

CLIC-NOTE-703

# **INVESTIGATIONS OF DC BREAKDOWN FIELDS**

T. Ramsvik, S. Calatroni, A. Reginelli, M. Taborelli

CERN, Switzerland

### Abstract

The need for high accelerating gradients for the future 30 GHz multi-TeV e<sup>+</sup>e<sup>-</sup> Compact Linear Collider (CLIC) at CERN has triggered a comprehensive study of DC breakdown fields of metals in UHV. The study shows that molybdenum (Mo), tungsten (W), titanium (Ti) and TiVAl reach high breakdown fields, and are thus good candidates for the iris material of CLIC structures. A significant decrease in the saturated breakdown field ( $E_{sat}$ ) is observed for molybdenum and tungsten when exposed to air. Specifically, at air pressures of 10<sup>-5</sup> mbar, the decrease in  $E_{sat}$  is found to be 50% and 30% for molybdenum and tungsten, respectively. In addition, a 30% decrease is found when molybdenum is conditioned with a CO pressure of ~1·10<sup>-5</sup> mbar. Surface analysis measurements and breakdown conditioning in O<sub>2</sub> ambience imply that the origin of the decrease in  $E_{sat}$  is closely linked to oxide formation on the cathode surface. 'Ex-situ' treatments by ion bombardment of molybdenum effectively reduce the oxide layers, and improve the breakdown characteristics of the metal drastically.

Presented a EPAC06, Edinburgh, UK June 26-30, 2006

Geneva, Switzerland June 2006

# **INVESTIGATIONS OF DC BREAKDOWN FIELDS**

T. Ramsvik<sup>#</sup>, S. Calatroni, A. Reginelli, M. Taborelli, CERN, 1211 Geneva 23, Switzerland

#### Abstract

The need for high accelerating gradients for the future 30 GHz multi-TeV e<sup>+</sup>e<sup>-</sup> Compact Linear Collider (CLIC) at CERN has triggered a comprehensive study of DC breakdown fields of metals in UHV. The study shows that molybdenum (Mo), tungsten (W), titanium (Ti) and TiVAl reach high breakdown fields, and are thus good candidates for the iris material of CLIC structures. A significant decrease in the saturated breakdown field  $(E_{sat})$ is observed for molybdenum and tungsten when exposed to air. Specifically, at air pressures of 10<sup>-5</sup> mbar, the decrease in  $E_{sat}$  is found to be 50% and 30% for molybdenum and tungsten, respectively. In addition, a 30% decrease is found when molybdenum is conditioned with a CO pressure of  $\sim 1.10^{-5}$  mbar. Surface analysis measurements and breakdown conditioning in O2 ambience imply that the origin of the decrease in  $E_{sat}$  is closely linked to oxide formation on the cathode surface. 'Ex-situ' treatments by ion bombardment of molybdenum effectively reduce the oxide layers, and improve the breakdown characteristics of the metal drastically.

#### **INTRODUCTION**

At CERN the feasibility of a 30 GHz multi-TeV e<sup>+</sup>e<sup>-</sup> Compact Linear Collider (CLIC) is under investigation [1]. To reach the required energy level within feasible length of the linacs, an acceleration gradient of 150 MV/m is chosen, yielding surface fields of  $\geq$  330 MV/m within the accelerating structure. To guarantee sufficiently low breakdown rates during operation, novel solutions of the choice of iris materials are needed [2]. The obvious complexity in testing various iris materials with RF triggered a comprehensive study of DC breakdown fields of metals in ultra-high vacuum (UHV) and micrometer sized gaps. Recent results indicate that a direct comparison of achieved  $E_{sat}$  from tests with 30 GHz RF and DC voltages are indeed meaningful [3].

The dc spark apparatus has now been used to investigate a wide range of vacuum and material effects. In this paper the consequence on  $E_{sat}$  of various gases are investigated for copper (Cu), tungsten (W) and molybdenum (Mo), i.e. materials presently considered as possible candidates for iris materials in the accelerating structure [2]. In addition, breakdown conditioning using titanium (Ti) electrodes is presented.

### **EXPERIMENTAL SETUP**

The breakdown experiments reported in this paper are performed using an experimental setup described in detail in [4].

For all the experiments described in this report, identical materials were chosen for the anode and the

cathode. The cathodes and anodes consist of polycrystalline sheets of 1 mm thickness, and 2.3 mm thick rods with hemispherical apex, respectively. The samples are copper, molybdenum, tungsten, titanium and TiVAl, all with purities > 99.9 %. Prior to be installed in the UHV chamber, all materials were cleaned according to the CERN standard procedure for UHV components [5].

XPS measurements have been carried out with a PHI ESCA 5400 multi-purpose system equipped with a spherical sector electron spectrometer (PHI model 10-360) operated at fixed pass energy and a non-monochromatized MgK $\alpha$  X-ray source.

