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Materials Characterization: Graphite-based and Cu-OFE Samples

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1 Abstract

Characterization of materials is an important field in Materials Science and Engineering. It provides valuable information about the materials and helping the scientific community to make decisions about materials selection, to evaluate failure, product development, among others. This Summer Student Programme project aims to support the Materials, Metrology and Non-Destructive Testing (MM) Section of CERN regarding the characterization of materials activities, with more focus on graphite-based samples from the Luli2000 laser facility and Cu-OFE samples from the Flash project to provide results about the height profile of the samples' craters and information about the grain size and hardness of the samples, respectively.

2 Introduction

The characterization and classification of a material is directly related to its structure, properties and fabrication. The characterization techniques allows the scientific community to determine the physical, chemical, mechanical and microstructural properties of materials and to understand causes of failure, product development, contaminants, comparison between structures, among others. Therefore, it is an important field in Materials Science and Engineering which allows the proper selection of materials for an application.

The Materials, Metrology and Non-Destructive Testing (MM) Section of CERN is responsible for characterization and analysis of materials. Besides, it gives support to other CERN's sections regarding the development, selection and specification of materials for the projects.

The Luli2000 laser facility is conducting experimental studies [1] to provide large data about the shock wave propagation, the laser-matter interaction and the damage into porous graphite-based samples after a high energy laser incision. In parallel, the Flash project [2] carries out studies to innovate the electron radiotherapy technology, in which is being evaluated the use of Cu-OFE as selected material for the radiofrequencies cavities of the linear accelerator. In both cases, materials characterization is needed to evaluate the damage on the graphite-based samples and to evaluate the material selection for accelerating cavities of the radiotherapy devices, respectively.

The CERN Summer Student Programme offers students the unique opportunity to join the day-to-day work of research teams at CERN while attending a series of lectures and workshops with experienced scientists and other students from all around the world, providing a multidisciplinary and multicultural environment enriching experience [3].

This 2022 Summer Student Programme project aims to support the MM section regarding the characterization of materials, with more focus on two activities. The first one is providing results about the height profile of the craters on both front and back side of 48 graphite-based samples for the Luli2000 laser facility. The second one is providing information about the hardness and grain size of Cu-OFE samples for the Flash project, to assist further studies about the hardness vs heat treatment's temperature and the recrystallization temperature.

3 Motivation

3.1 Damage measurements of Graphite-based samples

Collimators are beam limiting devices which are used in particle accelerators to narrow the beam [4]. The materials used to build them have to be chosen very carefully to be able to handle possible beam impacts on its walls. To simulate accidental scenarios in future accelerators, 48 disc-shaped samples with a 10 mm diameter of graphite-based materials were hitten by a high-power laser with high energy densities.

When the front side is hitten, the laser interacts with the matter of the target generating a plasma which expands. The expansion generates a shock wave which propagates through the material reaching the back side. Depending on the material and the thickness of the sample, the propagation of the shock wave causes more or less damage to the back side.

The aim of this project is to use the digital microscope to investigate the shape of the craters in the front and back side of the samples caused by the shock wave. The analysis of the results will help the Luli2000 laser facility [1] to understand the effects of laser and matter interaction in the collimators context. And also, it provides valueable information to select the proper material in the context of collimators for future high-energy accelerators.

3.2 Cu-OFE characterization

FLASH [2] is a project developed by CERN and Switzerland's Lausanne University Hospital (CHUV) to innovate in the electron radiotherapy technology field. Radiotherapy uses linear accelerators to produce a beam of high-energy electrons which is precisely targeted in the patient's body [5]. To accelerate the beam some devices are necessary such as radiofrequency (RF) cavities and the materials used to build those devices have to be chosen carefully to fit the application.

Cu-OFE is a high purity, oxygen free, non-phosphorus-deoxidized material, which means that it has no oxygen and is insensitive to hydrogen embrittlement while ensuring a very high electrical and thermal conductivity. Those characteristics ensure the requirements of superconducting and vacuum applications [6].

When the Cu-OFE is forged, usually the grains in the rolling direction become elongated in relation to the grains in the transverse direction and the material presents residual stress which can lead to deformation of the structure. To analyze if the material fits the requirements to be used in radiofrequency cavities, it is necessary to analyze the homogeneity and the residual stress of the structure regarding the heat treatment's temperature, as well as the recrystallization's temperature of the crystallographic structure.

