Field Emission Dark Current of Technical Metallic Electrodes

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Abstract

In the framework of the Low Emittance Gun (LEG) project, high gradient acceleration of a low emittance electron beam will be necessary. In order to achieve this acceleration a -500 kV, 250 ns FWHM, pulse will be applied in between two electrodes. Those electrodes should sustain the pulsed field without arcing, must not outgass and must not emit electrons. Ion back bombardment, and dark current will be damageable to the electron source as well as for the low emittance beam. Electrodes of commercially available OFE copper, aluminium, stainless steel, titanium and molybdenum were tested following different procedures including plasma glow discharge cleaning.

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1 Introduction

In the framework of the Low Emittance Gun (LEG) project, an X-ray free-electron laser based on a field emitting cathode is expected to deliver six orders of magnitude higher peak brightness than current state-of-the-art light sources, and thus with a thousand times shorter pulses [1, 2].

To quickly accelerate the electrons emitted by the electron source, and keep the emittance low, a stable pulsed voltage in the megavolt range is needed. The first project phase is to design and test an ultra high vacuum (UHV) 500 kV pulser using a resonant air-core transformer (Tesla coil) [3]. A pulse of 250 ns (full width at half maximum), -500 kV, working at 10Hz, will be applied between the cathode holder and an extracting anode. During this time, electrodes material should sustain the field without arcing and the dark current should be kept as low as possible. This dark current will ionize the residual gas as well as desorbing neutral and ions by the known electron stimulated desorption process (ESD) [4, 5]. Those ions will be accelerated toward the cathode and the field emitter

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array (FEA), aka the electron source, and induce sputtering. It is known that very low energy ions, 500 eV, do sputter differently different surfaces and already produce measurable damages [6, 7, 8]. The damages induced, from any kind of energetic ions, will then reduce the electron emission and the lifetime of the field emitter, as it is the case for AsGa photocathodes, for example, used as polarized electron source for accelerators [9, 10]. During the after pulse, the reversed field will accelerate the ions toward the extracting anodes producing a current of electrons which will also back bombard the field emitter. The gas desorbed can induced a pressure bump which might not disappear before the next pulse, giving room to build a dangerous plasma, which could in a worse case lead to breakdown and sputtering of the anode material to the field emitter cathode. Recent work, and thorough review over a century of vacuum breakdown research, in many areas, [11, 12, 13] is still not enough to choose blindly the electrodes of choice.

In order to investigate the electrodes material, a DC high gradient test stand was built to test different metals susceptible to be used as electrodes in the pulser. The goal is to find the most suitable material, for our needs, which can sustain high field without breakdown, and does emit almost no electron. In situ cleaning by plasma glow discharge was also tested to see whether an improvement was noticeable in the mitigation of the dark current. This technique of gas conditioning to lower the field enhancement factor β has already been reported [14, 15, 16] and used successfully in accelerators to process niobium accelerating cavities, see description in [12], as well as for curing other issues [17].

2 System setup and electrodes preparation

2.1 System setup

The ultra-high vacuum (UHV) system shown on Fig.1, outside its metal confinement bunker for radiological protection, is pumped by a 150 l/s diode ion pump. The average pressure reached is in the low 10^{-9} Torr scale after a quick bake of the ion pump. A more thorough bake brings the pressure in the mid 10^{-10} Torr. An injection line, a leak valve and a Torr capacitance gauge allow the controlled injection of different gas in the system to prepare the glow discharge in between the electrodes.

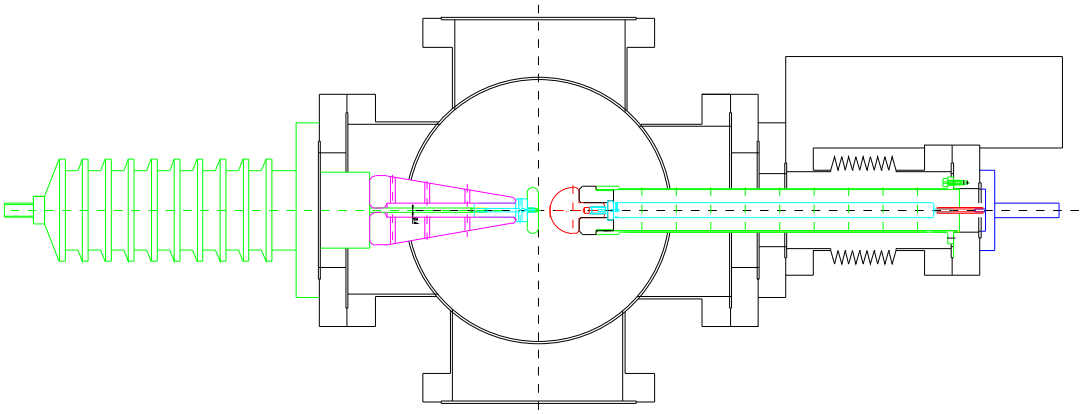

Figure 1: Dark Current -100 kV DC test stand. The flat cathode is mounted on the left side of the system.

A negative and continuous 0 to 100 kV bias is applied to the cathode through an insulating ceramic, left side of the picture (Fig.1). The anode is grounded. The capacitance of the system is closed to 300 pF, hence an energy potentially stored of 1.5 J at 100 kV. The current flowing from the cathode to the anode is measured across a 1 MΩ resistor with a digital Fluke^{TM} voltmeter. The gap separation between the electrodes are adjustable via a translation feedthrough and controlled with a mechanical comparator. The sagging due to the weight at the end of the rods makes that the two electrodes are not centered on each other. We do not expect this off centering to be of any consequences on the high voltage processing. However, it explains the off center damages seen on cathodes. Accurate measurement of the current to the 50 pA level is achieved.

