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Instrumental Developments for In-situ Breakdown Experiments inside a Scanning Electron Microscope

Muranaka, T (Department of Physics and Astronomy, Uppsala University) *et al*

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Corresponding Author: Dr. Tomoko Muranaka, Ph.D.

Corresponding Author's Institution: Uppsala University

First Author: Tomoko Muranaka, Ph.D.

Order of Authors: Tomoko Muranaka, Ph.D.; Tobias Blom, Ph.D.; Klaus Leifer; Volker Ziemann

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Instrumental Developments for In-situ Breakdown Experiments inside a Scanning Electron Microscope

T. Muranaka[a], T. Blom[b], K. Leifer[b], V. Ziemann[a]

[a]*Department of Physics and Astronomy, Uppsala University, Uppsala, Sweden*
[b]*Department of Engineering Sciences, Uppsala University, Uppsala, Sweden*

Abstract

Electrical discharges in accelerating structures are one of the key issues limiting the performance of future high energy accelerators such as the Compact Linear Collider (CLIC). Fundamental understanding of breakdown phenomena is an important part of the CLIC feasibility study. The present work concerns the experimental study of breakdown using Scanning Electron Microscopes (SEMs). A SEM gives us the opportunity to achieve high electrical gradients of 1 kV/μm which corresponds to 1 GV/m by exciting a probe needle with a high voltage power supply and controlling the positioning of the needle with a linear piezo motor. The gap between the needle tip and the surface is controlled with sub-micron precision. A second electron microscope equipped with a Focused Ion Beam (FIB) is used to create surface corrugations and to sharpen the probe needle to a tip radius of about 50 nm. Moreover it is used to prepare cross sections of a voltage breakdown area in order to study the geometrical surface damages as well as the elemental composition of the breakdown.

Keywords: Electron microscopy, Field emission, Breakdown, CLIC

Email address: `tomoko.muranaka@physics.uu.se` ()

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1. Introduction

The feasibility of future high power accelerators for particle physics studies such as the Compact Linear Collider (CLIC) are under investigation. The high accelerating field of 100 MV/m is generated by sending radio-frequency (rf) waves through specially designed accelerating structures. The high electrical fields tend to cause surface degradation of accelerating structures [1]. The probability for an electrical breakdown will increase with the created surface roughness which is critical to the performance of the accelerator. One of the key conditions for the reliable operation of CLIC is limiting the breakdown probability on the order of 10^{-7}. In order to obtain that condition, studies of fatigue of the surface under rf condition with prototype accelerating structures [2, 3] and direct current (dc) breakdown experiments for example [4, 5, 6] are under way at CERN, SLAC and KEK. The dc experiments have similarities in many aspects to high gradient rf tests but are more easily equipped and controlled. However, existing dc experiments use electrodes on the mm scale to give high gradient electric field on the sample that is much larger than the surface roughness of the accelerating structure. This macroscopic approach needs to be improved with respect to the following issues

a) observed breakdowns and measured field emissions are averages in mm^2 areas;

b) the surface conditions of both electrode and sample surface are unknown during experiments;

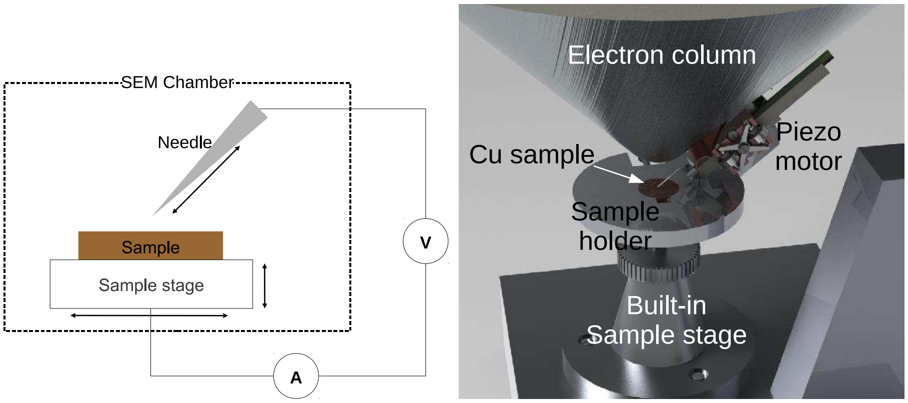


Figure 1: Schematic of experimental setup for field emission measurements and breakdown study (top), drawing of inside view of the ESEM chamber (bottom): both Cu sample and piezomotor are set on a same holder plate that is mounted on a built-in sample stage.

c) the gap between the electrode and the sample surface is not known to μm resolution.

