NOVEL FIELD EMISSION SCANNER FOR SURFACE STUDY OF NIOBIUM SRF CAVITY

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Abstract

Field emission strongly depends on Nb surface properties which are determined by its surface treatment and handling. Novel field emission scanner (FES) developed allows us to measure a distribution of the field emitting sites over a sample surface at a given field strength along with its SEM observation and energy dispersive x-ray analysis. A compact FES head driven by precise 3D stepping motors can be located in a space between the SEM object lens and a sample to be observed. For this reason, the scanner head is not required to be retracted for SEM-EDX observation. This design enabled us to have unique features of in-situ mapping of field emitters with a resolution of 250 nm together with SEM and EDX observation with an imaging resolution of 1.5 nm, vibration-free scanning, quite easy position matching between FES images and SEM images and easy cooperation of the two equipments. Preliminary result of the electropolished Nb surface with a plenty of segregated sulfate particles showed basic performance of this FES. It tuned out that the existence of insulating sulfate grown at Nb surface in electropolishing process would cause blasting of sulfate to deteriorate cavity performance.

INTRODUCTION

It is mandatory to investigate field emission on Nb SRF cavity systematically since strong field emission often limits the cavity performance. The emission intensity and the number of emission sites strongly depend on Nb surface properties which are determined by its surface treatment and handling. One of good tools to reveal field emitters on the surface is a so-called field emission scanning microscope. A pioneer work of the field emission scanning microscope was done by E. Mahner et al.[1] and some remarkable works were carried out at Jefferson Laboratory [2] and University of Wuppertal[3, 4]. We have developed a similar equipment named as a field emission scanner (FES). However there is a distinct difference in concept. The basic concept of our FES is to install it into an existing SEM-EDX (Scanning Electron Microscope - Energy Dispersive X-ray Spectroscopy). This will allow us to measure a distribution of field emitting sites over a sample surface along with in-situ SEM-EDX observation in the same specimen chamber. This article describes development of the FES and its preliminary results of the application to niobium samples.

COMBINED SYSTEM OF FES AND SEM-EDX

Our FES consists of an anode needle driven by small and precise 3D stepping motors and an eucentric SEM sample stage. A compact scanner can be located into a space between the object lens and a sample to be observed. Hence retraction of the scanner head is not needed for SEM-EDX observation and operation switching between FES and SEM can be done in seconds keeping a SEM working distance. One of the most important point for this kind of scanning microscope is to keep a tip-surface distance constant over the surface because the sample might be macroscopically tilted. This means the scanned plain by the tip should be parallel to the sample surface. For this purpose, three steppers for linear motion to control the scanning tip in X, Y and Z axes and two steppers for tilt motion to control the tip in rotation around X and Y axes were installed on the SEM sample stage. These motions are illustrated in Fig. 1 and a schematic view is shown in Fig. 2.
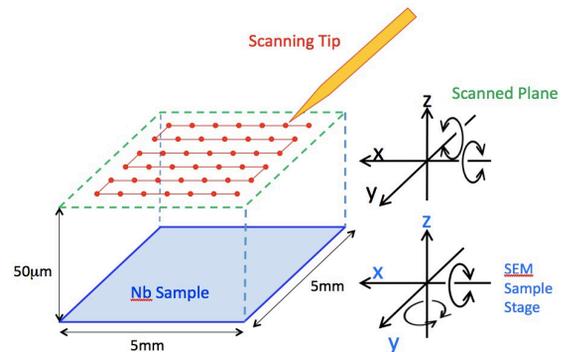
Figure 1: Three steppers for linear motion and two steppers for tilt motion were installed on a SEM sample stage with five axes.
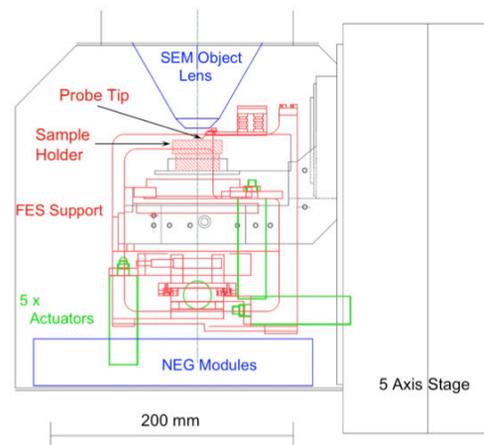
Figure 2: Cross sectional schematic view of the SEM stage and FES.