2.2 Electrodes' choice

Due to the long history of research on vacuum breakdown, it seems that the choice of the electrodes should be easy. However, as there is no universal quantifying theory to explain the process of vacuum breakdown depending on the material, its surface state (physical and chemical), it is necessary to do our own test for our own application. In order to pick the most appropriate material, one choose to look at some elements properties. Many tables of elements are then compiled in order to make an educated choice. In our case, our electrodes should sustain a DC pulse of 500 kV and not produce or have a low electron dark current. Also as a FEA will be installed in the middle of the cathode, if any arcing occurs sputtered, or vaporized, anode material will deposit on the FEA. This can lead to the death of the FEA, and the necessity to exchange it; with all the trouble that it implies. We have then look at elements, cf Table.1, comparing the secondary electron yield (SEY) the sputtering rate, the melting point, and the tensile modulus.

Table 1: Secondary electron yield maximum [18], sputtering yield by 500 eV incident Ar [6] and self-sputtering rate at 500 eV of different elements [19, 20].

Elements	SEYmax	Atm/Ar inc	Self Sputter rate	Melting Point T°C	Young Modulus GPa
Cu	1.3	2.3	$>$ 1	1083	110
Al	1.0	1.05	$<$ 1	660	69
Au	1.4	2.4	$>$ 1	1063	78
Ti	0.9	0.5	$<$ 1	1668	116
Mo	1.25	0.6	$<$ 1	2610	329
Zr	1.1	0.65	$<$ 1	1852	68
Fe	1.3	1 (SS 1.3)	$\sim$ 1 [21]	1536	200
W	1.4	0.57	$<$ 1	3410	411
Ta	1.3	0.57	$<$ 1	2996	186
Nb	1.2	0.6	$<$ 1	2415	105

From Table.1, some of the bad candidate could be Cu and Au. Result obtained with RF waveguide support this point [22]. Despite its good electronic and ionic properties Al should be discarded as the combination of melting point and elastic modulus is low, compare to other materials. Al will probably coat thoroughly a FEA in case of arcing, as it happened in a setup with a FEA and a gold coated anode. That implies that spark

processing to reach high gradient, despite being efficient, should be avoided [23, 24], or used with an adequate procedure. Some of the other material which look good in this table, would be probably discarded in regards to their yield strength versus the temperature or their electrical or thermal conductivity. Also the choice of the cathode and anode should be made separately as one good cathode material might not be as well adapted as an anode. All being decided by the geometry of the system, and by evacuation, eventually, of the heat generated by the dark current.

2.3 Electrodes preparation and testing

All our electrodes tested have the same shape, see Fig.2 & 3 for the cathode and anode, respectively. The mean roughness was, by design, defined to be less than $R_a \leq 0.2\mu$m. On the anodes picture, Fig.3), the copper anode, has a hole in the middle. This hole was made to mimic the behaviour of the extracting anode of the 500 kV pulser. None of the other electrodes have this hole. The R_a of the electrodes was checked after high gradient testing.

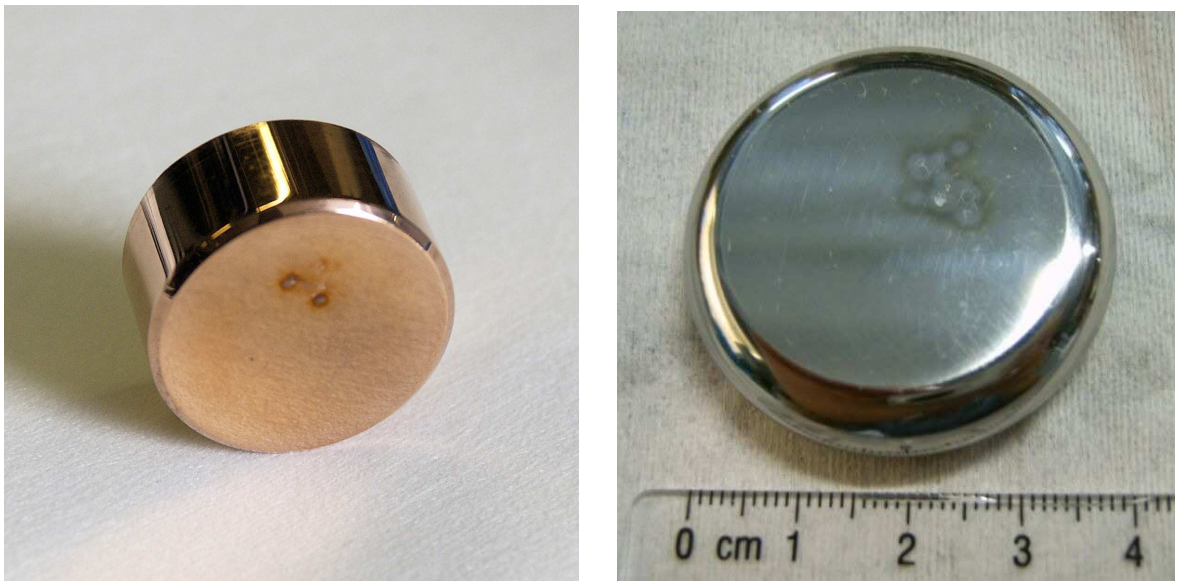

Figure 2: Cleaned Cu cathode after high voltage testing, left. Damaged Mo cathode, right picture Damages can be clearly seen.

The electrodes were cleaned by use of acetone and alcohol in an ultra-sonic bath, before installation in the UHV system. Unless specified otherwise none of the surfaces have been mirror finished. All materials were commercially obtained from Goodfellow^{TM}. Technical materials refer to commercially available material, which is exposed to air before installation.

Ti and Mo electrodes were obtained from the same machining company. Electrodes were thoroughly cleaned in acetone and alcohol before use. Ti electrodes were installed as it, and Mo electrodes were vacuum fired at 900°C for 3 h, during that time. After Mo testing, Ti electrodes were also vacuum fired and reused. From the literature, it was shown that heating up the material is beneficial in improving the breakdown strength [25].