The aim of this project is to perform in two Cu-OFE bars grain size analysis to verify the homogeneity of the sample, and Vickers hardness tests after different heat treatment's temperatures to build a curve of hardness vs temperature and evaluate the recrystallization's point.

4 Description of the techniques

4.1 Digital microscopy

A microscope uses light and lens to amplify images and enable the samples to be analyzed. An optical microscope structure can be divided in three basic parts: the head, which houses the eyepiece lenses and tube; a base, which houses the nosepiece, the objective lenses, the stage clips, an aperture, a condenser, iris diaphragm, the coarse focus knob, the fine focus knob and the illuminator; and the arm, which connects the base and the head. The digital microscope follows the same principle, however, the image is obtained by a digital camera and observed by a computer. [7, 8]

The Fine Depth Composition function of the Keyence digital microscope works by focusing on the lowest and uppest area of the sample and moving the camera up and down to capture images sequentially at 50 frames per second. Then, the Keyence algorithm processes the images data to compose the depth profile of the sample analyzed, generating a 2D or 3D profile display [9].

Grain Size Analysis (Intercept) function of the Keyence digital microscope works by using the intercept technique to estimate the average grain size. A pattern of curves is drawn in the picture and the number of the grain boundaries intersecting the curves are counted. Then, the number of intersections is divided by the total length of the curves to find the average grain size [9].

4.2 Vickers Hardness Test

The Vickers hardness test consists of applying a force into the surface of the sample using a diamond indenter, which is shaped as a right pyramid with a squared base and with specified angle between opposite faces at the vertex, as represented in Figure 1 below. An indentation is obtained and its depth will depend on the hardness of the material, the smaller the indentation the harder the material is. The diagonal lengths formed on the surface are measured and the software calculates the hardness by dividing the test force by the area of the indentation, which is assumed to be also a right square-based pyramid with the same vertex angle as the indenter [10, 11].

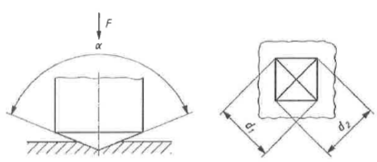

Figure 1: Representation of the hardness test principle. Source:[10]

5 Methodology

5.1 Surface profile measurements of Graphite-based samples

To investigate the shape of the craters in the discs, the Fine depth composition function on the Keyence Digital Microscope was used on each sample.

The sample was carefully placed in the microscope with the front side facing up. The Fine depth composition was used to create a 3D profile display of the crater. The two points profile line tool was used to draw a vertical line crossing the crater. Then, an horizontal line was drawn perpendicularly also crossing the crater. A height profile graph was generated for both regions. After, the same procedure was repeated for the back side facing up for all of the 48 samples.

The 48 samples were classified according to the material, thickness in μm and the shot of the laser, for example, sample CrGr_2000_shot33 is a CrGr sample of 2000 μm which was incised by the shot 33 of the laser.

5.2 Cu-OFE characterization

To perform the required analysis, the samples had to be prepared. For the grain size analysis, both Sample 1 and Sample 2 were cutted using the Mecatone T210 cutting machine to extract two samples, one for the rolling direction and one for the transverse direction, as represented in the Figure 2 below. The edges of the samples were smoothed in the polishing machine and taken to the mountain press machine with phenofree rouge resin, 15 minutes of heating, 5 minutes of cooling, 170 bar of pressure and 180ºC of temperature. The samples were taken to the Smart Lam polishing machine to be grinded with P80, 320, 600 and 1200 grit discs, from the bigger grain size to the smaller, and to be polished with magnetic discs with diamond suspension of 6 μm, 3μm and 1μm. Then, chemical attack was done using Ammonium Persulfate 10% immersion for 25 seconds.

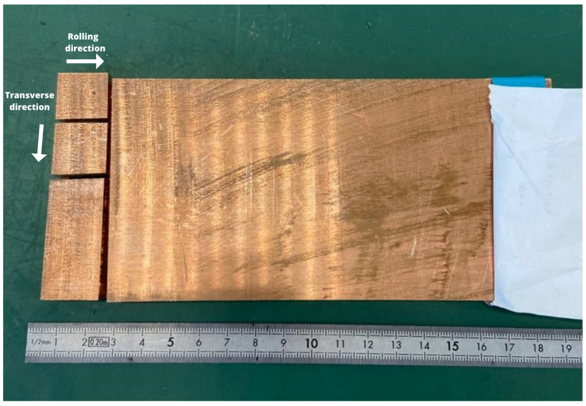

Figure 2: Representation of the cutting directions on Sample 2.

