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

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Abstract

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 is 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. All electrodes underwent systematic metallurgical analyses, as detailed in this paper.

METODOLOGY

A list of candidate materials was carefully selected for manufacturing of a future RFQ with better performance. In this paper we will focus on the results for three different materials, Oxygen Free Electronic Grade Copper (Cu OFE, UNS C10100), Niobium (Nb) purity of 99.9%, RRR300, and a Titanium grade 5 alloy (TiAl6V4), premium grade forged in α-β range with a final α-microstructure.

The materials were selected based on their usability for meter-long high gradient RF cavities and their potential resistance to blistering and breakdown phenomena. Nb and Ti grade 5 were chosen for their high hydrogen diffusivity, which should prevent accumulation of hydrogen and thus blistering. Ti grade 5 should also provide comparatively easier machinability. The machining of the electrodes was performed with high precision tooling. After machining electrodes have been submitted to metrology testing to assure that all the requirements are within the stipulated ranges. After this, electrodes have been submitted to a cleaning process and a brazing cycle, for the case of Cu, of 100 °/h up to 795 °C for 6 hours and 100 °/h up to 835 °C for 45 minutes.

Irradiation was performed at CERN for different materials using a low energy H^- beam at 45 keV. Each irradiation had a duration of approximately 40 hours, with a pulse duration of 600 μs, a repetition rate of 0.83 Hz and a peak current of 20 mA, which resulted in a deposition of 1.2x 10^19 H^-p/cm^2 on the target, corresponding to about ten days of beam losses during RFQ operation.

The LES testing was performed in electrodes after being exposed to irradiation and in electrodes that were not exposed to irradiation. This allows us to compare if the irradiation had a significant impact on the performance of each material in reaching a maximum and stable field. Detailed information for the results of the high electric field testing, on the studied electrodes, can be found in [4].

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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.

The results from the observation will help us understand the development of blistering, if any, created on the surface from the irradiation and a possible correlation with the location of the breakdowns from the LES.

RESULTS

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.

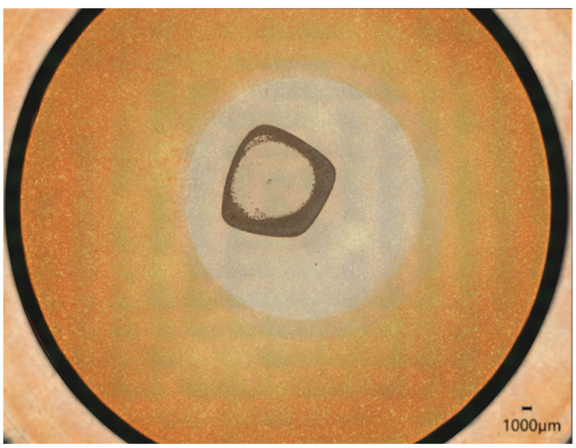

Figure 1: Optical microscope picture using 50x lens, including all the surface of the Cu cathode after irradiation and before preforming tests with high energy pulsing.

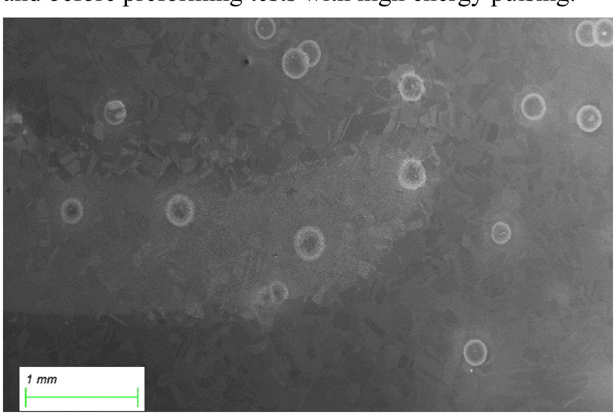

Figure 2: SEM imaging, using secondary electron detector, showing partially the transition zone (the irradiated zone corresponds to the zone less dark) and some of the breakdowns provoked by the high energy pulsing.

In Fig. 1 it can be seen an optical picture of all the surface of the electrode where is visible the result of the beam impact from the irradiation. Figures 2-4, show the results of Cu-OFE after the electrode being irradiated with the H-beam and then submitted to high electric field pulsing in the LES experiment. This pair of Cu-OFE electrodes have reached an average value of field 80 MV/m [4]. A different pair of electrodes made of Cu, which were not irradiated, were also tested in the LES system. In this case the electrodes have reached as well, an average field of 80 MV/m which make us conclude that the irradiation and their effects did not decrease the performance of this material.

We can observe from Fig. 2 that the breakdowns are well distributed on the surface and do not seem to be correlated with the irradiated zone (zone less dark due to the blistering effect). Figure 3 shows a breakdown in the transition zone. We have designated transition zone for the area where we start to see a decrease of density of blistering. In this zone it is visible that only some grains are affected by blisters. The fact that the phenomenon of blistering is more evident in just some of the grains may be correlated with the grain orientation of the material. Additional studies will be performed to understand this correlation.

The breakdowns have a circular shape with approximately 200 µm with splashes of melted material on their surroundings. The heat provoked by the breakdowns have shown to be significant enough to destroy some of the blisters close to it, as we can see in Fig. 4.