Figure 3: Three anodes used for HV testing, Ti on the left(grey black colour, after vacuum firing), Mo in the center and Cu on the right. Pictures taken after HV testing. Damages can be seen on the Ti electrodes

The processing history followed by the material tested are summarized in Table.2. The procedure of high gradient conditioning is the same for all the cathodes. The voltage between the electrodes is applied for a given gap, 4 mm, 3 mm, 2 mm, 1.5 mm and then 1 mm. The voltage is raised slowly, waiting for stable condition, by discreetness steps, to 60 kV and then the gap is closed with a reduce voltage equal to the previously obtained static electrical field. It was found that above 70 kV arcing sometime happened somewhere else in the system.

Table 2: Measurement history of air-exposed dark current electrodes.

Cathode	**Anode**	**As received**	**Plasma (He - Ar)**	**nth Plasma**
SS	SS	Yes	Yes	Yes
Al	Al	Yes	Yes	Yes
Al mirror Finished	Al (sme as abv)	Yes	Yes	Yes
Cu oxidized	Cu oxidized	-	Yes	Yes
Cu Polynox^{TM}	Cu Polynox^{TM}	Yes	Yes	-
Ti	Ti	Yes	Yes	Yes
Mo vac fired	Mo vac fired	Yes	Solely Ar	Yes
Ti vac fired	Ti vac fired	Yes	Solely Ar	Yes
Cu mirror Finished	Mo vac fired	Yes	Solely Ar	Yes

During conditioning, soft breakdowns might occur. During those breakdowns, current is measured and the pressure can increase by a factor 10. When observed, the voltage is, usually, manually reduced. The pressure recovers in a minute or two, and the voltage is again raised slowly to the previous level.

In this study, we do not reproduce quality preparation achieved in [26]. Instead, the interest is to see what is the behaviour of a technical material prepared with less stringent procedure. As we know that Plasma glow discharge (PGD) is an efficient way of cleaning the surface, and as field emission is also depending of surface contamination. Surface sputtering can be a way to insure the reach of a stable high electric field without breakdown or dark current. Mirror surface finish, obtained using not so trivial technique, will certainly be wasted during an agressive GD.

Plasma glow discharge is usually applied after we reached 1 nA of current at 1 mm

gap from the as received state. The gases injected for the GD are usually a mixture of He and Ar, with a composition of 50% He and 50% Ar. Sometime pure Ar is solely used. The total pressure is in all cases comprises between ∼0.15 Torr and ∼0.25 Torr. Noble gases are chosen to avoid chemistry on the surfaces. Helium is chosen because, for the same energy, its sputtering potency of material is less than Ar. The gases are coming directly from compressed gas cylinder and are injected via the leak valve. A +400 V to +600 V bias is applied between a third electrode, not shown on Fig.1, and the two electrodes to be tested. The pressure and the energy of the ions in the GD are adjusted, so that the plasma wraps around the electrodes. The distance between the three electrodes are around 6 cm and the time of the PGD can last between 40 to 60 minutes.

Finally, as it is known from literature that pressure can affect the breakdown onset threshold, and that dark current appearance is affected by the gas species [11]. The system is baked not only after air venting but also after each plasma. By this mean we are minimizing any role that the pressure and the gas composition would have in FE or arcing.

3 Results

Before presenting results obtained with our electrodes, it is of importance to have in minds results obtained by Furuta *et al* [26, 27] and Diamond [25, 28]. According to Furuta's publication, the design of their electrodes are equivalent to ours. They have obtained for stainless steel, Cu, Ti and Mo with mirror finished surfaces the results summarized in Table.3. Those results have been obtained not only with mirror finished surfaces, but the assembly of their system and the mounting of their electrodes, were done in class 1 and class 10, respectively, clean room.

Table 3: Field gradient (MV/m) between electrodes obtained at 1 mm gap for 1 nA of dark current or with no field emission (FE), second row.

	SUS	Cu	Ti	Mo	Mo - Ti	Al	Nb
1 nA [26]	36	47.5	88	84	103	-	-
No FE [25]	-	70	60	-	-	85	92

All the current vs electric field plots presented in [26] and in this work can be fitted using the following $I = c\ E^2\ e^{-a/E}$, see Fowler-Nordheim equation (equ.1), with I being the current, c and a are some constants, and E is the applied electric fields. From those fits F-N parameters, area and the field enhancement β, can be extracted.

$$I = A \cdot \frac{1.5 \cdot 10^{-6}}{\Phi} E_s^2 \cdot e^{\frac{10.4}{\sqrt{\Phi}}} \cdot exp\ (\frac{-6.83 \cdot 10^7 \Phi^{\frac{3}{2}}}{E_s}) \quad (1)$$

where $E_s = \beta \cdot E$ and the work function, for Mo, Φ is taken equal to 4.2 eV. However, the aim of this paper is not to try to compare another list of F-N parameters, nor trying to understand the field enhancement dependance vs the gap etc... Hence, all these topics won't be discussed here. The goal is to report how surface conditioning can lead to stable operation under the presence of an high electric field. It is also to report on the erosion of the materials upon the field processing.

