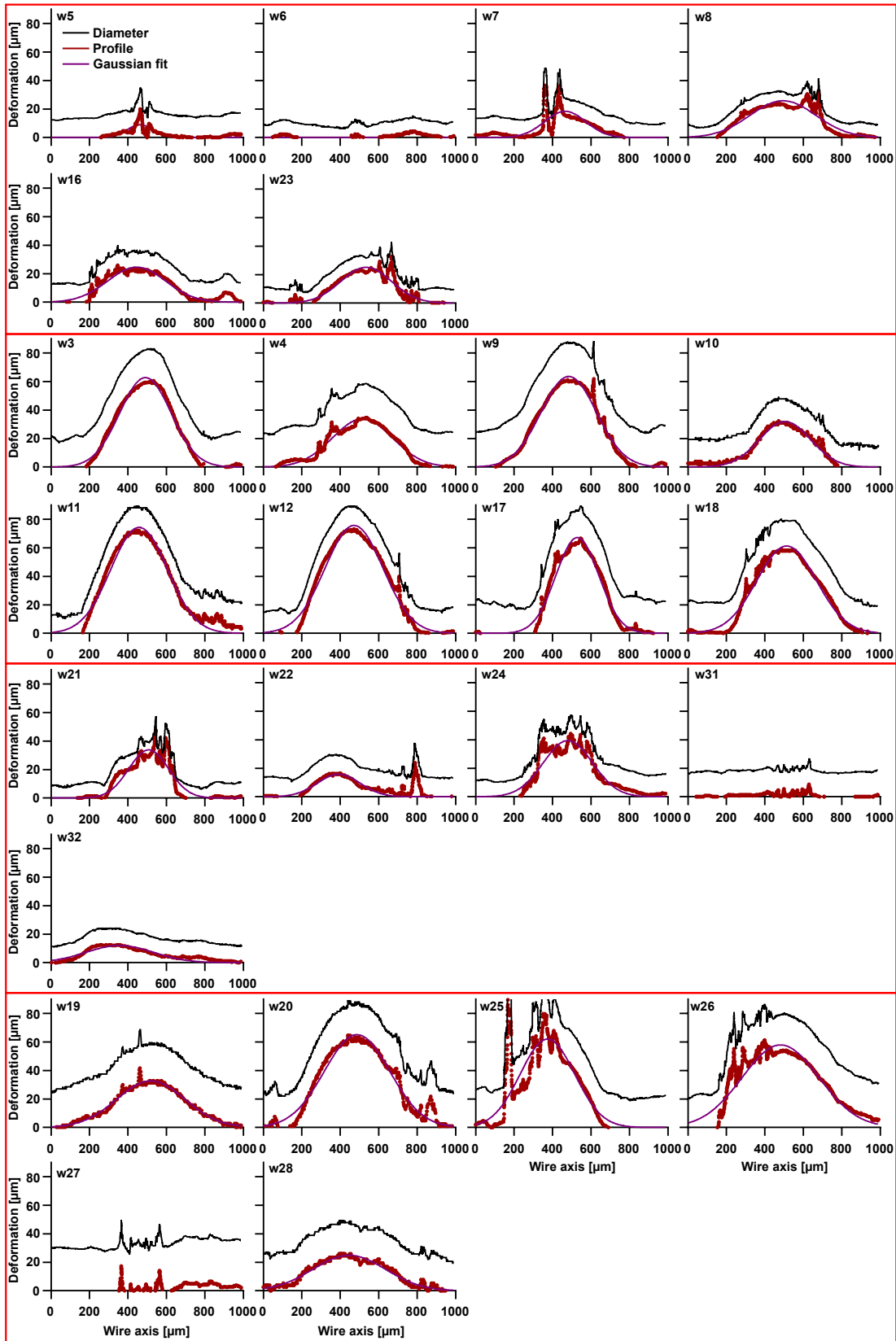


Figure A.5: Profile reconstruction and Gaussian fit the profile.