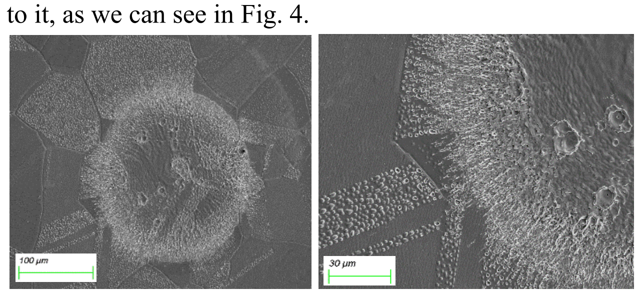

Figure 3: SEM imaging with different magnification, using secondary electron detector, showing a breakdown in the transition zone of the electrode.

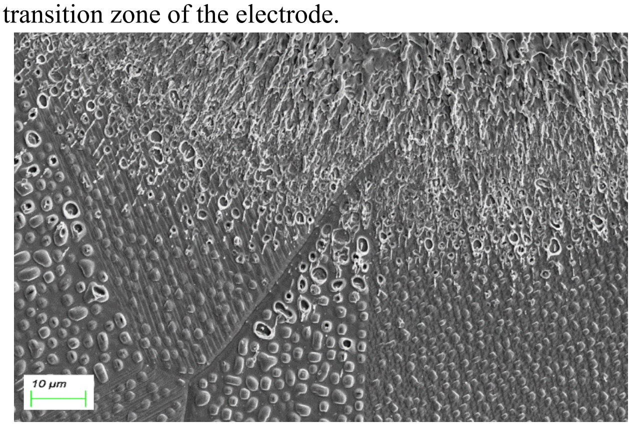

Figure 4: SEM imaging, using secondary electron detector, showing the effect of the breakdown on the surrounding blisters that were present on the surface.

Electrodes of Nb and TiAl6V4 were also submitted to irradiation, with the same parameters used on the Cu-OFE electrode described before. For the Nb and TiAl6V4 materials we have observed no blistering on the surface.

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Figure 5 shows the breakdowns on the TiAl6V4 irradiated cathode on the surface. Concerning the LES experiment after irradiation, the TiAl6V4 electrodes have reach a stable field of 90 MV/m [4].

Also, for this material we have seen a slight decrease of breakdown performance due to irradiation - the pair of TiAl6V4 non-irradiated electrodes that was tested in the LES system reached the average value of 100 MV/m [4].

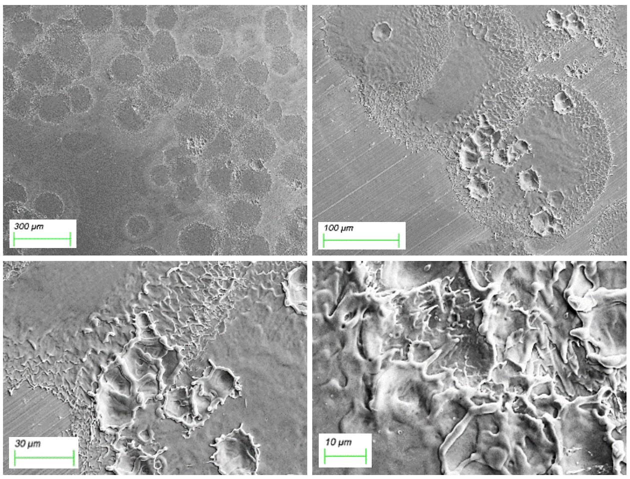

Figure 5: SEM imaging with different magnification, using secondary electron detector, showing the breakdowns triggered by the LES experiment, on the TiAl6V4 irradiated cathode.

Figures 6 and 7 show some breakdowns on the Nb irradiated electrode. The breakdowns are smaller and less deep when comparing with the Cu-OFE and TiAl6V4 electrodes. For the Nb material, the electrodes have reached an average field of 22 MV/m in the LES system, after irradiation. The second pair of electrodes which was not irradiated have reached a field in the LES system of 80 MV/m [4]. The considerable different in the fields values seems to 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, and investigations are ongoing to ascertain if this can be attributed to chemical surface modifications.

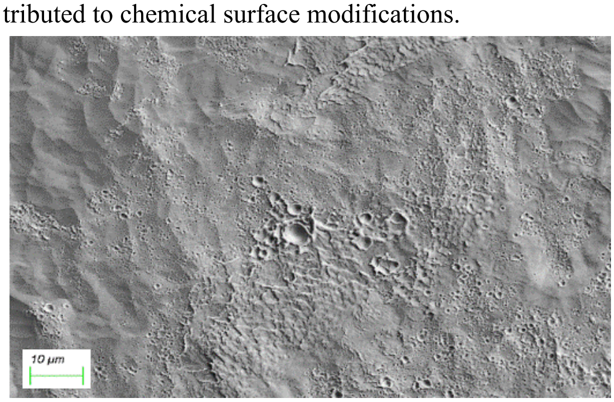

Figure 6: SEM imaging showing the breakdowns triggered by the LES experiment, on the Nb irradiated cathode, using secondary electron detector.

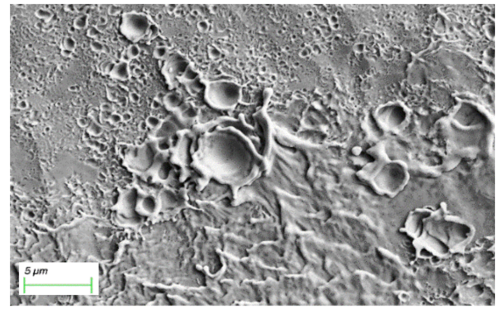

Figure 7: SEM imaging showing the breakdowns triggered by the LES experiment, on the Nb irradiated cathode, using secondary electron detector.

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.

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.

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