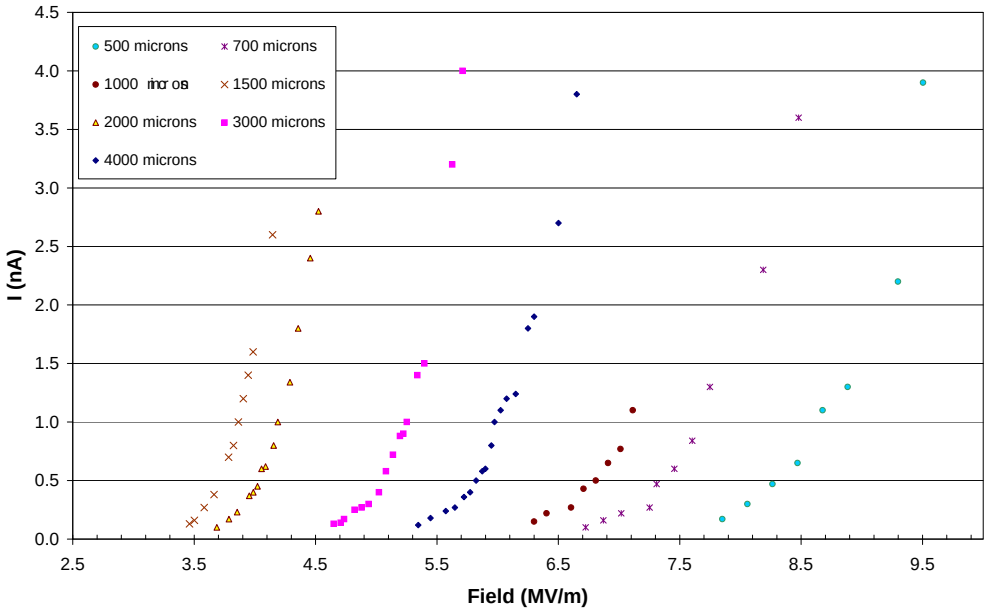

Figure 4: Dark current of Al electrodes after a system bake at 190°C for 100h and after electrical conditioning of 29 kV at a 4 mm gap drawing 7 nA of current from the cathode to the anode.

3.1 Aluminium Results

3.1.1 Al-Al electrodes

Pristine as received Al electrodes were tested. The first test after a thorough bake of the chamber led to a dark current of 1nA at a gap of 1 mm for a field of 7.1 MV/m. The gap between the electrodes was set at 4 mm and the electrodes were conditioned overnight by applying 29 kV and drawing 7 nA of current. The next day the dark current measured increased to 13 nA. Several dark current curves were then produced and compared to the as received test, Fig.4. The obvious conclusion is that at this level this DC electrical conditioning did not lead to any improvement.

The subsequent test was to clean and condition using first an He plasma of 0.26 Torr. The sputtering rate of 500 eV He ions on Al is 0.16 [6]. The 1 nA at 1 mm gap is reached for a field of 13.5 MV/m. Subsequent He plasma kept improving the results. However, a few breakdown did happen during the voltage processing. The best improvement was obtained after an ArGD at a pressure of ∼0.1 Torr and results are presented in Fig.5. The 1 nA at 1 mm gap (full circles) is reached for a field of 42 MV/m. When leaving the system at this level of field and dark current an improvement, decrease of dark current over time, is observed Fig.5 full circles. In subsequent test, also with different materials, this improvement was occasionally observed. However, in some cases the current increased more than the double of the reached value.

In the next test an He-Ar plasma was used to clean the same electrodes. The behaviour, after pumping out the noble gases, was that no FE was observed until breakdown. The Al electrodes did hold stably (12hours) a field of 42 MV/m without dark current at 1.5 mm gap. At some point in time, when holding the field at 45 MV/m an arc occurred and emission current 350 nA could be measured. Nevertheless, damages due to this breakdown as well as the other occurring during the voltage processing, where not sufficiently

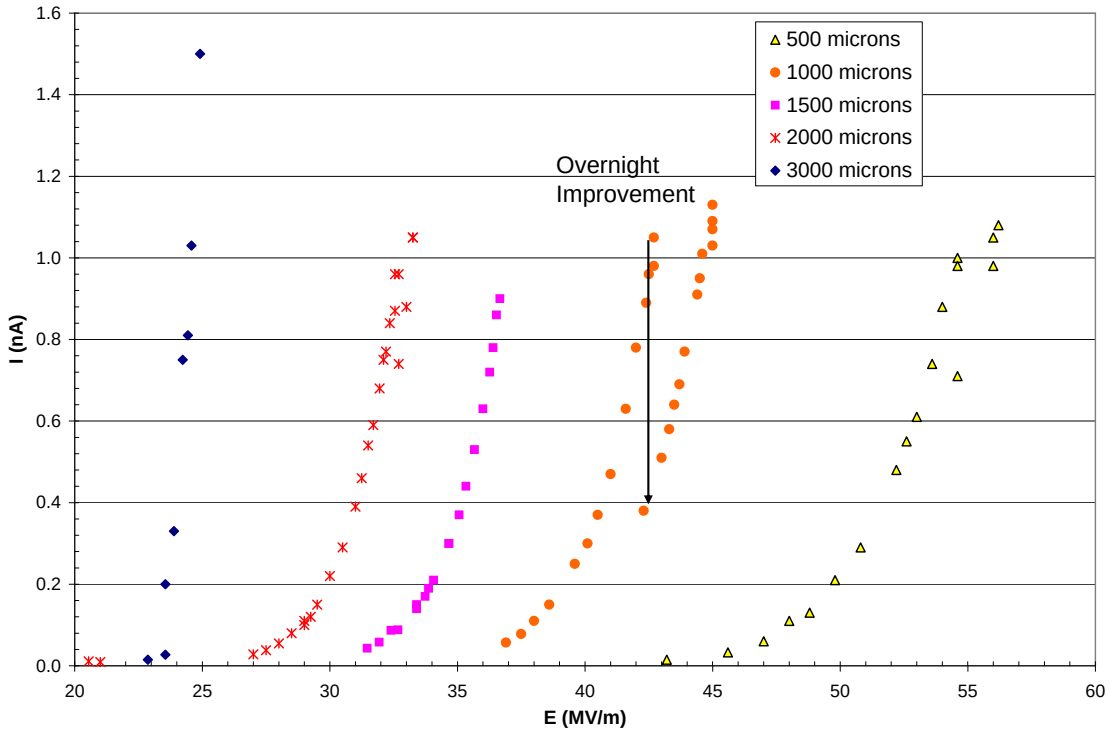

Figure 5: Dark current of Al electrodes after an Ar Glow Discharge, following the previous He processing and high field conditioning.

severe that still a current of 1 nA at 1 mm was measured for a field of 41.5 MV/m. Finally, the best result obtained playing this game of He-Ar GD, was to hold a field of 52 MV/m at 1 mm without dark current. However, at some point an arc more violent than the previous one, will sufficiently damage the cathode that no more GD will be able to restore the holding of the high electric field. A summary of the performance obtained with Al electrodes is shown in Table.4.