The samples were then analyzed in the Keyence digital microscope regarding the structure, and to investigate the grain size of each sample the Grain Size Analysis (Intercept) function was used.

Then, 5 more samples in each rolling and transverse direction for both Sample 1 and Sample 2 were cutted using the Diamond Saw cutting machine to reduce the possible stress in the samples. A representation of the cutted samples on the rolling and transverse direction for Sample 1 can be seen in Figure 3, in which the samples were named accordingly to the sample, direction and number of the cut, for example, 1L1 represents the first sample in the rolling direction of the Sample 1 cutted with the diamond saw machine. Then, Vickers hardness tests were performed in some of the samples before heat treatment, after 100°C heat treatment and 400°C heat treatment.

Figure 3: Representation of the cutted samples on the rolling and transverse direction for Sample 1.

6 Results and discussion

6.1 Surface profile measurements of Graphite-based samples

According to the procedure described in Methodology (Section 5.1), the 48 samples were analyzed using the Keyence digital microscope. As we can see in Figure 4 below, it is possible to observe the incision of the laser in the front side of the sample CrGr_2000_shot33 and the back side does not present any visible damage. On the other hand, Figure 5 presents the sample CuCr_750_shot45 which is damaged on both sides. The damage depends on how the wave shock interacts and propagates through the thickness of the material, therefore, some of the samples were damaged only on one side or on both sides.

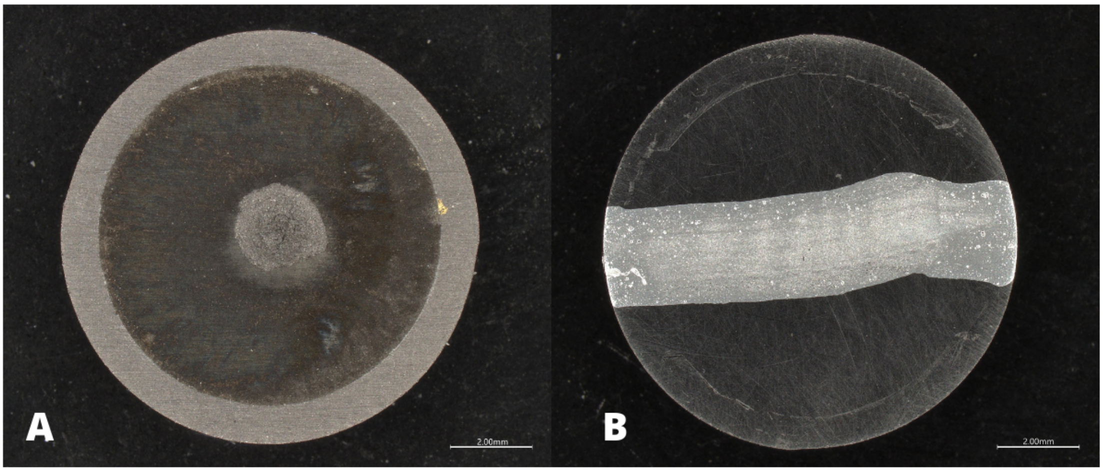

Figure 4: Front (A) and back (B) side of the CrGr_2000_shot33 sample.