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It is obvious that the FES support is decoupled from the SEM room floor as the whole FES was installed into the SEM body. Heating capability of a sample coupon were prepared during scanning to mainly simulate a baking process of a SRF cavity. Several NEG cartridges and NEG modules were installed to enhance the pumping speed at the specimen chamber. In addition, the whole system was newly equipped with a sample load-lock system for existing UHV suitcases. Therefore a sample coupon to be observed is hardly exposed to air, contaminants and dust particles during the transportation from a clean room in an electropolish (EP) facility for example.

These ideas bring us to have unique features of in-situ mapping of field emitters with a resolution of 250 nm together with SEM and EDX observation with an imaging resolution of 1.5 nm, vibration-free scanning, quite easy position matching between FES images and SEM images and easy operation of the two equipments. Table 1 shows the detailed specifications and a side view of the FES and SEM stage including the object lens is shown in Fig. 3.

Table 1: KEK FES Specifications

Tip Motion in X, Y, Z	± 10mm
Step Resolution	250nm (40k steps)
Motion Speed	20mm/36s @max
Tilt of Scanned Plane	$\pm 3°$
Tip Voltage	<15kV <5kV/20μm=250MV/m
Measurable Field Emission Current	> 0.1pA
Probe Tip	Positively Biased Tungsten
Sample Size	$< \phi 8$
Raster Scan Area	<5mm x 5mm
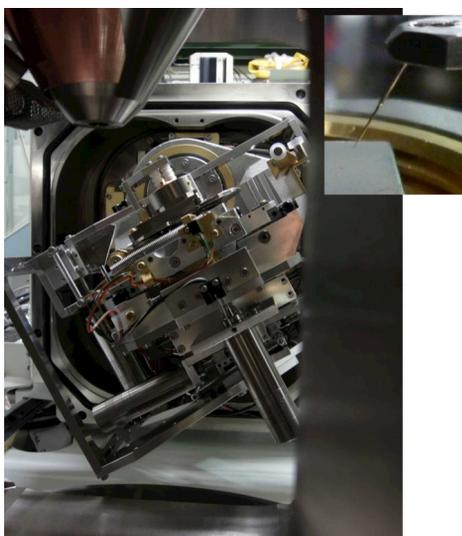
Figure 3: Side view of the FES and SEM stage including the SEM object lens. The inset shows a tungsten tip just above the sample.

RESULTS AND DISCUSSIONS

Nb sample coupons were EPed with a high current density (50 mA/cm^2) and an aged acid (7.6 g/l) at a laboratory EP facility in KEK. Under these conditions, the EP generated a large quantity of sulfate particles which were confirmed with XPS (X-ray Photoelectron Spectroscopy) and SEM-EDX techniques (see more detail in a presentation of MOPC092). These sulfate petals seem to be grown product as a result of surface segregation of sulfate. Fig. 4 (a) and (b) show SEM images of those petals in different imaging scales. It was found that these petal crystals gradually disappeared, viz, evaporated due to the SEM primary electron beam when the observation lasted for 5 to 10 minutes.
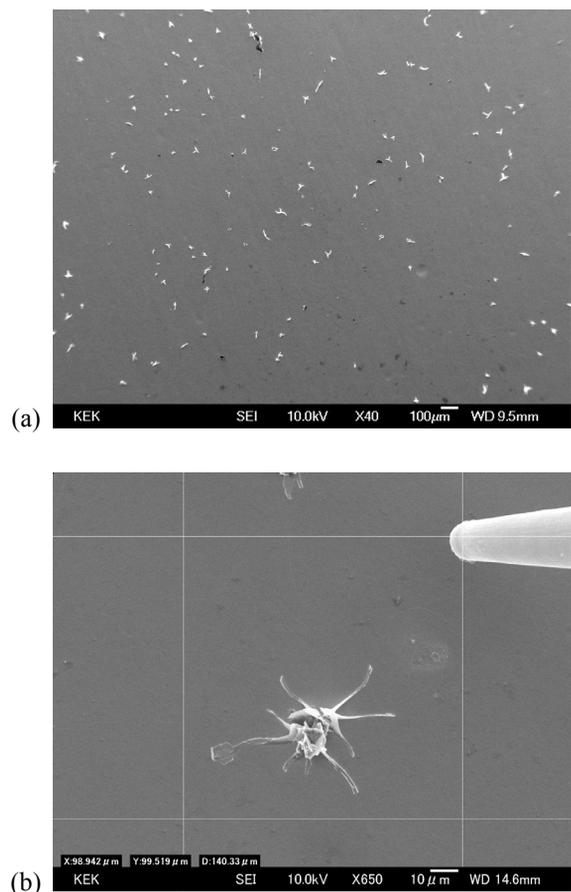
Figure 4: (a) SEM image of petals on Nb surface EPed with high current density (b) SEM image showing one petal, the scanning tip and the scanned area of 100x100 μm^2 with 4 lines.

