STUDY ON NIOBIUM SCRATCH AND TANTALUM OR CARBONACEOUS CONTAMINANT AT NIOBIUM SURFACE WITH FIELD EMISSION SCANNER

S. Kato [#], H. Hayano, T. Kubo, T. Noguchi, T. Saeki, M. Sawabe, KEK, Tsukuba, Japan

Abstract

It is mandatory to investigate field emission from niobium SRF cavity surface systematically since even small field emission often limits the cavity performance terribly. In this study typical three cases where field emission is problematic are investigated, that is, niobium scratches at the surface, tantalum contaminant and carbonaceous contaminant at niobium surface. A KEK field emission scanner (FES) allows us to measure a distribution of the field emitting sites over a sample surface at a given field strength along with its SEM (scanning electron microscope) observation and EDX (energy dispersive x-ray) analysis [1]. This article describes results of the FES-SEM-EDX application to those defects at niobium surface.

INTRODUCTION

The field emission has been much improved by careful handling in the material preparation, machining, welding, polishing and so on. However, this issue is still a big concern, specially in mass production process. There was no good tool to find and quantify the field emitters, strongly depending on both their surface topography and surface atomic composition. A field emission scanner (FES) was newly developed in KEK and was firstly applied to measure a distribution of the field emitting sites over a sulphur segregated EPed Nb surface along with its SEM observation and energy dispersive x-ray analysis [2]. A couple of so-called field emission scanning microscopes are reported [2-5]. Our FES features a combined system with an existing UHV SEM-EDX (Scanning Electron Microscope - Energy Dispersive X-ray Spectroscopy). The field emission intensity and the number of emission sites strongly depend on Nb surface properties and some contamination on the Nb surface. which are determined by its surface treatment and handling.

Typical defective surfaces are of surfaces with scratches, pits and bumps, dust particles, and some residues. This time the FES was applied to three different problematic surfaces that is, tantalum contaminated niobium surface, well-known scratched surface and surface with carbonaceous contaminant. Carbonaceous contamination is a new issue [6]. This contaminant including mainly both carbon and oxygen and sporadically nitrogen was often found to be segregated on BCPed or EPed surface with a size of several μm to several tens of μm [6]. There is a strong doubt that this carbonaceous contamination causes additional field emission from the surface. This article describes investigation of field emission from those surfaces with the FES.

[#]shigeki.kato@kek.jp

FES-SEM-EDX SYSTEM

Since the detail of the FES was described elsewhere [1], the brief explanation is given here. The FES consists of an anode needle driven by small and precise stepping motors and an eucentric SEM sample stage.

Keeping a tip-surface distance constant over the scanned surface is very important 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 used. These motions are illustrated in Fig. 1.

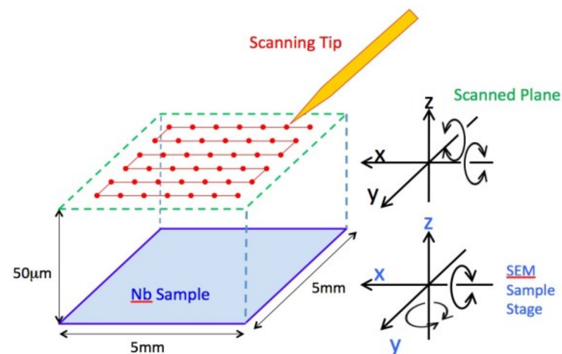

Figure 1: Three steppers for linear motion and two steppers for tilt motion were installed on a SEM sample stage with five axes.

In this work, some additional improvements were made like installation of two differential-transformer type linear position sensors to avoid internal backlash of X and Y steppers. This results in maintaining a position accuracy of about one μm when the backlash causes irregular movement of the anode tip during scanning.

This FES enables us in-situ FES mapping and SEM-EDX observation, vibration-free scanning, no need of sample transfer between two modes of the mapping and the observation and easy switching of the FES and SEM-EDX operation modes keeping the same SEM working distance. Fig. 2 and Fig. 3 show the schematic view and a top view of the FES and SEM stage respectively.