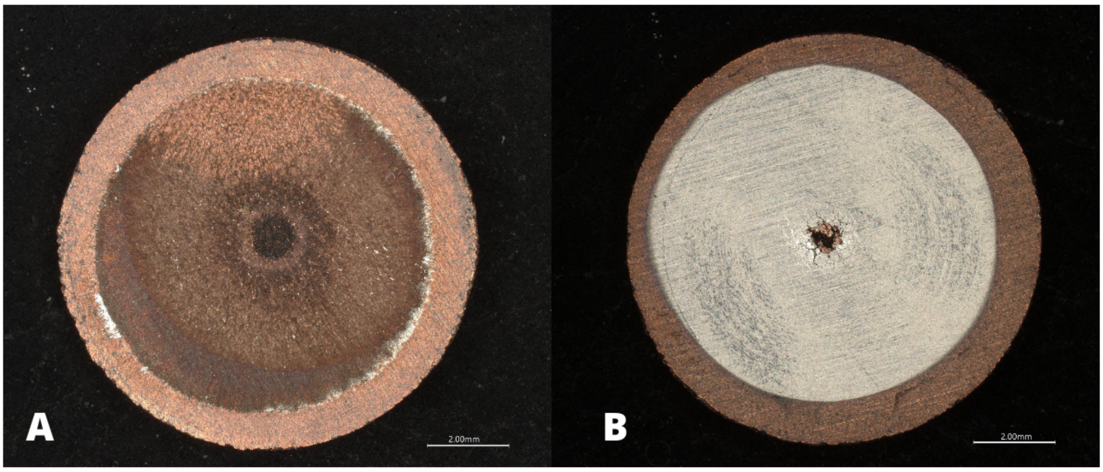

Figure 5: Front (A) and back (B) of the CuCr_750_shot45 sample.

The fine depth composition in the vertical and horizontal direction was done for each side of samples. Figure 6 and Figure 7 show examples of the profile graph for the vertical direction in respectively the front side and back side of sample CrGr_2000_shot33, in which we can observe the depth profile of the incision in the front side and that the damage in the back side was minimal.

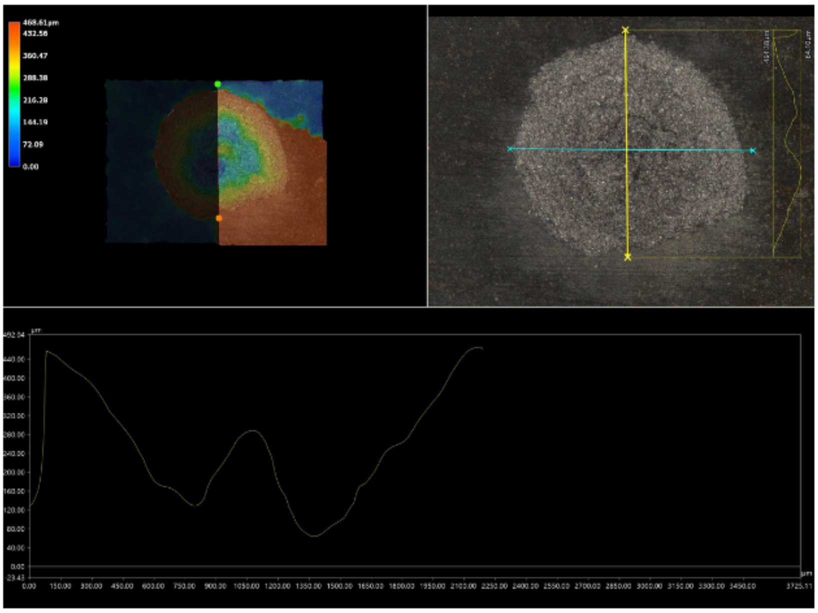

Figure 6: Results of the surface measurements of the CrGr_2000_shot33 in the vertical direction of the front side.

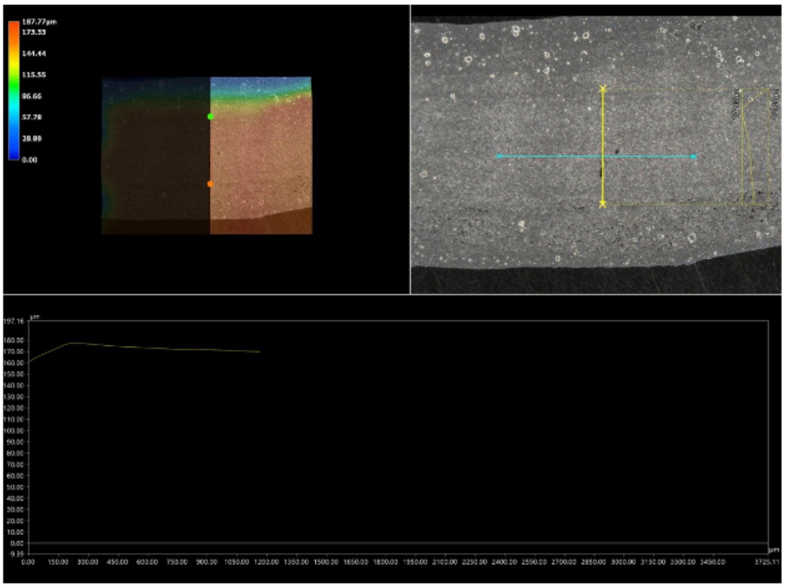

Figure 7: Results of the surface measurements of the CrGr_2000_shot33 in the vertical direction of the backside.