Table 4: Electric field in MV/m held in between two Al electrodes at 1 mm gap for the given dark current in nA.

	Al - Al		Al mirror finished - Al	
State / Dark Current	$<$ 0.05 nA	1 nA	$<$ 0.05 nA	1 nA
As Received	-	7.5	36 (2 mm)	29
After Plasma	52	30	73 (stable) (92 at 750μm)	31

3.1.2 Mirror Finished Al Anode

We then replaced the damaged Al cathode by a pristine mirror like finish Al (6082) cathode, machined to an R_a of 3 nm. The previously damaged mushroom anode was reused, after having been solely wiped with alcohol before reinstallation in the UHV system. The damages on the anode were localized around the summit of the anode, and similar to the one shown in Fig.6 right picture.

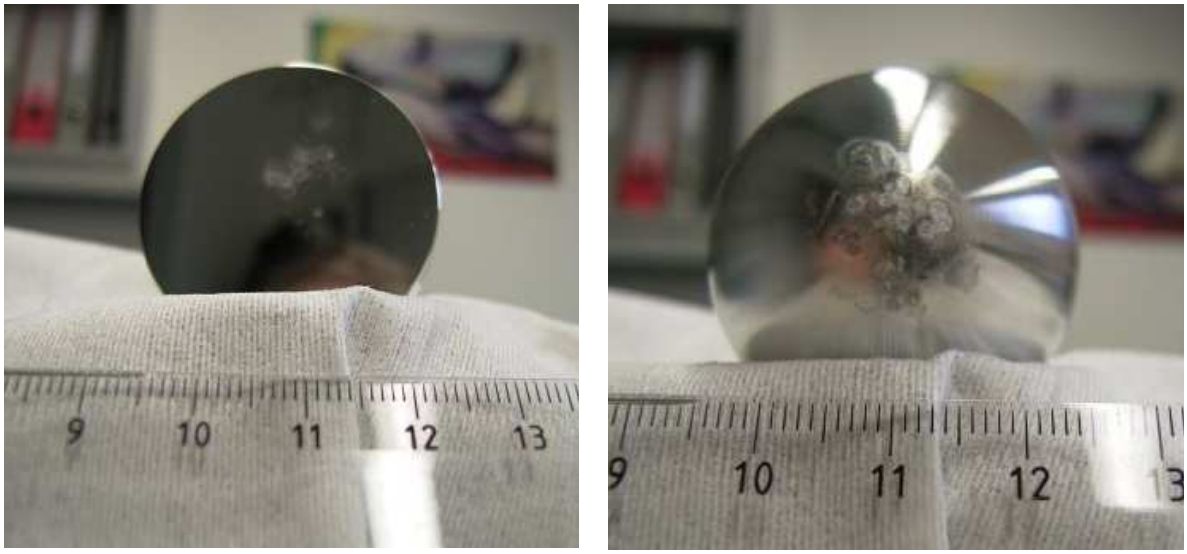

Figure 6: Breakdown damages withstood by the Al cathode (mirror finished-left) and anode (right) during the conditioning period. Scale in cm.

With this configuration, the as received system held stably without dark current, an electric field of less than 36 MV/m. At this value, an arc occurred. Inspection of the cathode through the viewport of the UHV system showed pitting, hence damages. An He-Ar GD was then applied to cure and clean the electrodes. Results are summarized in Table.4. The column labelled <0.05 nA shows the field strength held without measuring any FE. The System held the 90 MV/m electric field at 750 μm, and broke down at 92 MV/m. The vacuum arcing was so severe that no further PGD was able to restore such fields. Final damages of the Al electrodes upon removal are shown in Fig.6. The results show that the breakdown is cathode initiated as the cathode was pristine and the anode was already severely damaged.

3.2 Copper results

Oxidized copper electrodes were tested solely after a PGD. Even after a PGD and the voltage processing, the electrodes are still very oxidized. Cleaner spots around the hole of the anode were observed at the end of the testing. Damages on the cathode were also visible. The electrodes were then cleaned by chemical etching by use of a phosphoric based solution Polynox®. They were subsequently rinsed under tap water and after drying, cleaned with ethanol. A last test was conducted by installing a mirror diamond turned OFHC Cu cathode ($R_a \sim 3$ nm) and the already used Mo and then a SS anode.

3.2.1 Cu-Cu electrodes

The results obtained seem to show that there is no influence from the anode hole in the obtention of the high gradient. If we compare to results in Table.3 [26] for clean copper. All damages were localized around the hole of the mushroom. Comparing to Al, craters in the Cu were neither as deep, neither as extended see Fig.2 and the 2 spots, compared to damages in Fig.6. The damages on the anode did stay localized near the hole. That suggest two possibilities without excluding a combination of the two. The energy in the

Table 5: Electric field in MV/m held in between Cu electrodes at 1 mm gap for the given dark current in nA. (*) obtained at 3mm gap

	Cu oxidized		Cu clean		Cu-Mo	
State / Dark Current	< 0.05 nA	1 nA	< 0.05 nA	1 nA	< 0.05 nA	1 nA
As Received	-	-	24	26	18.2 (*)	13.8 (*)
After Plasma	32	29.3	55	19	21.6	25.4

arc was not sufficient to vaporize the Cu materials, by melting and sputtering the melted Cu; and thus despite that Cu has a higher sputtering rate than Al. Or the field was not strong enough to pull out droplets of Cu which could have then been vaporized [29].

3.2.2 Mirror finished Cu anode

As results from Al seems to indicate that the breakdown is cathode initiated, a combination of a mirror finished cathode with previously used Mo (vacuum fired) and SS anode was tested. Those anodes have sustained damaged far less important than the Al anode pictured in Fig.6. Results of the Cu-Mo conditioning is shown in Table.5. The conditioning of the "as received" electrode happened following the "breakdown processing" or spark processing, and thus until a more severe breakdown brought the Cu anode to emit at the level of 800 nA, at 3 mm gap. During the first 500 eV ArGD, small breakdown could be seen on the Cu cathode. Probably being dust burning away. Results obtained after plasma processing did improve the situation but not to the level of the two clean Cu electrodes. It was expected that higher fields could be reached, as in the case of using two Cu electrodes, Table.5, or two Mo electrodes (see Table.6).