The present work is intended to overcome these issues and provide an opportunity to investigate localized breakdown phenomena by conducting dc experiments inside a Scanning Electron Microscope (SEM). By using a SEM, we achieve the resolution of the electron probe in the few-nm range, which is of great advantage as the surface roughness of the polished accelerating structures is in the same scale. It is noteworthy that this is also a unique experiment in material science due to its mesoscopic approach that bridges atomic scale (with atomic force or tunneling microscopes for example) and macroscopic (mm) optical scale. The present paper will report on instrumental developments for SEM in-situ experiments and analysis methods to investigate the effect of a voltage breakdown on the surface.

2. Instruments

2.1. Experimental setup in the ESEM

A drawing of the experimental setup is shown in Figure 1. This setup is installed in the vacuum chamber of a FEI XL30 Environmental SEM (ESEM) equipped with a field emission gun at the Ångström laboratory at Uppsala University. Measurements of field emission and breakdowns are conducted mainly in this microscope. In the present work, a copper sample in the form of circular disc with the diameter of 12 mm and the thickness of 2 mm is fixed in a hole of a portable sample holder. The holder is set on a built-in sample stage of the ESEM which has five degrees of freedom of motion: three linear (x, y and z), rotation and tilt. The stage is electrically insulated from the ESEM chamber. A piezoelectric linear motor Piezo LEGS Linear 10N Non Magnetic Vacuum (PiezoMoter AB [7]) is mounted on a ramp attached on the same holder. The ramp keeps the piezo motor linear motion in 50 degrees with respect to the sample holder plane in order to avoid touching the objective lens of the ESEM and to be perpendicular to the sample surface. A tungsten needle with a typical length of 15 mm is attached to an edge of the rod of the piezo motor that is electrically insulated from the motor chassis. The commercially available needle has a diameter of 500 μm with a tip radius of about 500 nm. The tip can be sharpened further as is explained in section 2.2. The needle can be moved close to the surface of the Cu sample by the piezo motor. A PMD90 micro-stepping driver controls the motor with a longitudinal positioning resolution in the several-nm range. The thin needle tip permits to investigate field emissions from a localised area on the surface, thus addressing point a) in the table above. Field emission currents

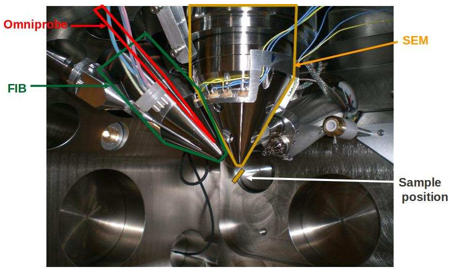


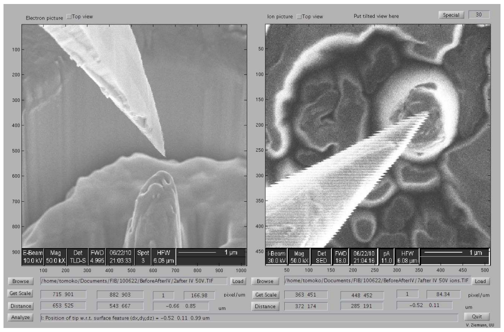
Figure 2: Inside view of the FIB: the FIB column is mounted at 52 degrees at an angle of 52 degrees with respect to the standard SEM column. The focusing distance of both beams are fixed in order to intersect at the sample surface. A W needle is mounted on the omniprobe manipulator.

between the needle and the sample are measured with a Keithley 6430 sourcemeter. The background current level of the experimental setup is limited to about 30 pA when the electron beam is turned on. With this low background current, one can observe surface conditions of both anode and cathode before, during and after experiment on site and overcome the problem b). It enables us to fix the target surface condition in the sub-μm range and provides an opportunity to investigate the evolution of the surface under high electrical field gradient condition. The images are typically acquired by using the standard secondary electron detector. A tomographic technique allows us to determine the distance between the tip and the sample from SEM images: As the sample setup is fixed on one holder mounted on the stage, one can take images from a different point of view by tilting the stage. Images from two different angles enables us to determine the gap between the anode and the cathode with sub-μm accuracy, thus addressing point c) on the list.