On the other hand, Figure 8 and Figure 9 show the other situation in which both sides of the sample CuCr_750_shot45 presents craters. It can be observed in the profile graphs for the horizontal direction of the front side and back side that the wave shock caused damage in both sides.

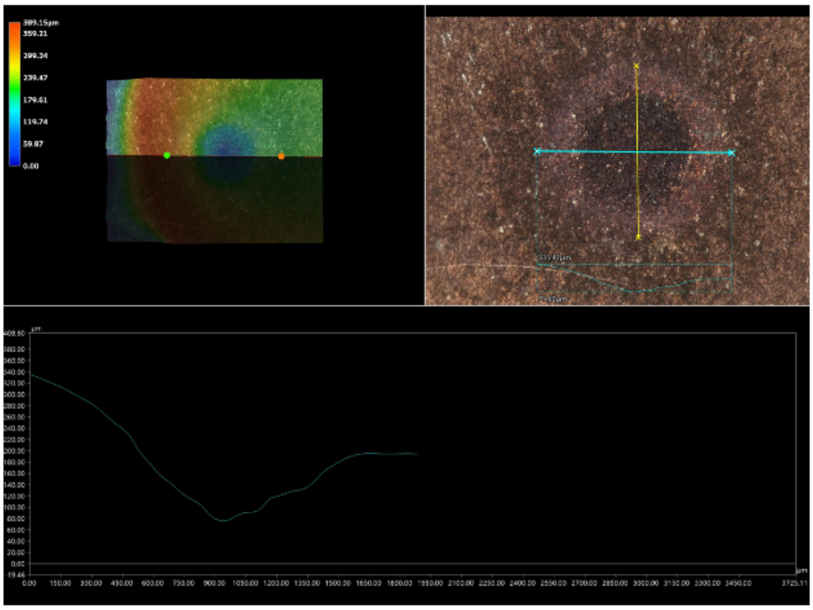

Figure 8: Results of the surface measurements of the CuCr_750_shot45 in the horizontal direction of the front side.

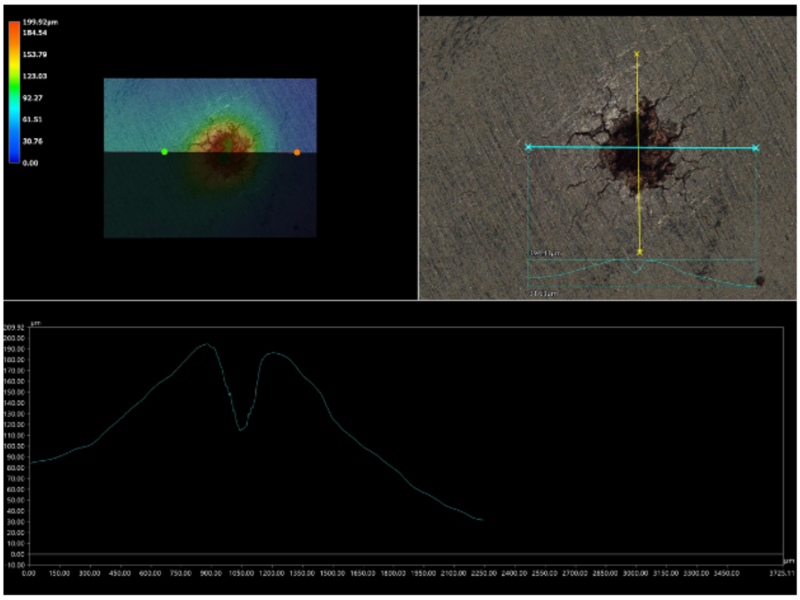

Figure 9: Results of the surface measurements of the CuCr_750_shot45 in the horizontal direction of the back side.