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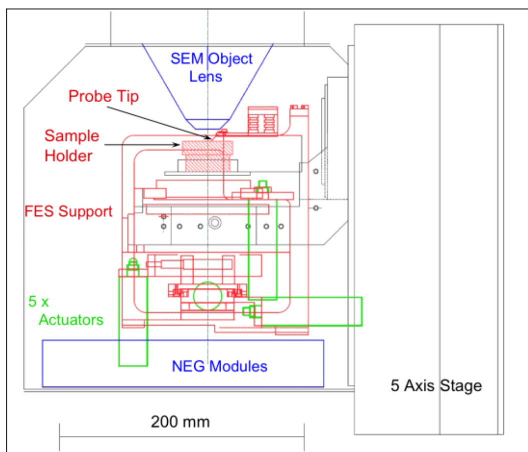


Figure 2: Cross sectional schematic view of the SEM stage and FES. Two position sensors for the X and Y steppers are additionally installed beside of the steppers.

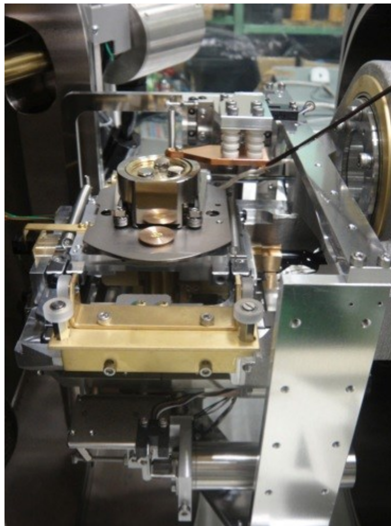


Figure 3: Side view of the FES and SEM stage. Three disk samples are seen in the center.

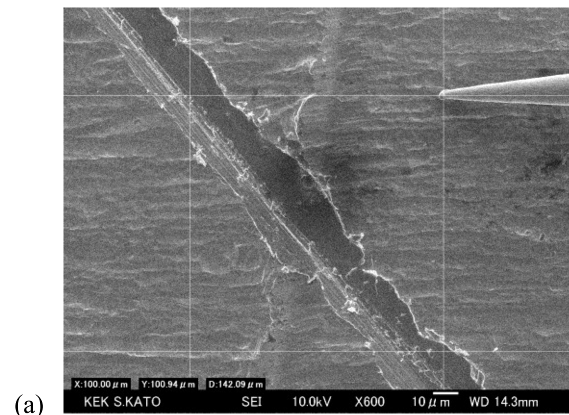
RESULTS AND DISCUSSIONS

Nb samples for niobium scratches and tantalum contaminant were as-received from Tokyo Denkai Co. Ltd. and Nb samples for carbonaceous contaminant were BCPed in KEK. The scratch and tantalum contaminant on Nb surfaces were artificially made with a diamond tip and spot-welding of a tantalum film, respectively. Carbonaceous contaminant on Nb surface was simply prepared with normal BCP.

Scratches at Niobium Surface

A SEM image of a scratch on Nb surface is shown in Fig. 4(a). The square with lines in the SEM image represents a scanned area of 100x100 μm^2 with the scanner tip on the corner. A contact current measurement with the tip touching on the Nb surface allows us to find the peak in this area and measure the height. A peak height of 1 μm above the Nb flat level was measured.

(a)



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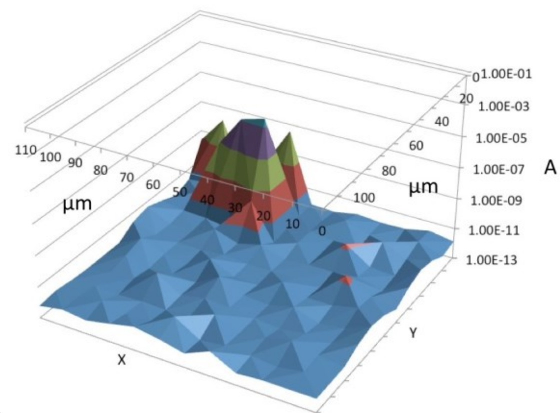