### **RESULTS AND DISCUSSION**

## Cu, Mo and W: Influence of gases

Figure 1A shows a typical breakdown conditioning curve using molybdenum electrodes, where the maximum breakdown field is plotted as function of the accumulated number of breakdowns events. A detailed description of the realization and interpretation of such measurements can be found in [4]. From figure 1A one sees that the maximum obtained breakdown field increases rapidly during the first ~50 breakdowns, followed by a slow improvement until ~200 breakdowns. These values are found to be typical for virgin cathode/conditioned anode, which was the situation prior to the measurements.

When saturation is reached, air is introduced into the chamber at the pressures indicated in the upper part of figure 1A. At 10<sup>-6</sup> mbar a gradual and significant decrease  $E_{sat}$  occurs, eventually reaching saturation in approximately 150 MV/m below the level reached by ultra-high vacuum (UHV) conditioning, i.e. (465±20) MV/m. When the low pressure is recovered (here low  $10^{-5}$ mbar range)  $E_{sat}$  slowly recovers the initial value. Similar experiments have been performed on different molybdenum samples and with different gases. The results in figure 1B show the saturated breakdown fields as function of the gas pressure recorded inside the vacuum chamber. The lower horizontal axis shows the absolute pressure of air, Ar, CO and H<sub>2</sub>, while the upper shows the absolute pressure of oxygen, shifted by a factor of 5 to lower values to correctly account for the ratio of N<sub>2</sub> and O<sub>2</sub> in air. Similar breakdown field degradations are observed for both dry and laboratory air up two  $10^{-6}$ mbar ranges. Measurements with elevated pressure of argon and H<sub>2</sub> up to the 10<sup>-5</sup> mbar range did not result in any detectable decrease in the breakdown field on molybdenum. For increasing pressures of CO, a significant decrease of  $E_{sat}$  is observed, though somewhat weaker than for air exposure. Note that the shaded areas are guide to the eyes only to emphasize the different evolutions of the breakdown field between CO and air.

Identical measurements have been performed on copper (Cu) and tungsten (W). The results are summarized in table 1, together with the results from the molybdenum measurements. Due to better statistics,  $E_{sat}$  for Mo is higher than reported in [4], and is more in accordance with the breakdown results reported for RF [2,3]. As for Mo, also for W a significant decrease in the saturated breakdown field is recorded when the air pressure is increased from UHV to 1.10<sup>-5</sup> mbar, though the percentage decrease is smaller. For copper no such decrease is observed. Upon exposure to CO only molybdenum exhibits a decrease in the maximum breakdown field, whereas no effect occurs for copper and tungsten. The relative decreases given in table 1 are based on single gas exposure measurements; hence large uncertainties are expected.



Figure 1: Breakdown fields for molybdenum in UHV and the effect of gas pressures of air,  $O_2$ , CO,  $H_2$  and argon. *A*: The lower graph shows the conditioning curve of

molybdenum, where the vertical axis gives the maximum achieved applied electric field in MV/m before breakdown. The upper graph shows the average pressure of *air* for each measurement point. The horizontal axis gives the accumulated number of breakdowns with constant cathode/anode position.

*B*: Variation of the saturated breakdown field (vertical axis) with gas pressures (horizontal axis). The shaded areas surrounding the datapoints symbolize the variation as function of air and CO pressure, respectively. The vertical bars denote the resulting standard deviations.

In order to understand which air component is relevant, further experiments have been performed with pure O<sub>2</sub>. Apart from the expected factor of 5 in pressure, the effect on  $E_{sat}$  is similar. Indeed, the relative decreases using O<sub>2</sub> are measured to be 25% and 30-35% at 2·10<sup>-7</sup> mbar and 2·10<sup>-6</sup> mbar O<sub>2</sub>, respectively. In comparison, 27% and 50% are obtained for air pressures of 1·10<sup>-6</sup> mbar and 1·10<sup>-5</sup> mbar. As for air, the highest  $E_{sat}$  can be regained when the O<sub>2</sub> gas is pumped out and after sufficient re-conditioning. These results strongly point towards oxygen as the main source for the observed decrease of  $E_{sat}$  upon air exposure.

Table 1: Saturated breakdown fields at  $p \le 10^{-9}$  mbar for Cu, Mo and W, and the effect of pressure of air, CO, H<sub>2</sub> and Ar.

|    | Esat         | Relative decrease in $E_{sat}$ by increase of gas pressure to $10^{-5}$ mbar |    |                  |              |
|----|--------------|--|----|------------------|--------------|
|    |              | Air  | СО | $\mathbf{H}_{2}$ | Ar           |
|    | MV/m         | %  | %  | %                | %            |
| Cu | $164 \pm 30$ | 0  | 0  | not<br>meas.     | not<br>meas. |
| W  | $313\pm47$   | 30   | 0  | not<br>meas.     | not<br>meas. |
| Мо | $438\pm32$   | 50   | 30 | 0                | 0            |

To study in more details the role oxidation has on the final  $E_{sat}$ , similar experiments have been performed on molybdenum, where the oxide layers are removed from the cathode surface by Kr<sup>+</sup>- ion bombardment in a separate vacuum chamber, and the surface composition is analyzed by XPS. Roughly 30 minutes was needed to perform the air transfer of the Kr<sup>+</sup> sputter cleaned molybdenum foil to the system dedicated for breakdown experiments, and to pump the vacuum chamber to pressures  $\leq 10^{-4}$  mbar.