The results will be further compared to the roughness profile results from the Metrology Section and later explored by the Luli2000 laser facility regarding the laser-matter interaction. The results will also be correlated to the results of simulations regarding the quantity of material removed from both the front and back side of each sample due to the impact of the high-energy laser.

6.2 Cu-OFE characterization

According to the procedure described in Methodology (Section 5.2) , the prepared Sample 1 and Sample 2 can be seen in Figure 10 below, in which both contain the rolling and the transverse direction samples.

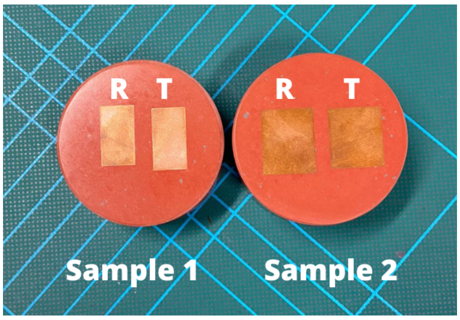

Figure 10: Rolling (R) and transverse (T) direction of Sample 1 and Sample 2.

The samples were then observed with the Keyence digital microscope regarding their surface structure. The results can be seen in Figure 11 below, in which A refers to the rolling direction of Sample 1, B to the transverse direction of Sample 1, C to the rolling direction of Sample 2 and D to the transverse direction of Sample 2. It is possible to see that the samples do not present remarkable differences in the grain structures.

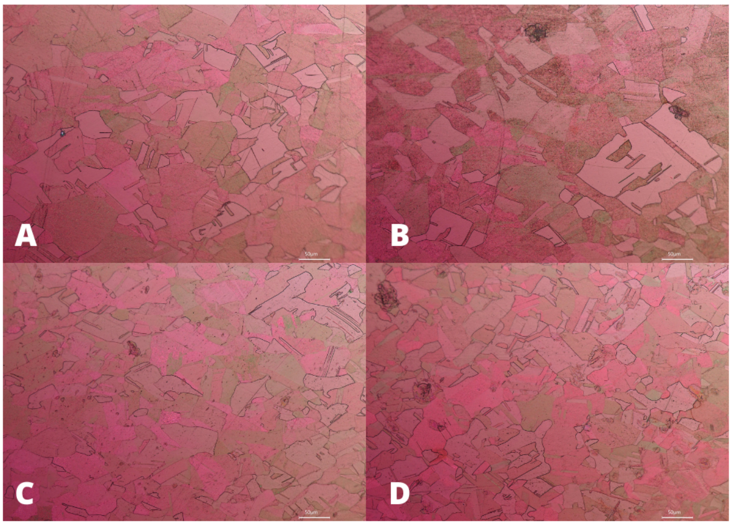

Figure 11: Cu-OFE structures from (A) rolling direction of Sample 1, (B) transverse direction of Sample 1, (C) rolling direction of Sample 2 and (D) transverse direction of Sample 2.

The results of the grain size analysis were given by Mean Grain Size No. and the corresponding grain size diameter were checked in the standard for grain size evaluation [12], which can be seen in Table 1 below. The rolling and transverse direction of both samples do not differ in the grain size diameter and Sample 1 and Sample 2 present close values, which indicates homogeneity of the structure.

Table 1: Grain size results for rolling and transverse direction of Sample 1 and Sample 2.

Grain Size results		
	Mean Grain Size No.	Grain Size Diameter [μm]
Sample 1 - rolling direction	6.5	37.8
Sample 1 - transverse direction	6.5	37.8
Sample 2 - rolling direction	7.0	31.8
Sample 2 - transverse direction	7.0	31.8

The results of the Vickers hardness test can be seen in Table 2 below. It is possible to see that before the heat treatment, the hardness in the rolling samples are a little higher than the transverse samples, which can be explained by the residual stress from the fabrication process, however, samples in the same direction of the same sample present close values for hardness. After the heat treatment, it is possible to observe that the samples which were heat treated at 400ºC present the hardness reduced to almost half of the value and the samples which were heat treated at 100ºC present close values for hardness compared to the ones before the heat treatment. This can be explained by the recrystallization of the structure and further relaxation of the structure, since 100ºC is before and 400ºC is after the possible recrystallization point.

Table 2: Vickers hardness test results before and after heat treatment.