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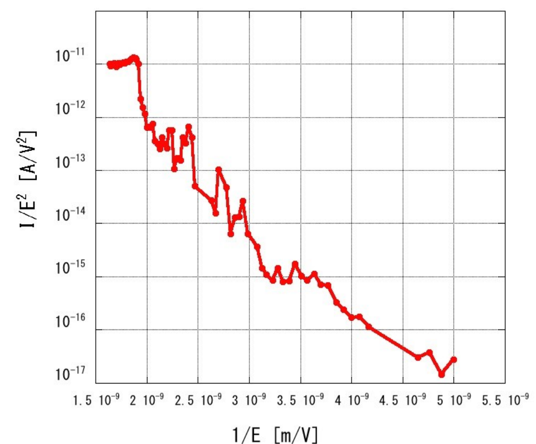

Figure 4: (a) SEM image of a scratch on Nb with the scanner tip. The square shows scanned area of 100x100 μm^2. (b) FES map of a field emission current over the square. (c) Fowler-Nordheim plot at the peak of the FES

Subsequently the tip scanning with a measurement pitch of 10 μm and a gap distance of 2 μm between the tip and the plain was carried out to obtain a field emission current over the square as shown in Fig. 4(b). The data acquisition time for each data point is 30 s and the point to point traveling time is 30 ms. A peak current of 5 μA was measured with an electric field of 600 MV/m. An

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obtained Fowler-Nordheim plot at the peak of the FES map was shown in Fig. 4(c) resulting a field enhancement factor of 13 with an assumption of a work function of 4.3 eV of clean polycrystalline Nb.map.

Tantalum Contamination

Ta contaminated Nb surface was also observed and measured with the SEM-EDX-FES. Fig. 5(a) and (b) show a SEM image and an EDX spectrum of the flake which is welded on Nb surface. The round flake was confirmed to be Ta. Then an area of 100x100 μm^2 was scanned under the same conditions with a case of scratched Nb surface. The measured peak height of this Ta flake above the Nb flat level was 57 μm. FES map of the Ta contaminated Nb surface is shown in Fig. 5(c). Relatively large peak currents of a couple of 10 μA were measured with an electric field of about 300MV/m at three positions. A Fowler-Nordheim plot was measured at a position close to those three high current peaks in the map. A field enhancement factor of 8.2 was obtained with an assumption of a work function of 4.25 eV of clean polycrystalline Ta. The map does not seem to represent the SEM image. This might be mainly due to oxide and/or alloy formation because of the welding causing a different work function from the work function of the clean surface.

Carbonaceous Contamination

A typical carbonaceous contaminant found after BCP at Nb surface is shown in Fig. 6. EDX measurement showed that contaminant consists of mainly both carbon and oxygen and sporadically nitrogen. Its size ranges from several μm to several hundreds of μm. The SEM image implies it is surface of relatively low secondary electron yield. From additional SEM observation, it also turned out that carbonaceous contamination shows characteristics of not perfectly electrically conductive, volatile or non heat-resistive and elastic material as it gradually disappears and slowly moves under the electron beam irradiation. Fig. 7 shows a zoomed SEM image in the image of Fig. 6. Searching the highest peak in this area with FES, a height of 30 μm was found in the center from the Nb plain level. The electrical resistivity of the carbonaceous contaminant was roughly measured in the center with two terminal contact method where the tip contacted with the contaminant and was applied with a DC voltage of 15 V. The obtained resistivity was measured to be around 3x10^7 Ωm assuming a contact area

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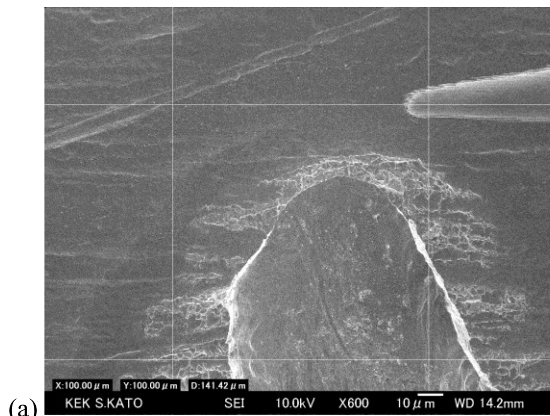

