DC BREAKDOWN FIELD MEASUREMENTS OF HEAT TREATED MOLYBDENUM

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Abstract

The effect of heat treatments of molybdenum on the resistance to breakdown and conditioning speed has been investigated in the DC spark setup. The conditioning speed is strongly improved by treatment at 875°C in UHV ex situ compared to chemically cleaned surfaces. This temperature is sufficiently low to prevent a re-crystallization of the material which could influence mechanical properties, as hardness. The saturation field remains unchanged. The beneficial effect is preserved for 8 h of air exposure after treatment.
1. INTRODUCTION

The feasibility of the future 12 GHz multi-TeV $e^+e^-$ Compact Linear Collider (CLIC) is nowadays under investigation at CERN. In order to limit this machine to an acceptable length, extremely high accelerating gradients of the order of 100 MV/m are required. With such fields, RF breakdowns are likely to occur and to produce damages on the accelerating cavities. Therefore, a material capable of sustaining high electric fields and showing low damages after breakdowns is needed. Furthermore, in practical operation, breakdowns can also lead to the loss of the accelerated beam due to random kicks. Thus, the structures have also to produce a breakdown rate as low as possible, typically of the order of $10^{-6}$.

In order to test candidate materials and also to have a better understanding of the breakdown mechanism, a DC breakdown study is underway at CERN in parallel to RF testing. DC breakdown measurements are indeed much simpler than RF measurements. Different materials such as Cu, Mo, W and Ti have already been tested [1-3]. Molybdenum shows promising results with a relatively good DC saturated breakdown field (440 MV/m) after conditioning (i.e. the improvement in the capability of holding high fields, obtained after repetitive sparking on the same spot), a relatively good conditioning speed and a stable electrode gap after many sparks [2]. Molybdenum is therefore a good candidate for the high field regions of CLIC structures.

Other studies have shown that vacuum heat treatments significantly improve the conditioning of copper and stainless steel electrodes [4-5]. These treatments improve the conditioning speed and the breakdown saturation field as well, by reducing field emission sites and foreign species at the electrodes surfaces. Such heat treatments could therefore be an interesting way to progress towards the CLIC requirements. First DC conditioning measurements on heat treated molybdenum electrodes have been done at CERN and are presented in [6]. The latest results are presented here.

2. EXPERIMENTAL SETUP

The electrodes are molybdenum 99.95% in purity. The anode is a cylindrical rod, 2.3 mm in diameter, with a hemispherical rounded tip. The cathode (sample) is a 45x10 mm$^2$ rectangular plane surface, 1.5 mm in thickness. The heat treatment is performed ex-situ on the cathode only, in a UHV niobium oven. The sample is heated during 2 hours at the nominal temperature (875°C, 1000°C or 1200°C) and at a pressure below 5x10^{-7} mbar, and is then exposed to ambient air for about 30 minutes during transfer and mounting into the spark test chamber.

The electrodes are placed in the UHV spark test chamber at a typical pressure of 5x10^{-9} mbar. The gap distance between the electrodes can be adjusted with a micro-positioning device, and is set typically around 20 μm. The zero distance is found by bringing the electrodes into contact and measuring short-circuit.

The anode is connected to high voltages via a 28 nF capacitor which is beforehand charged with a high voltage source (up to 12 kV), and the cathode is grounded. The electrodes are exposed to high voltage during $\tau = 2$ s, with the use of a high voltage switch placed between the capacitor and the anode. The initial electric field $E_i$ applied between the electrodes is therefore simply calculated knowing the applied voltage and the gap distance. By measuring the remaining charge on the capacitor after the exposure time $\tau$, one can determine the charge which has eventually flown through the gap due to field emission or breakdown (spark), and one can also calculate the remaining field $E_r$ between the electrodes after the exposure time.
The applied voltage is first set to a low value (500 V) and is increased by 150 V steps until breakdown occurs. At the breakdown, almost all the charges accumulated in the capacitor are discharged through the gap and produce the spark, resulting in $E_t$ close to zero. The breakdown field $E_b$ is then determined from this $E_t$-$E_{\tau}$ curve. This cycle is repeated several times in order to have conditioning of the tested spot on the sample surface. The breakdown current peak is measured with a current transformer probe connected to an oscilloscope.

A detailed presentation of the experimental setup can be found in [1].

3. RESULTS

3.1 Effect of the heat treatment on conditioning

As shown in figure 1, the heat treatment has a clear effect on the conditioning speed of Mo electrodes. While it takes roughly 60 sparks to reach a breakdown field of 400 MV/m on an untreated degreased Mo sample, it takes only less than 20 sparks on a sample previously heated at 875°C (see black lines on figure 1). This remarkable improvement can be attributed to the removal of molybdenum oxides at the sample surface due to heating. XPS measurements confirm that oxides are drastically reduced by the heat treatment [6]. On the other hand, the saturated breakdown field is not improved by the treatment, remaining around 440 MV/m for Mo.