2.2. The Focused Ion Beam Microscope

Considering the plasma-assisted arcing model [8, 9], it is desirable to investigate the geometrical dependence of field emission on the topography of the sample. Therefore, it is important to be able to modify the topography of both the tip and the sample surface. A Focused Ion Beam (FIB) Dual Beam device (FEI Strata DB235) is applied as an essential part to optimise and understand the breakdown and tunnel-current experiments. The FIB is used for 3 purposes in this work: 1) pattern the surface by ion sputtering, 2) optimisation of the radius of curvature of the manipulator tip, 3) the depth analysis of surface transformations of the sample under or close to break down conditions. The FIB instrument consists of an ion beam column mounted at an angle of 52 degrees with respect to the SEM column. The instrument is also equipped with an Omniprobe$^{\mathrm{TM}}$manipulator to which the same tungsten needle that is used on our custom-made manipulator can be attached. An inside view of the FIB instrument is shown in Figure 2. The samples are tilted by 52 degrees for patterning by the perpendicularly incident ion beam. Note that images can be taken by both the SEM and the FIB immediately after patterning. An example of surface patterning by creating a pillar is shown in Figure 3 taken by SEM (left) and FIB (right). The size of the geometrical shapes in this pattern can be defined with a precision in the 10 nm range.

The FIB can be used to vary the manipulator needle sharpness as well. A needle mounted on the omniprobe manipulator can be sharpened to the tip diameter of about 50 nm. Small corrugations on the sample surface can therefore be precisely addressed and biased by the sharpened tip. Varying



/home/tomoko/Documents/FIB/100622/BeforeAfterIV/2after IV 50V.TIF

715 901 | 882 903 | 1 | 166.98 pixel/um

653 525 | 543 667 | −0.66 0.85 um

/home/tomoko/Documents/FIB/100622/BeforeAfterIV/7after IV 50V ions.TIF

363 451 | 448 452 | 1 | 84.34 pixel/um

372 174 | 285 191 | −0.52 0.11 um

I: Position of tip w.r.t. surface feature (dx,dy,dz) = −0.52 0.11 0.99 um

Figure 3: Capture image of the Matlab analysis program for the gap height calculation. Images of a needle and a test surface pattern are taken by SEM (left) and by FIB (right) in FIB instrument. Here the milling time is about 7 minutes for the pillar with an ion energy of 30 keV and a beam current of 3 nA. For further refinement of the pattern, lower beam currents down to 10 pA are used.

combinations of controlled surface pattern and needles with different radius of curvature will give us the information on the geometrical dependence of field emission.

As an example of the tomographic determination of the distance between the manipulator tip and the sample surface, a pair of images at different angles, here 0 and 52 degrees, is taken in Figure 3. The analysis program was developed with Matlab® [10] and gave a tip-sample distance of 1.0 μm. The program can be used to analyse pairs of images of the sample and the tip taken at different tilt angles in the ESEM discussed in the previous section. Furthermore, surface corrugations can be analysed by this program.

3. Experiments

As the first step of experiment with the newly developed equipment, we report in this section a demonstration of breakdown in the ESEM and the depth analysis of surface transformation after the breakdown. Further experiments such as tunnel current measurements still remain to be seen.