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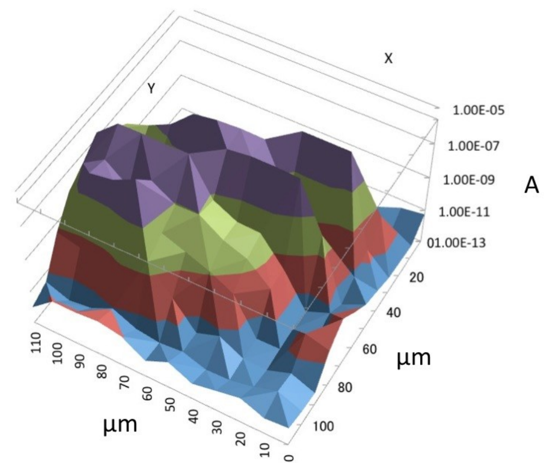

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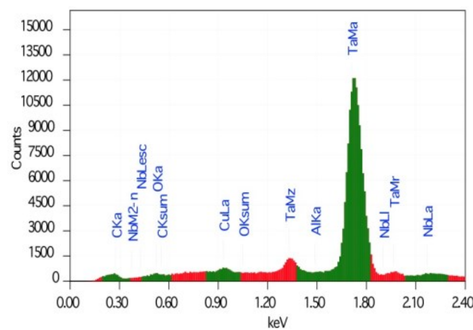

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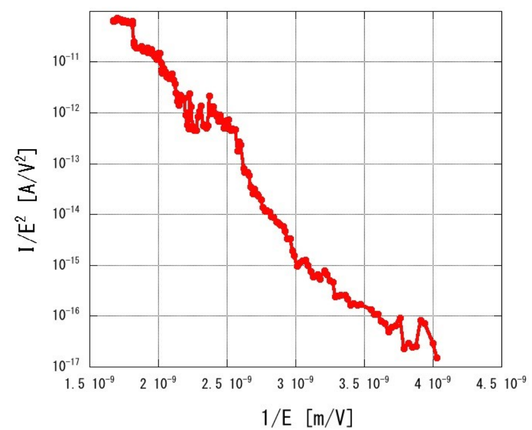

Figure 5: (a) SEM image of a contaminant on Nb with the scanner tip. The square shows scanned area of 100x100 μm^2. (b) EDX spectrum at the flake. (c) FES map of a field emission current over the square. (d) Fowler-Nordheim plot at a position among the three current peaks of the FES map.

ISBN 978-3-95450-143-4

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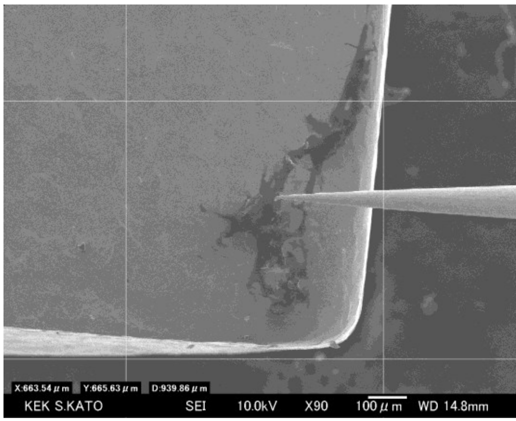


Figure 6: SEM image of typical carbonaceous contaminant found after BCP at Nb surface with a low magnification.

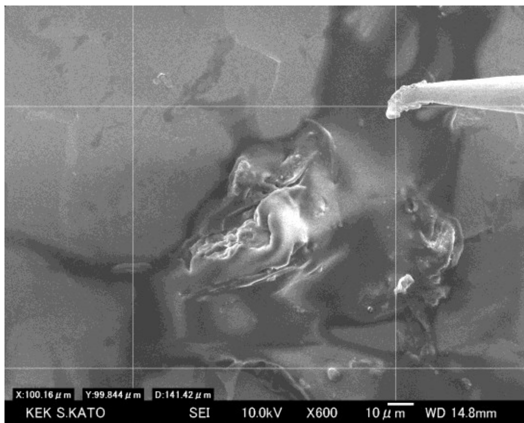
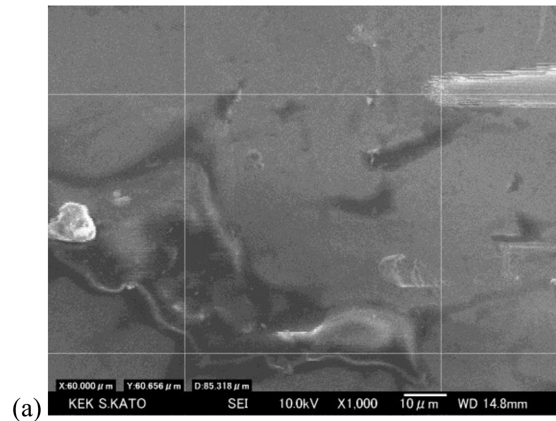