A preliminary FES image where the scan was carried out over the same size as Fig. 4(b) (100x100 μm^2) including one petal is shown in Fig. 5. The square area enclosed with 4 lines in Fig. 4(b) is the exact area scanned with the FES. The tip-surface distance and the voltage between these were set to be 20 μm and 3.5 kV, respectively. The scan step of 2 μm for this area made a

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total number of measured pixels to be around 2500. The position of X=0, Y=0 in Fig. 5 is corresponding to a standby position of the probe tip in Fig. 4(b).
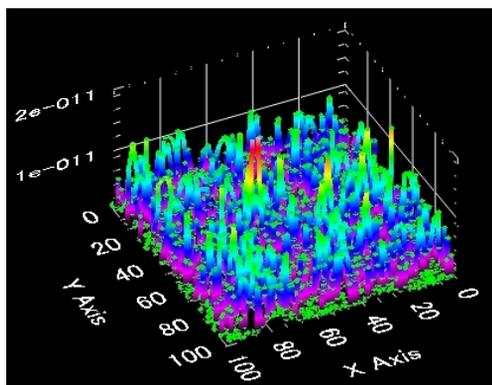
Figure 5: FES emission current mapping over the same size as Fig. 4(b) (100x100 μm^2) including a petal. The units for the vertical axis and for the X, Y axes are ampere and μm, respectively

There were some small current peaks in a range of 1-10 pA in Fig. 5 though surface with no current peaks is obviously ideal and unfortunately the scanning did not reveal the petal itself on the FES image. As a remark it should be emphasized here that in contrast with Nb surface, our CNT field emission arrays (FEA) [5] which was tested as another extreme of an electron emitter showed a reasonable mapping of FEAs and a current in the order of 10 μA with a voltage of around 1 kV at the same tip-surface distance. The result shown in Fig. 5 suggests the most of the petal consists of insulator. The suggestion is supported by the fact that bright imaging parts in a SEM image are generally parts which have high secondary electron yields. This means most of those parts on the surface consist of insulator like oxide or hydroxide. However the small peaks of the emission current might be interestingly corresponding to both the rooted sulfate part at the petal feet that would be semiconductive and the small, still conductive sulfate with a size of 10-100 nm. The small sulfate were visible in a highly magnified SEM image and are considered to be the initial growth stage of sulfate petals.

As shown in a SEM image (Fig. 6) after the FES mapping, it was observed that strong blast of the petal occurred during the scanning and the main part of this insulating emitter disappeared. There remained some roots of the blasted petal in the Nb sub-surface, the fragments and the round melted particles. This is probably due to a quite short discharge that was certainly unable to be recorded with a sampling of 50 Hz of the current meter. This microscopic phenomenon would be the exact phenomenon occurred when the Nb SRF cavities with sulfate on the surface are processed. If a remarkable quantity of insulating sulfate with strong adhesion to Nb surface remains on the surface, cavity performance would

be obviously limited due to this kind of sulfate blasting since high pressure rinsing after EP may be unable to remove those crystals.
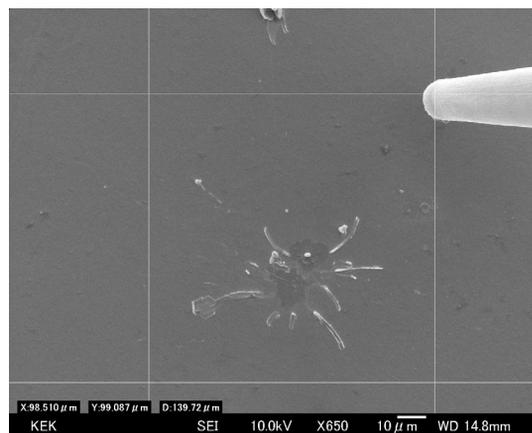
Figure 6: SEM image after the FES mapping. The petal was blasted over. The round melted fragments and the remaining petal roots can be observed.

CONCLUSION

The novel FES was designed and built, featuring in-situ mapping capability of field emitters with a resolution of 250 nm over 5x5 mm^2 together with SEM and EDX observation, vibration-free scanning, quite easy position matching between FES images and SEM images and easy cooperation of the two equipments. Preliminary result of the electropolished Nb surface with the high current density showed basic performance of the combined system of FES and SEM-EDX, revealing quite interesting phenomena of segregated sulfate particles on the surface under a high electric field. It can be pointed out that the sulfate petal mainly consists of insulator and existence of the insulating sulfate grown at Nb surface would cause blasting of sulfate to deteriorate cavity performance. It was also found that small current peaks in the FES mapping would be corresponding to conductive parts like the sulfate at the roots and the sulfate with a quite small size before it grows up as a petal.

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