

MICROSCOPY INVESTIGATION ON DIFFERENT MATERIALS AFTER PULSED HIGH FIELD CONDITIONING AND LOW ENERGY H-

C. Serafim^{1,2}, S. Calatroni¹, F. Djurabekova², A. T. Perez Fontenla¹, R. Peacock¹, W. Wuensch¹, S. Sgobba¹, A. Grudiev¹, A. Lombardi¹, E. Sargsyan¹, S. Ramberger¹, G. Bellodi¹



¹CERN Organisation Européenne pour la Recherche Nucléaire, Geneva, Switzerland
²University of Helsinki, Helsinki, Finland



During operation the RFQ (Radio-Frequency-Quadrupole) of the LINAC4 at CERN is exposed to high electric fields which can lead to vacuum breakdown. It is also subject to beam loss that can cause surface modification, including blistering, which can result in reduced electric field handling and an increased breakdown rate. An experimental study has been made to identify materials with high electric field capability and robustness to low-energy irradiation. In this paper we briefly discuss the selection criteria, and we analyse these materials investigating their metallurgical properties using advanced microscopic techniques such as Scanning Electron Microscope, Electron Back Scattered Diffraction, Energy-dispersive X-ray Spectroscopy and conventional optical microscopy. These allow to observe and characterize the different materials on a micro and a nanoscale and to compare results before and after irradiation and breakdown testing.

INTRODUCTION

Surface modifications of materials due to the blistering phenomenon caused by the hydrogen retention it's a known problem [1]. Blistering is dependent on many factors, such as the radiation dose (minimum dose required for appearance of blisters), the ion energy, the temperature of the target and angle of incidence [2], as well as the metallurgical properties of the material. In the case of the RFQ the energy of the incident H⁻ beam at its entrance, where most beam losses are localised, is 45 keV. This corresponds to a range of approximately 250 nm in copper, with the H possibly accumulating under the surface and leading to blistering.

Two pairs of anode-cathode electrodes (see below) of different materials were manufactured for the purpose of the studies. For the study of the blistering phenomena, and the material properties at its origin, a test stand that replicates the low energy beam transport in the LINAC4 tunnel at CERN was used. In this test stand, specific hardware was developed to use a cathode as the target for irradiation and allows for a rapid turn-over for various irradiation runs. For the study of the breakdown phenomena, we have used the Large Electrode system (LES) in which we tested the electrodes that were manufactured to be compatible with the chamber geometry. Detailed information about this system can be found in [3].

For a better understanding if any blistering phenomena caused by the beam irradiation could have a consequent effect in triggering breakdowns, we have irradiated first the materials on the LINAC 4 test stand and then tested them on the LES. For comparison, a pair of non-irradiated electrodes was also tested in the system.

METODOLOGY

The materials were selected based on their usability for meter-long high gradient RF cavities and their potential resistance to blistering and breakdown phenomena, with the purpose of manufacture a future RFQ with better performance. Irradiation was performed at CERN for different materials using the parameters of Table 2, which corresponds to about ten days of beam losses during RFQ operation.

Table 1 – List of materials that we will present in this poster.

Material I	Cu OFE	Oxygen Free Electronic Grade Copper, UNS C10100
Material II	TiAl6V4	Premium grade forged in α - β range with a final α -microstructure
Material III	Nb	Niobium, Purity 99.9% and RRR300

Table 2 – Parameters set-up for the irradiation campaign. All the electrodes were irradiated using the same conditions and parameters.