Figure 7: Zoomed SEM image of the image of Fig. 6. The resistivity of the carbonaceous contaminant was measured in the center.

of 100 μm^2. After this measurement, the tip was inserted into the contaminant and moved along with the surface keeping the same gap distance causing back-and-forth movement of the contaminant under the SEM observation. As partially mentioned above, it was found that the contaminant is some sort of elastic material that seems to be softer than rubber but harder than jelly and is not sticky with the tungsten tip. Furthermore the contaminant was never split during the movement.

FES mapping of 60x60 μm^2 with a gap distance of 8 µm between the tip and the plain at another zoomed area (Fig. 8(a)) in the image of Fig. 6 was attempted. A peak height of 6 µm above the Nb flat level was measured in advance. The other measurement conditions are the same as the previous. There was no field emission up to an applied electric field of 600 MV/m when the mapping was repeatedly carried out. However at a peak field of 700MV/m, unexpected discharge occurred during the mapping. Fig. 8(b) shows a SEM image after the discharge. It was observed that a bottom left part of the square was remarkably blasted and the anode tip apex seemed to be covered with some fragments from the carbonaceous contaminant.



(a)

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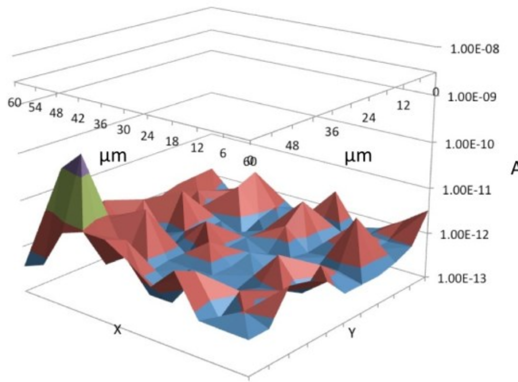

(c)

Figure 8: (a) SEM image of carbonaceous contaminant on Nb surface with the scanner tip. The square shows scanned area of 60x60 μm^2. (b) SEM image of the same view field after a discharge. (c) FES map of a field emission current over the square.

A new FES map after the blast was acquired under the same conditions as shown in Fig. 8(c). A new current peak of field emission has appeared. This would be because of emission from the covered emitter with the carbonaceous contaminant or emission from a new emitter generated by the blast. The blasting may also make additional cause of discharge since distinct quantity

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of contaminant fragments will scatter over cavity surface if this happens in a SRF cavity.

CONCLUSION

FES can provide information not only of field emission of contaminants at Nb surface but also of its mechanical and electrical properties. Three different cases of scratched Nb surface, tantalum contaminant on Nb surface and carbonaceous contaminant on Nb surface are studied with FES. For scratched Nb surface and tantalum contaminant, FES maps, heights of contaminant, field enhancement factors were obtained. Qualitative mechanical characteristics of carbonaceous contaminant and its electrical resistivity were also revealed by FES. Carbonaceous contamination that is a new issue against Nb cavity performance was also investigate with FES-SEM-EDX. EDX showed this contaminant mainly consists of carbon and oxygen while nitrogen and fluorine was also found in some cases. From SEM observation it was found the carbonaceous contaminant showed characteristics of poorly conductive, non heat-resistive and elastic material. From FES, some mechanical property and electrical conductivity of the contaminant was obtained though the mapping was often disturbed the unexpected discharge.

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ISBN 978-3-95450-143-4