The Mo anode was exchanged for a SS anode, and the Cu cathode was turned 180 on its axis. As the anode arm sags, damages on the cathode are not localized on the center of the anode, hence allowing to expose a pristine area. the maximum field held with this pair was 11 MV/m at 3 mm gap, and thus after an ArGD. From these last result, no conclusion should be drawn on the coupling of this last pair, as some experimental hypothesis could explain them.

3.3 Stainless steel, titanium and molybdenum results

Main results obtained, at 1 mm, for stainless steel (SS), Ti and Mo are summarized in Table.6.

3.3.1 Stainless Steel

SS electrodes were electrically processed the same way as Al electrodes. After plasma treatment, the best field achieved was 68 MV/m with dark current below the 10 pA detection limit. The latest breakdown, was not recoverable by the use of a PGD. Upon removal of the electrodes, damages were located at the top of the mushroom. However, craters were not as profound or as wide as for Al electrodes (in Fig.6). The damages are less extended as the one observed on the Cu electrodes. If we looked at the data in Table.1, it is then not surprising. Effectively, both the melting point and the young

Table 6: Electric field in MV/m held in between two SS two Ti and two Mo electrodes at 1 mm gap for the given dark current in nA.

	State / Dark Current	< 0.05 nA	1 nA
SS	As Received	40	42.5
	After Plasma	68	35
Ti	As Received	50	46.6
	After Plasma	63	67 (0.1nA)
Ti Vac Fired	As Received	29.6	32.5
	After Plasma	39	41.4
Mo Vac Fired	As Received	37	45.2
	After Plasma	44	61.3

modulus of SS are far above the copper's values. However, as tiny amounts of vaporized or sputtered material from the anode can be prejudicial to the working of FEAs; avoiding even soft breakdowns seems to be a good strategy.

3.3.2 Titanium

In the case of Ti, we have observed after plasma treatment, a stable field holding at 52 MV/m without FE. From 53 MV/m on, dark current appeared and reached 1 nA at 62 MV/m, cf Fig.7 (diamonds). The field held there for a few minute until arcing, bringing the current above 1 μA. The field was then reduce to 35 MV/m in order to get a 1 nA current value, cf Fig.7 (squares.) This current value did decrease over 56 hours of 35 MV/m field processing. A few MV/m were then gained to bring back the current to 1 nA. It is possible that after a soft breakdown leading to dark current emission around 1 μA, no rise in pressure can be detected. At a pressure of 2.10^{-9} Torr, gas released by ESD process can in principle be detected. A simple calculation will show that such a current will produce a gas flux of $\sim 3.10^{-9}$ Torr.l.s^{-1}, hence an increase of pressure of $\sim 2.10^{-11}$ Torr; assuming an ESD coefficient of 10^{-2} molecule/electron. This is below the resolution of our instrumentation.

A subsequent ArGD for 90 minutes at 580 eV under pressure varying from 0.156 Torr to 0.174 Torr was done. During this PGD, the Ar gas was evacuated several time and then replaced by new Ar coming from the cylinder. The field held after this plasma was 61 MV/m. The voltage was then increased and fluctuation in the emitted current below 50 pA were observed, above 63 MV/m the dark current was fluctuating around 0.1 nA and thus until 67 MV/m, see Fig.8. Field conditioning over a week showed, like in the case of Al or SS, an improvement in the dark current emission. The final stable electric field obtained for 1 nA at 1 mm gap is 55 MV/m, see insert in Fig.8. Above this level of field, the dark current does not diminish but it increases with time, Fig.8.

Upon inspection of the electrodes after their extraction from the chamber; damage spots can be seen on either one of the electrodes. Damages are located on the anode around the top of the mushroom, and 4 separated spots can be seen on the cathode spread over a trail of 1 cm long. Damages on Ti anode are similar to the one seen on the SS and Cu anodes, in extension and morphology (melted area).