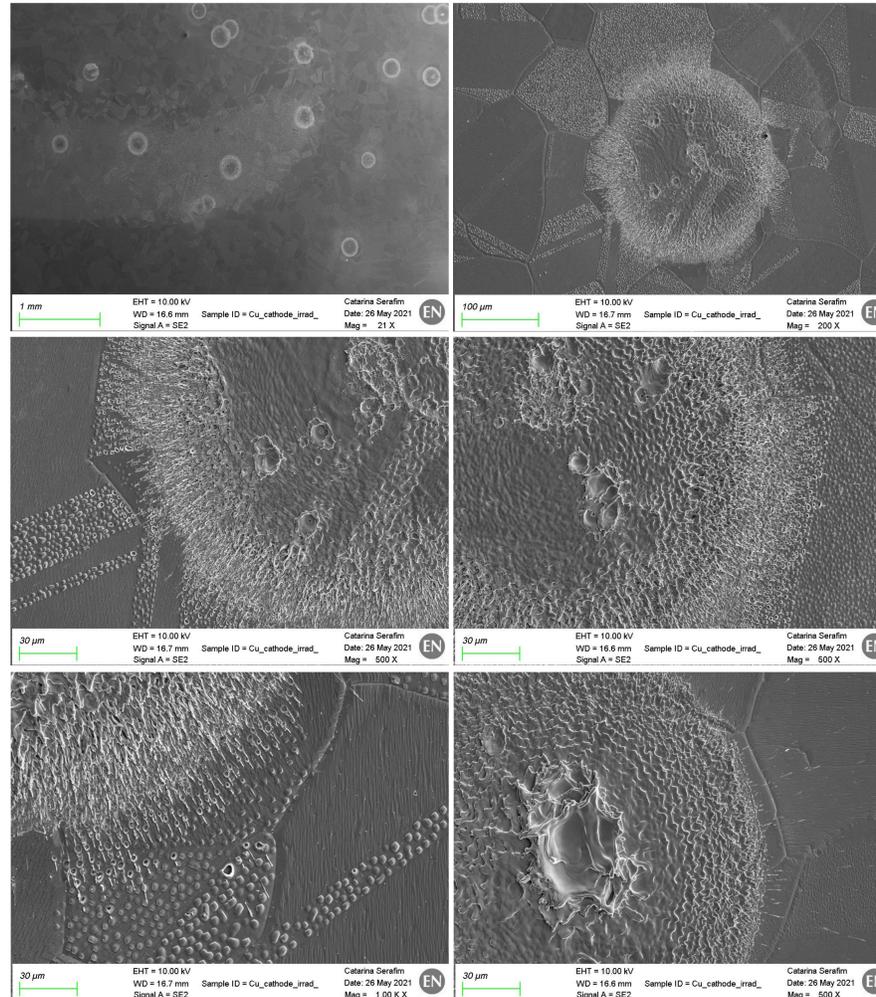
Low Energy H- beam	45 keV
Duration	40 hours
Pulse duration	600 μ s
Repetition Rate	0.83 Hz
Peak current	20 mA
Deposition of particles on the target	1.2×10^{19} H ⁺ /cm ²

The LES testing was performed in electrodes after being exposed to irradiation and in electrodes that were not exposed to irradiation. Detailed information for the results of the high electric field testing, on the studied electrodes, can be found in [4].

In order to inspect the state of the electrodes between each test, microscopy equipment was used. The samples were analyzed using a digital microscope from KEYENCE and a Scanning Electron Microscope (SEM) Sigma from Zeiss, equipped with Secondary Electron (SE), InLens (InLens), Everhart-Thornley SE (SE2), Backscattered Electron (AsB) and Electron Back-Scattered Diffraction (EBSD) detectors.

OBSERVATIONS

Observations from the SEM equipment showed that the low energy beam irradiation caused the appearance of blisters on the Cu-OFE electrode's surface. These blisters are concentrated on the impact zone. It is visible that the areas where we had the maximum deposition of beam the blisters have a higher density in quantity comparing with zones where the beam was less intense. No blistering was observed in both TiAl6V4 and Nb electrodes after irradiation.



Figures 1-6: SEM imaging with different magnification, using secondary electron detector showing the results of Cu-OFE cathode after being irradiated with the H⁻ beam and then submitted to high electric field pulsing in the LES experiment. The breakdowns have a circular shape with approximately 200 μ m with splashes of melted material on their surroundings. It is also visible that the density of blisters seems to be dependent on the copper crystal orientation.

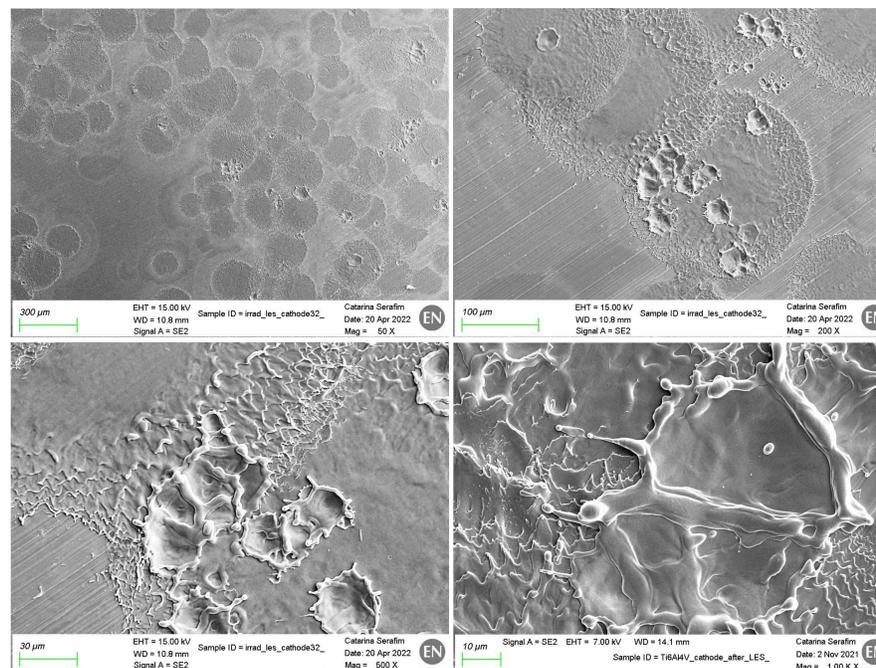


Figure 7-10: SEM imaging with different magnification, using secondary electron detector, showing the breakdowns triggered by the LES experiment, on the TiAl6V4 irradiated cathode.