Hardness test BEFORE heat treatment	
Sample	**Hardness [V]**
Standard Sample 1	127.2
1T1	128.27
1T2	121.26
1T4	128.57
1R2	139.47
1R5	139.53
Standard Sample 2	127.57
2T2	117.8
2R2	131.36

Hardness test AFTER heat treatment		
Sample	**Hardness [V]**	**Temperature (°C)**
1T2	66.07	400
1T3	59.33	400
1T4	129.93	100
1T5	128.67	100
1R2	53.00	400

6.2.1 Further studies

Further studies will be carried out regarding the evaluation of the residual stress and hardness of the samples cutted with the Wire Saw machine. An XRD device will be used to evaluate the residual stress of the samples in different heat treatment temperatures and the Vickers hardness test will be performed also for those temperatures. A hardness vs temperature and a stress vs temperature graphs will be obtained and the possible recrystallization point will be extracted. Besides, a range of heat treatment's temperature will be evaluated to select a recrystallization's temperature. It should be high enough to reduce the residual stress, which is important for the brazing operators of accelerating structures, but also not too high to avoid softening of the material, which would damage the machining tools.

7 Conclusion

Materials characterization provides important information about the properties of materials and it is an important tool for projects to evaluate the applications of some material. For the graphite-based samples, it was possible to observe that the laser-matter interaction depends on the material and the thickness of the sample because some of the samples were damaged in both sides and some others were only damaged in the incision side. For the Cu-OFE samples, it was possible to observe that the material presents a level of homogeneity, because the grain sizes analysis provided close values for the rolling and the transverse direction. Besides that, it was possible to observe that the hardness results before the heat treatment presented close values for the same direction, and after heat treatment the hardness values were decreased almost in half when the temperature exceeds the possible recrystallization point while the values maintained the same when the temperature was lower than the possible recrystallization point.

Therefore, this project provided information about the damage measurements of Graphite-based samples, which will help the Luli2000 laser facility to evaluate the laser-matter interaction, and about the gain size and hardness of Cu-OFE samples, which together with the further studies will help the Flash project to select the proper material to be used for the RF cavities.

References

[1] Gabriel Seisson, Gabriel Prudhomme, Pierre Antoine Frugier, David Hébert, Emilien Lescoute, et al.. Dynamic fragmentation of graphite under laser-driven shocks: Identification of fourdamage regimes. International Journal of Impact Engineering, Elsevier, 2016, 91, pp.68-79. 10.1016/j.ijimpeng.2015.12.012. hal-02271620.

[2] FLASH An innovative electron radiotherapy technology. CERN. Available on: < https://videos.cern.ch/record/2295068>. Access on August 08, 2022.

[3] Summer Student Programme CERN Accelerating science. Available on: <https://home.cern/summer-student-programme>. Access on August 08, 2022.

[4] What is a Collimator?. Azo Optics. Available on: <https://www.azooptics.com/Article.aspx?ArticleID=54 Access on August 08, 2022.

[5] Radioterapia com Elétrons. Irmev - instituto de radioterapia. Available on: <http://www.irmev.com.br/tratamentos/radioterapia-com-eletrons>. Access on August 08, 2022.

[6] Cu-OFE. Lamineries Matthey SA. Edition 2014/02. Page . Available on: <https://www.matthey.ch/fileadmin/user_upload/downloads/fichetechnique/EN/Cu-OFE_C.pdf >. Acess on August 07, 2022.

[7] Compound Microscope Parts. Microsope.com. . Available on: <https://www.microscope.com/compound-microscope-parts>. Acess on August 08, 2022.

[8] Mokobi, Faith. Digital Microscope- Definition, Principle, Parts, Types, Examples, Uses. Microbe Notes. Available on: <https://microbenotes.com/digital-microscope/>. Acess on August 08, 2022.

[9] Basics of Digital Depth Composition. Microscope Glossary. Available on: <https://www.keyence.com/ss/products/microscope/microscope_glossary/observation/depth_compositio Acess on August 07, 2022.

[10] Metallic materials - Vickers hardness test. British standard. BS EN ISO 6507-1:2005.

[11] Vickers Hardness Test: What it is and how it's measured . Matmatch. Available on: <https://matmatch.com/learn/property/vickers-hardness-test>. Acess on August 08, 2022.

[12] ASTM International. Standard Test Methods for Determining Average Grain Size. Designation: E112 – 10.