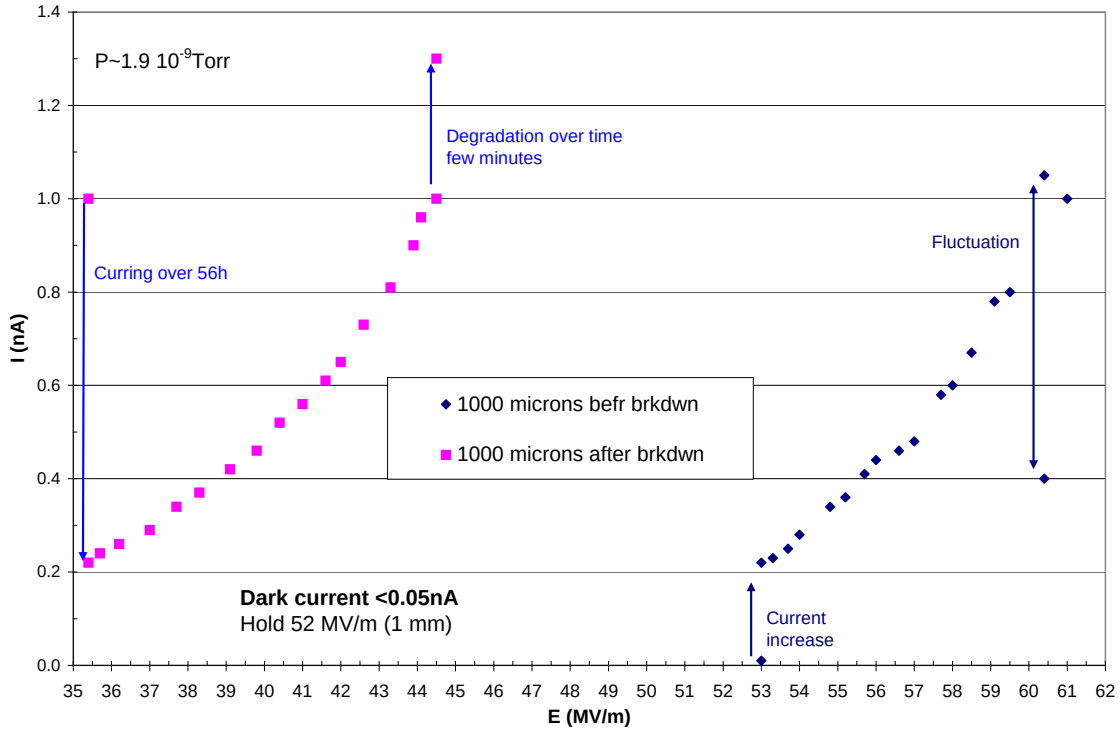

Figure 7: Dark current evolution of Ti electrodes after a 2^{nd} Ar Glow Discharge. The square curve is obtained after a surface breakdown at 62 MV/m

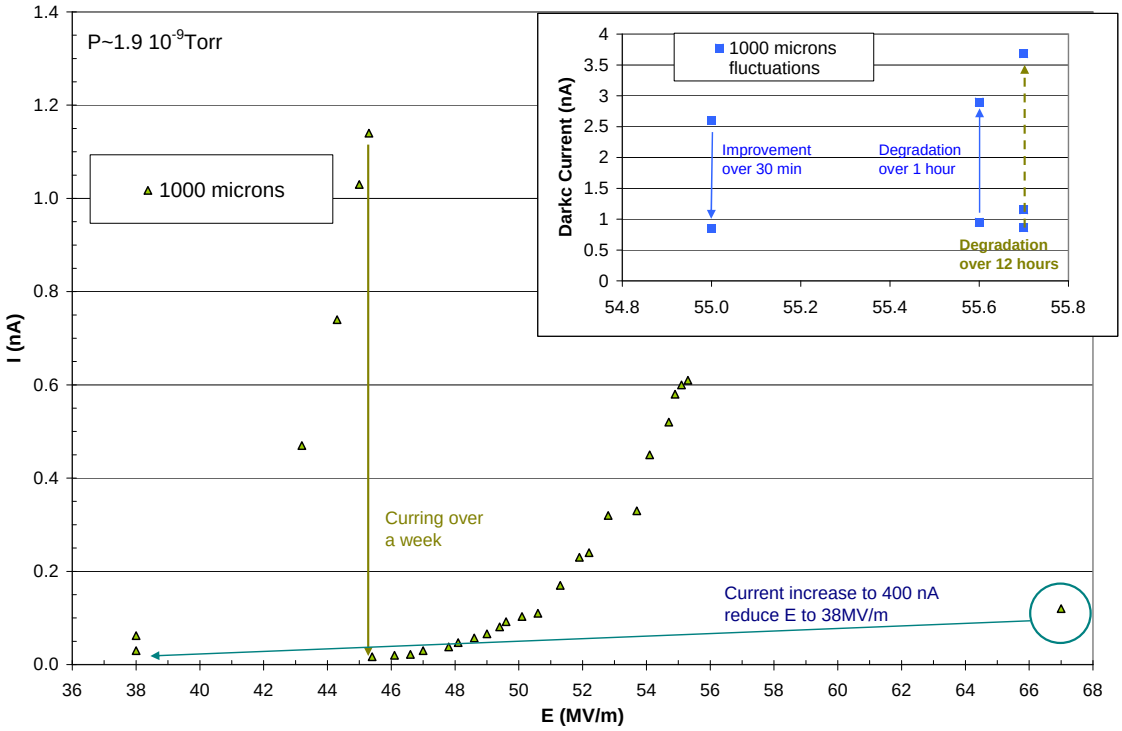

Figure 8: Dark current evolution of Ti electrodes after a 3^{rd} Ar Glow Discharge.

3.3.3 Molybdenum

Vacuum fired Mo electrodes were installed in the system, the best results are summarized in Table.6. A simple 25Hz optical camera was viewing the electrodes hence the space in between. When running, no light is present in the system enclosure. During the soft events, a flash is seen on the TV screen and a jump in current intensity, below 0.1 nA, is measured sometime associated to a pressure increase. The flash is localized and take the full space occupied between the electrodes. During harder breakdowns, sound can be heard coming from the enclosure. After these soft breakdowns, the voltage could still be increased. In order to have a chance to detect breakdown precursor, fast acquisition and very high sensitivity, to single photon, are mandatory. However, such simple optical system can be used as an interlock system for our FEL.

As for the Ti electrodes, dark current appears at some voltage. Raising the field increased the dark current until breakdown occurs. Dark current plots from as received electrodes(triangles), and after three plasma treatments are presented in Fig.9. The first ArGD push the limits of the dark current onset (diamonds) until a hard breakdown occurs, bringing the current over the μA. The onset and the 1 nA limit is then greatly reduced (squares). The second ArGD allows to partially recuperate from this breakdown (asterisks). A third plasma He-Ar, does not bring any extra improvement (crosses in squares). Mo conditioning from broad electrodes at large gap ($> 500\mu$m) is similar as conditioning with short gaps. Mo reach higher gradient than Cu but it sparks more to get there [24], hence showing more damage than Cu, Fig.2. From this, it is obvious that a special electrodes conditioning procedure must be used to process them, and thus to protect the electron source.