A breakdown experiment in the ESEM was carried out with a tungsten needle and a diamond-turned copper sample. The needle was accidentally bent prior to the experiment with a bi-forked tip which results to give only a rough estimation of the gap about 2 μm to the sample surface. The breakdown occurred by putting negative voltage of more than 500 V on the needle. As shown in Figure 4, both the needle and the sample surface melted and the morphology was modified. The tip is damaged up to a region where it is about 2 μm thick, whereas droplets of molten material in a size range of 0.1 to 1 μm can be identified in a region of approximately 20 μm diameter. The exact place of the breakdown cannot be identified, but in view of the damage

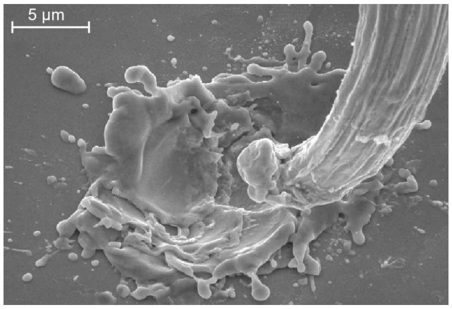


Figure 4: A SEM image of the first breakdown inside ESEM. Both cathode and anode are damaged. Note that the strong bending of the W tip was already present prior to the breakdown experiment.

caused on the sample, the place where the arc occurred was most likely not more than 2 to 3 μm away from the actual tip position. Considering that the sample surface is flat with a roughness in the some nanometer range, we expect that the arc forms symmetrically around the original tip position. The fact that the tip is not in the centre of the damage of the Cu surface could be related to heating and partial melting of the W tip which, in turn, might have caused an additional bending of the W tip. In order to investigate the modifications of the sample structure near the surface we use FIB to sputter a rectangular recess into the sample in the breakdown region. The region of interest was coated with a thin film of Pt in order to protect the structures caused by breakdown, using electron and ion beam induced deposition prior to sputtering [11]. Observing a side of the recess with the SEM or with the FIB by tilting or rotating the sample permits us to observe a cross section through the sample surface [12]. In the SEM image shown in Figure 5 the Pt film is visible as a surface layer. Projections of a rounded protrusion with a diameter of 0.3 μm and a petal-like structure with a thickness of sub-micron are shown under the Pt layer. Some white spots in the petal indicate the composition of the formed feature with different materials. Layers with different contrast are also shown in the thickness of about 0.3 μm from the top of the flat sample surface. It might be possible to determine the energy deposited on the surface during breakdown by calculating melted volume of the sample by controlling the numbers of breakdown.

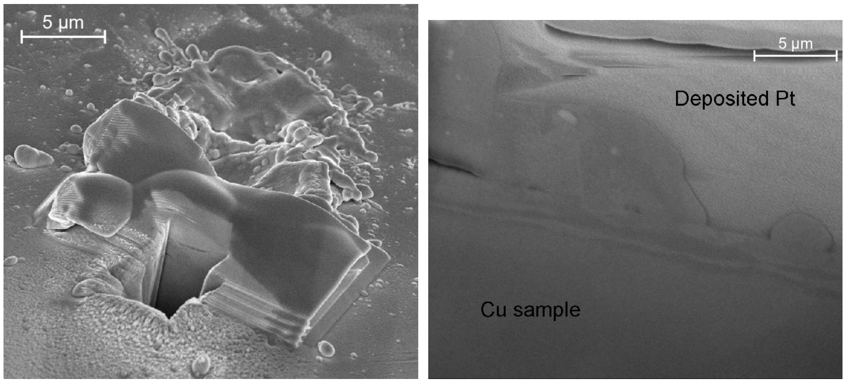


Figure 5: SEM images of breakdown area partially covered by Pt and milled by staircase patterning (top) and closeup of its cross section (bottom). Petal-like structure with thickness of submicron has clearly seen on the surface which caused by breakdown.

4. Conclusions and Outlook

In the present work we have developed an experimental setup for the electrical and structural analysis of field emission and vacuum discharges phenomena at the sub-micron scale. This set-up consists of a piezo controlled in-situ nanomanipulator tip inside an SEM. We demonstrated the use of a FIB for structuring of the cathode (sample) and anode (tip) surface to enable field emission measurements as a function of sample topology and tip radius (down to a radius of about 50 nm). The first breakdown experiments were conducted in a SEM where the depth profile of the locally molten Cu surface was determined by cross sectional FIB milling and subsequent SEM observation. The customisable diameter of the high voltage tip as well as the demonstrated modification of the topography of sample by the FIB together an accuracy of the needle positioning in the several ten nanometers range enable the local characterisation of breakdown phenomena. These measurements of the dependence of field emission on surface structure in accelerator materials at the sub-um scale are essential for understanding the mechanism of electric discharge in high power accelerators. Such set-up makes scanning electron microscopy and focused ion beam methods a useful tool in the understanding and development of surface structures for future accelerators.