Titre : Etude des effets du cuivrage et de l'irradiation par proton à 440 GeV sur les propriétés mécaniques de fils en nanotubes de carbone pour l'instrumentation des faisceaux au CERN

Mots clefs: Nanotubes de carbone, Résistance à la traction, Analyse de Weibull, Instrumentation des faisceaux, Wire-scanner, Irradiation par proton, HiRadMat, CERN

Résumé : L'utilisation d'instruments interceptifs tels que les wire-scanners pour mesurer les profils transversaux des faisceaux de particules, est aujourd'hui remise en question par l'augmentation de l'énergie et de l'intensité des faisceaux de la chaîne d'accélérateurs du CERN (SPS, LHC). L'objectif de ce travail est d'étudier la possibilité de remplacer les fils en fibre de carbone par des fils en nanotubes de carbone (NTC). Une première partie est consacrée à l'étude de l'impact du cuivrage superficiel, utilisé actuellement pour le maintien en position des fils, sur les propriétés mécaniques des fils en NTC, par une campagne de tests de traction combinée à une analyse de Weibull. Une étude des déformations du fil lors d'un scan est menée par l'intermédiaire d'un code numérique Matlab. Une seconde partie est dédiée à l'étude du comportement physique (simulations numériques utilisant FLUKA) et mécanique (tests de traction) de ces fils après irradiation par proton à 440 GeV.c⁻¹. Pour ceci, une expérience a été conçue, développée et installée

sur la ligne expérimentale HiRadMat au CERN. Ce travail montre tout d'abord un impact direct du cuivrage qui diminue les propriétés mécaniques des fils NTC, menant à des spécifications non tenables sur les déformations maximales du fil pour des vitesses de scan de 20 m.s⁻¹. L'analyse de l'émission des électrons secondaires durant l'irradiation ne montre pas d'impact direct causé par l'accumulation de des faisceaux successifs, mais des déformations locales importantes sont toutefois observées au microscope électronique, peut-être en lien avec des particules de catalyseurs initialement incluses dans les fils. Les résultats des tests de traction montrent une nette dégradation des propriétés mécaniques après l'irradiation. L'ensemble de ce travail montre l'intérêt à trouver des solutions alternatives aux fibres de carbone actuellement utilisées, mais également les limitations atteintes par les produits actuels (fils NTC) qui nécessiteraient encore des améliorations pour répondre positivement aux besoins du CERN.

Title : Study of the effects of copper coating and proton irradiation at 440 GeV on the mechanical properties of carbon nanotube wires for particle beam instrumentation at CERN

Keywords : Carbon nanotube, Tensile resistance, Weibull analysis, Beam instrumentation, Wire-scanner, Proton irradiation, HiRadMat, CERN

Abstract : The use of wire-scanners, interceptive instruments to measure the transverse profiles of particle beams, is currently challenging due to the increase in the energies and intensity of the beams at the SPS and the LHC, CERN accelerators. The objective of this work is to study the possibility of replacing carbon fiber wires with carbon nanotube (CNT) wires.

The first part is the study of the impact of surface copper plating currently used to fit the wire in the holders, on the mechanical properties of CNT wires, by a campaign of tensile tests combined with a Weibull analysis. A study of the deformations of the wire during a scan is carried out using a Matlab numerical code. A second part is the study of the physical (numerical simulations using FLUKA) and mechanical behavior (tensile tests) of these wires after proton irradiation at 440 GeV.c⁻¹. For this, an experiment has been developed and installed on the HiRadMat

experimental line at CERN.

This work first shows a direct impact of copper coating which decreases the mechanical properties of CNT wires, leading to untenable specifications on the maximum deformations of the wire for scan speeds of 20 m.s⁻¹. The analysis of the emission of secondary electrons during irradiation does not show any direct impact caused by the accumulation of successive beams, despite significant local deformation modifications observed under the electron microscope, possibly due to the presence of catalytic iron particles. The results of the tensile tests show a clear degradation of the mechanical properties after irradiation.

This work shows the interest in finding alternative solutions to the carbon fibers currently used, but also the limitations reached by current products (CNT wires) which still need improvements to meet a CERN's requirements.