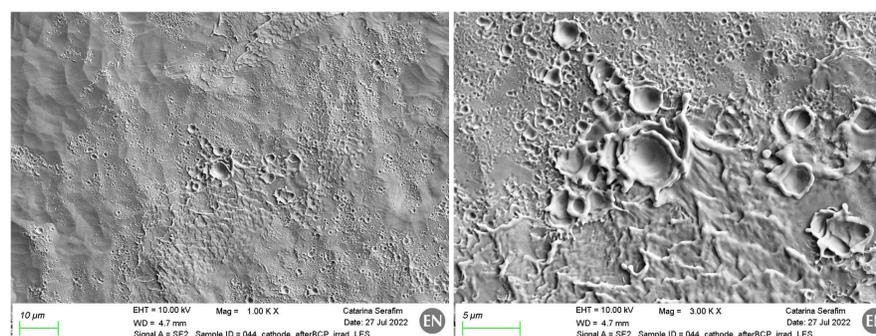


Figure 11-12: SEM imaging with different magnification, using secondary electron detector, showing the breakdowns triggered by the LES experiment, on the Nb irradiated cathode.

Table 3 – Stable high pulsing field reachable for each pair of electrodes (irradiated and non-irradiated) in the Large Electron System. Details about this results can be found in [4].

Material	Pair of electrodes	Average high pulsing field reached from the LES system	Observations of blistering phenomena from irradiation
Cu OFE	Irradiated pair	80 MV/m	Blistering in irradiated zone
	Non-irradiated pair	80 MV/m	x
TiAl6V4	Irradiated pair	90 MV/m	No blistering
	Non-irradiated pair	100 MV/m	x
Nb	Irradiated pair	22 MV/m	No blistering
	Non-irradiated pair	80 MV/m	x

CONCLUSION

From the irradiation experiments only the Cu material has presented blistering on the surface. The affected areas, where the blisters have appeared, are coincident with the H⁻ particles from the beam deposition.

Regarding high electric field pulsing tests, we have not observed, for the three materials (Cu, Nb and TiAl6V4), a higher concentration of breakdowns in the irradiated zone. Breakdowns are located in all the surface of the electrode with no evidence of being triggered by any topographic feature caused by the irradiation. Also, for the non-irradiated electrodes breakdowns have shown to be dispersed throughout all the surface.

We have concluded that the Cu and TiAl6V4 materials have shown the best performance in terms of reaching a stable field. For TiAl6V4, the good results for the irradiated pair of electrodes in the LES, makes us conclude that the irradiation didn't provoke any decrease of performance for this material. In the case of copper, even with the presence of blistering there was also not a significant impact on the breakdown appearance and performance. For the Nb material, the bad performance in terms of reachable high field for the irradiated electrodes, with the additional downside of being difficult to machine, have made us exclude it from the list of possible materials for a future RFQ. The considerable difference in the field values between irradiated and non-irradiated pairs of Nb seems likely that have been caused by effects from the irradiation experiment. However, microscopy observations have shown no particular features in the surface or blistering effects that can be correlated with the decrease of performance.

One important observation was that the phenomenon of blistering is more evident in just some of the grains, which may be correlated with the grain orientation of the material. Additional studies will be performed to understand this correlation. Finally, it should be underlined that in all the electrodes that have been used for the irradiation testing, some deposition of carbon on the surface was observed. Further investigations are also under way to better understand the source of the carbon and to avoid it in future experiments.

ACKNOWLEDGEMENTS

We wish to extend our sincere thanks to all members of the Working Project WP10. A special thank you for all the members of the MME team at CERN that has shared all the knowledge, open access to the microscopy facilities, and for the manufacturing of all the electrodes, and the VSC team for materials surface treatments. Also, we would like to thank the test stand of LINAC4 working team which has given access to the facilities and manipulated the procedure for irradiation.

REFERENCES

- [1] Fundamentals of Radiation Materials Science, Metals and Alloys, 2nd Edition, Gary S. Was, Springer New York, 978-1-4939-3436-2, 2017
- [2] <https://indico.cern.ch/event/966437/contributions/4245468/attachments/2203398/>
- [3] High Field Studies for CLIC Accelerating Structures Development, I. Profatilova, CERN, Geneva, Switzerland, Institute of Applied Physics, National Academy of Sciences of Ukraine, Vol. 1/2017, CERN-Proceedings, 2017
- [4] Pulsed DC high field measurements of irradiated and non-irradiated electrodes of different materials, R. Peacock et al, LINAC2022 Conference (Ref.TUPOPA13), UK, 2022