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Figure1aGrey

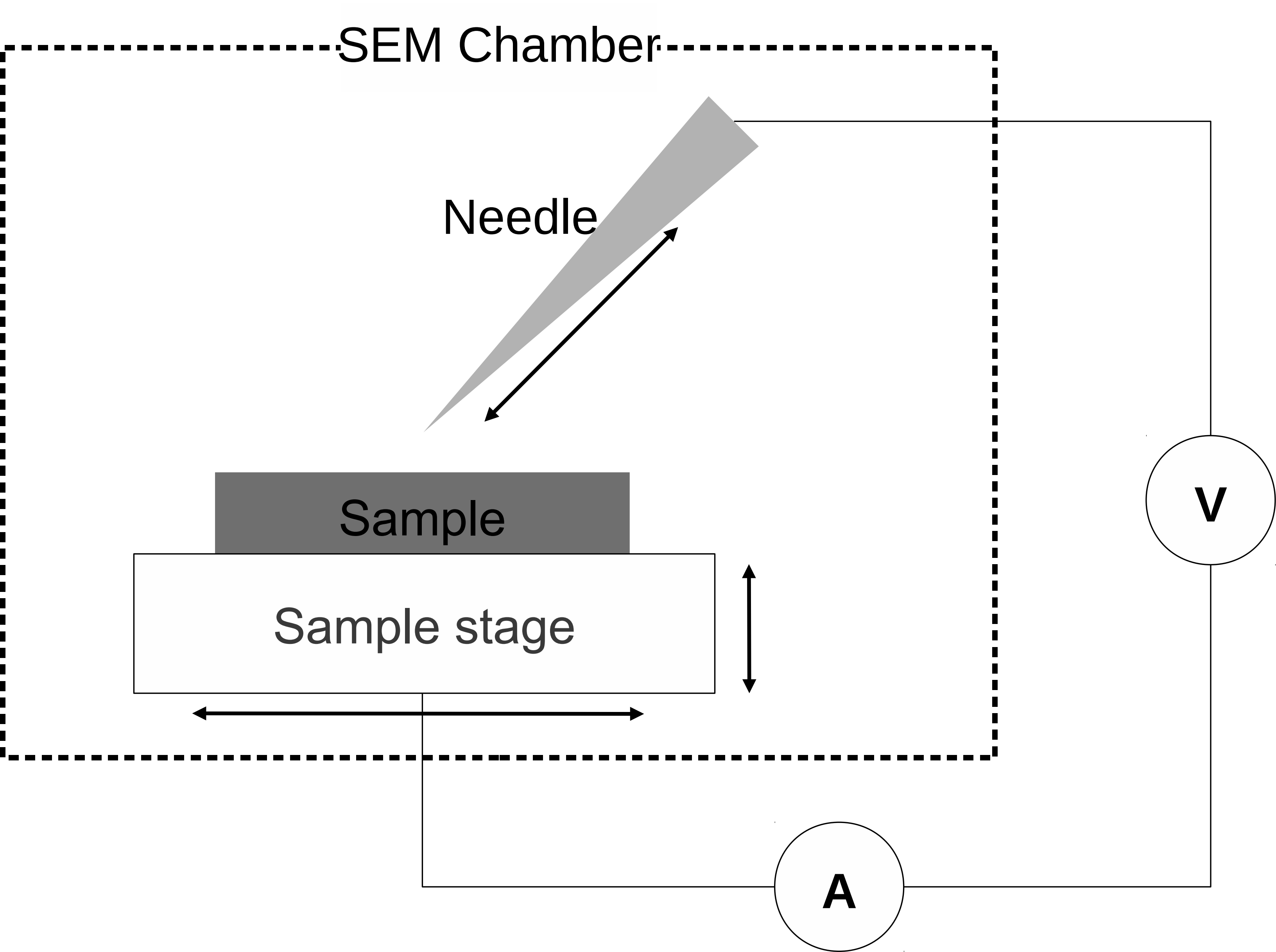

Figure1bGrey