![Fig. 1: Effect of the heat treatment on conditioning: (a) untreated Mo; (b) Mo heated for 2 hours at 875°C.](image)

Although the amount of oxides is very low, the conditioning of heated samples is not immediate. They still need 15-20 sparks to reach the saturated breakdown field. This is probably due to presence of field emitters at the surface of the electrodes. The topography of the sample has to be smoothed by a few sparks (i.e. the strongest emitters have to be destroyed) before the saturated field can be reached.

3.2 Treatments at different temperatures

In order to avoid deformation, the CLIC structures can not be heated at too high temperatures. The use of a heat treatment is indeed relevant only if the conditioning is significantly improved without any modification of the structure shape. The temperature of the heating has thus to be chosen carefully.
Samples heated at three different temperatures have been tested: 875°C, 1000°C and 1200°C. The corresponding conditioning curves are shown in figure 2.

A slight improvement in conditioning speed is observable with increasing temperature. XPS measurements confirm these results: the sample heated at 1200°C is slightly less oxidized than the sample heated at 875°C [7]. But the results are globally similar for the three treatments: a breakdown field of 400 MV/m is reached within 10-20 sparks. The saturated breakdown fields are also comparable for the three samples and are roughly equal to the saturated field of untreated Mo.

The hardness of the samples has been measured after the heat treatments by Vickers tests. The results are summarized in table 1. While the sample heated at 875°C shows a hardness similar to that of untreated Mo, the hardness of the samples heated at higher temperatures is significantly lower. This indicates that recrystallization occurs beyond 875°C. Even if the conditioning speed is slightly lower after a treatment at 875°C than at higher temperatures (but still far improved compared to untreated Mo), a treatment at 875°C is considerably preferable. Deformations and fragilization of structures are indeed more likely if the metal recrystallizes.

**Table 1: Hardness of the Mo samples after heat treatment (Vickers hardness test HV20)**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Hardness (HV20)</th>
</tr>
</thead>
<tbody>
<tr>
<td>untreated Mo</td>
<td>257</td>
</tr>
<tr>
<td>Mo 875°C</td>
<td>252</td>
</tr>
<tr>
<td>Mo 1000°C</td>
<td>184</td>
</tr>
<tr>
<td>Mo 1200°C</td>
<td>174</td>
</tr>
</tbody>
</table>

3.3 **Effect of air exposure on heat treated Mo**

Since oxidation of electrodes reduces their conditioning speed, the exposure time to air of the electrodes after the heat treatment has to be minimized. In the case of the CLIC structures tested at CERN, their mounting in the RF test facility can last a few hours. The influence of such air exposure times on the conditioning speed of heated Mo samples has been studied in the DC spark test system. Figure 3 shows conditioning curves of a Mo cathode heated at 875°C without and with additional dry air exposures. The measurements are performed on the same sample but on different virgin spots. Between each measurement, the UHV test chamber is vented with dry air at atmospheric pressure for 4 hours.
Even after 8 hours of air exposure, the conditioning speed is not altered: 400 MV/m is reached within 20 sparks. Thus, a heat treatment on the CLIC structures tested in the RF facility should still be effective after a mounting time of 8 hours. Longer air exposures have also been tested. After 60 hours, a small amount of Mo\(^{+IV}\) oxides is observable with XPS [7], but the conditioning speed is still not significantly reduced.

The effect of air exposure has also been studied on already conditioned spots. Once the saturated breakdown field was reached, the sparking was stopped, the chamber was vented and then re-pumped, and finally the sparking was continued exactly on the same position.

Although a 4 hours air venting does not produce any alteration in conditioning speed on virgin spots, a small decrease in breakdown field is measured on conditioned spots, as shown in figure 4. The saturated field is reached again after a few tens of sparks. Conditioned spots are probably more reactive to oxide formation than virgin spots, due to the intensive cleaning and surface modification induced by the sparks. Therefore, oxides are probably easily formed on the spot as soon as air is introduced in the chamber, which leads to this small decrease in breakdown field observed afterwards. These newly formed oxides are then removed by the following 20-30 sparks.

4. CONCLUSION

The effect of a heat treatment is significant on the DC spark conditioning of Mo electrodes. Whereas the saturated field is not improved, the conditioning speed is reduced
roughly by a factor three compared to untreated Mo. This improvement is attributed to the removal of molybdenum oxides at the electrode surfaces during heating. A treatment at 875°C for 2 hours gives already a clear reduction in conditioning speed without recrystallization of the electrodes. This temperature is therefore an interesting choice for the treatment of CLIC structures. Since air exposures up to 8 hours after the treatment do not produce any decrease in the conditioning speed of the heated electrodes, the exposure of CLIC structures to air during the installation in the RF test facility should not be damageable.

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REFERENCES