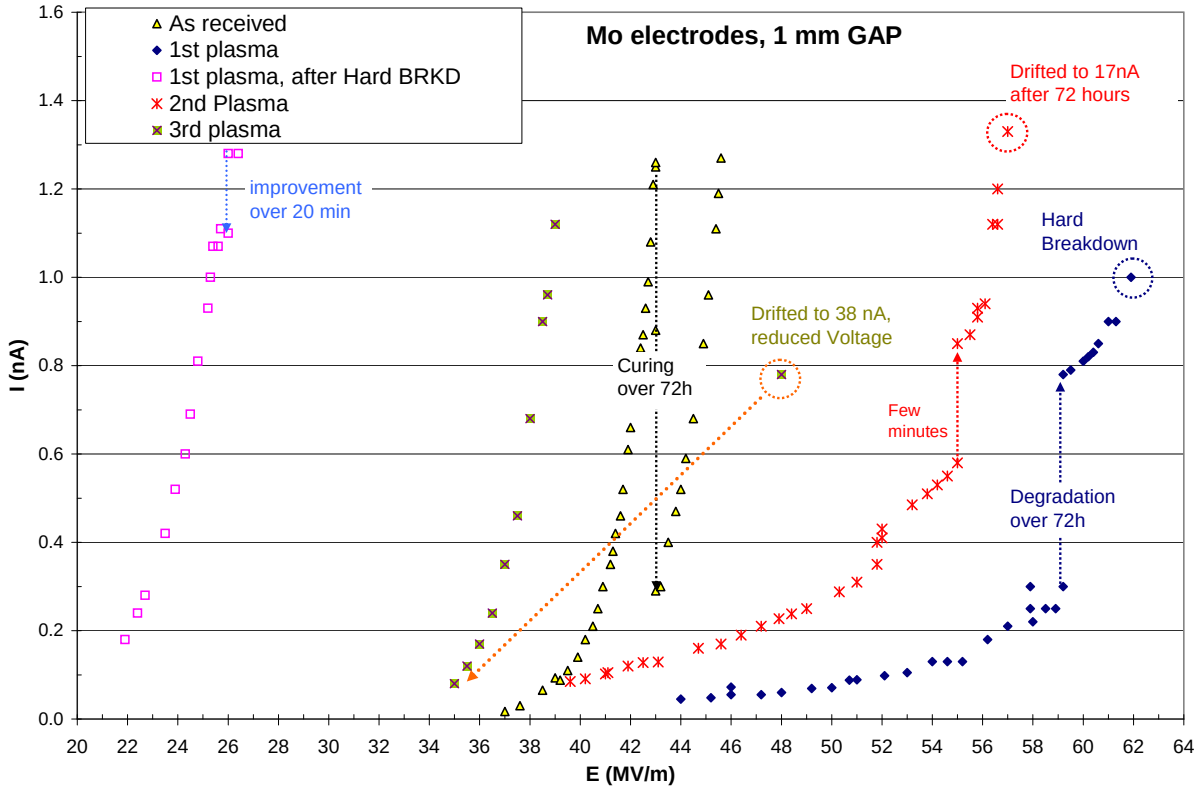

Figure 9: Dark current evolution of Mo electrodes after processing.

3.3.4 Titanium vacuum fired

Original Ti electrodes were re-installed after vacuum firing and the results, for comparison with non fired Ti, are presented in Table.6. After vacuum firing the Ti became gray black. This color can be the mark of TiH_2 or TiO_2 in the rutile form, or Ti_3O_5. Damages, all localized on the top of Ti anode, can be seen on Fig.3. Upon a close look, the involuntary coating is removed by the severe breakdowns sustained by both electrodes.

Given the rather poor performances, in field holding for the Mo electrodes, compare to literature values, and the colour of the Ti electrodes; it is suspected that the vacuum firing did degrade the Mo properties instead of improving them. The pressure in the vacuum furnace should have been around 10^{-5} Torr. Ti and Mo cathodes were sent for surface analysis, X-ray photoelectron (XPS) and Auger (AES) spectroscopy. Upon AES analysis, both electrodes surface exhibit high contents of carbon (C) and oxygen (O), higher than normal air exposure. Nitrogen (N) is also present on the Mo surface. Nitrogen cannot be seen on Ti as Ti and N overlaps under AES analysis. Under XPS analysis, a shift of about 5 eV to higher binding energy, for both Mo $3d_{5/2}$ and Ti $2p_{3/2}$ is observed. Such shift can be the marked of TiO_2 and MoO_3 [30]. The Mo shows no discoloration, suggesting the formation of a thin trioxide film. The conclusions of it are, first of all a small air leak might have been present in the furnace. Secondly, a bad heat treatment will bring the opposite results in term of field holding.

4 Conclusions

Plasma glow discharge is a very effective way to enhance the DC field holding in between two broad electrodes. It allows also to restore and sometime improve the DC field limit achieved after a breakdown event leading to dark current emission. This dark current follows the FN law.

The downside of such treatment, for Al, Cu and SS electrodes, is that the surfaces hold the field until breakdown with no or little warning. No increase of pressure is recorded, but sometime some current variation in the tens of pA might appear. Such fluctuations can be the sign of a forthcoming breakdown, if any, but the time scale can vary from minutes to hours. In the hunt of the precursor breakdown, in the framework of an interlock for the 500 kV pulser, some highly sensitive and fast photomultiplier will be tested in this 100 kV DC test stand.

For Ti and Mo electrodes dark current appears and increases to some intensities until breakdown occurs. However, during the processing a few sparks occurs, sometime in a "spitfest" regime. Those sparks are beneficial as they condition the surface. Dark current at a level of 1 nA can either drift to hundreds of nA, fall back to less of hundred of pA or stay stable. So far, the prediction for its evolution is only empirical.

Finally, we have without stringent procedure match, or exceeded results obtained by other labs. However, results for non mirror finished Ti and Mo were below the ones obtained elsewhere. In the case of Mo, it is suspected that the vacuum firing did contaminate the Mo as it did for Ti, leading to poorer performances as usually reported in the literature.

In order to find the Grail material, the which will hold our requested field without emitting dark current, Niobium seems to be a material of interest. Ion implantation, with

nitrogen, is known to harden materials [31, 32]. It is maybe possible that this technique of hardening can be useful to increase the breakdown threshold of soft materials, as it seems to have for harder ones [33]. Dark current from electrodes can be lowered by depositing a pure monolayer of oxygen on the surface; the which will increase the work function of the electrodes. However, and in the framework of an accelerator electron source, this layer might have to be regenerated frequently as back bombardement from residual gas ion will cleaned up the surface.

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