Breakdown conditioning of the sputtered Mo sample shows close to immediate increase in the maximum breakdown field to saturation levels typically found for untreated molybdenum electrodes.

After the breakdown conditioning, the molybdenum cathode was transferred to the XPS vacuum, leading to an additional half an hour of exposure to atmospheric air. Figure 4 I shows the resulting XPS spectrum recorded from an area unaffected by breakdown conditioning. From the indicated binding energy positions of the two spin-orbit splitted metallic Mo3d<sup>5</sup> [6], it is clear that some oxides are still present on the surface. Comparison with literature identifies the oxidation states to be Mo<sup>n+</sup>  $(n \le 4)$  [7]. The mid XPS spectrum (figure 4 II) shows the measurements performed inside the area modified by UHV breakdown conditioning. The dominant peaks are in this case coinciding with the binding energy positions of metallic molybdenum, Mo<sup>0</sup>. The lack of oxide confirms that the conditioning effectively removes it. Spectrum III in figure 4 shows the measurements inside the breakdown field area when breakdown conditioning was performed in air pressures of up to 1.10<sup>-5</sup> mbar, i.e. where a significant decrease of  $E_{sat}$  has been confirmed. Again,

comparison with literature [7] identifies the different contributions to originate from  $Mo^{n+}$  (n $\leq$ 5) oxidation states.



Figure 4: Mo  $3d^{5}_{5/2-3/2}$  photoemission spectra after sputter cleaning with 400 eV Kr<sup>+</sup> ion, plus 1 hour exposure to atmospheric air. (I) Surface area unaffected by breakdown conditioning. (II) UHV breakdown conditioned area. (III) Area affected by breakdown conditioning during exposure of ~1·10<sup>-5</sup> mbar air.

#### Titanium (Ti)

Breakdown conditioning of titanium electrodes was carried out. One of these conditioning curves is shown in figure 5. The saturated breakdown field is in this case determined to be (695±9) MV/m, i.e. considerably higher than those given in table 1. Important electrode erosion is observed either because of intrinsic material properties or because of the intensity of the fields which is beyond that applied to other materials. Attempts with TiVAl, a titanium alloy with higher tensile strength than pure titanium, show high  $E_{sat}$ , but do not reduce the amount of erosion.



Figure 5: Breakdown fields using titanium electrodes. Due to significant material erosion leading to variation in the gap distance of several micrometers, regular controls

of the actual distance by moving the electrodes into contact were performed (change between empty and full circles). The electrical breakdown field was then recalculated by assuming a linear dependence between each gap measurements.

## CONCLUSION

In this paper the influence of various gases on the breakdown hold-off capability of molybdenum, tungsten and copper have been investigated. For the refractory metals tungsten and molybdenum, a significant decrease of the saturated breakdown field is observed at elevated air pressures. For molybdenum this effect is also observed during similar CO exposures. Breakdown conditioning in oxygen ambience and x-ray photoemission spectroscopy measurements strongly point towards oxide formation as the source for the observed decrease. "Ex-situ" ion bombardments to remove oxide surface layers have proved to be an effective technique to improve the breakdown capabilities of molybdenum.

Titanium and TiVAl show significantly higher  $E_{sat}$  than molybdenum, tungsten and copper, and are thus considered promising for future applications in accelerating structures.

The reported results will influence the conditioning process and material preparation for the CLIC test program.

#### REFERENCES

- J. P. Delahaye et al., Proceedings of the 1999 Particle Accelerator Conference, vol. 1, p. 250 (New York, NY USA, 1999)
- [2] C. Achard, H. Braun, S. Döbert, I. Syratchev, M. Taborelli, I. Wilson, W. Wuensch, CLIC note 569 (CERN, Geneva, Switzerland 2003)
- [3] M. Taborelli, M. Kildemo, S. Calatroni, Phys. Rev. S-T, 7 (2004) 092003.
- [4] M. Kildemo, Nucl. Instrum. Methods Phys. Res., A 530 (2004) 596.
- [5] C. Scheuerlein, M. Taborelli, Appl. Surf. Sci., 252 (2005) 4279.
- [6] Handbook of X-ray Photoelectron Spectroscopy, Perkin-Elmer Corp., Physical Electronics Division, Eden Prairie, MN (1978), 104.
- [7] A. Galtayries, S. Wisniewski, J. Grimblot, J. El. Spectr. Rel. Phen., 87 (1997) 31. and ref. therein