Field emission measurements on niobium cathodes of high purity

E. Mahner, R. Heiderhoff, N. Minatti, G. Müller, H. Piel, N. Pupeter
Bergische Universität-Gesamthochschule Wuppertal, Germany

ABSTRACT
In this paper we present a microscope to study field emission sites on metallic cathodes. A description and a first test of our UHV field emission scanning microscope (FESM) with high resolution provided by a scanning tunneling microscope (STM) is given. With this apparatus systematic field emission measurements from broad-area niobium cathodes of high purity are planned.

1. INTRODUCTION

Enhanced field emission (EFE) is the electron emission that occurs when an electrical field of typical 10-50 MV/m is applied to a large metallic cathode in a vacuum system. This behaviour can often be described by the Fowler-Nordheim law with a field enhancement factor β and an apparent emitting surface S. Typical values are 100-500 for β and 10^{-12}- $10^{-7}cm^2$ for S. The emission originates at local sites which are often particles or metallic inclusions on the surface. At the University of Geneva Ph. Niedermann and Ø. Fischer studied the properties of field emission (FE) sites on flat niobium cathodes [1,2]. They found that often particles of micron or submicron size are associated with the emission process. The emitters show a big variety of elemental composition and can be greatly reduced by heat treatment (HT) above 1200 °C [3]. With HT emission-free niobium surfaces up to 200 MV/m were produced. Nevertheless, the physical mechanism which is responsible for EFE is still unknown. In the last years several groups have shown that enhanced field emission is the limiting factor to reach higher electrical field gradients in rf superconducting accelerator cavities [4]. Because of its technological importance for accelerators like LEP or TESLA [5], we want to study EFE on niobium cathodes of high purity (RRR→1500) and niobium single crystals of centimeter size. Therefore, we designed and built a FESM and a STM for high spatial resolution FE scans.

2. FIELDEMISSION SCANNING MICROSCOPE

The field emission measurements are done in a standard VG Escalab system which is pumped by turbomolecular and Ti sublimation pumps to a base pressure of $3*10^{-11}$ mbar. A scanning electron microscope with a LaB_6 filament and 700 Å resolution is included (Fig.1). Our Escalab also contains a preparation chamber with a base pressure of $1*10^{-9}$ mbar and a fast entry airlock. A flexible sample transport system allows the transfer to the analysis chamber under UHV condition (Fig.2).

SRF91F08

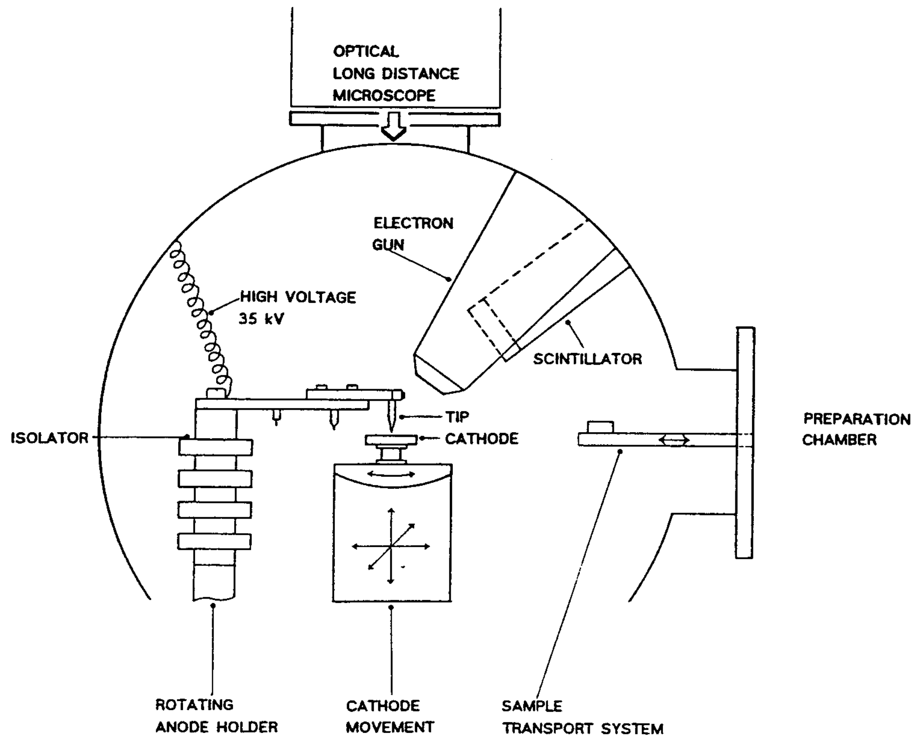

Figure 1: The Escalab chamber with field emission scanning microscope (FESM), scanning electron microscope (SEM), and long distance microscope (LDM) for optical control.

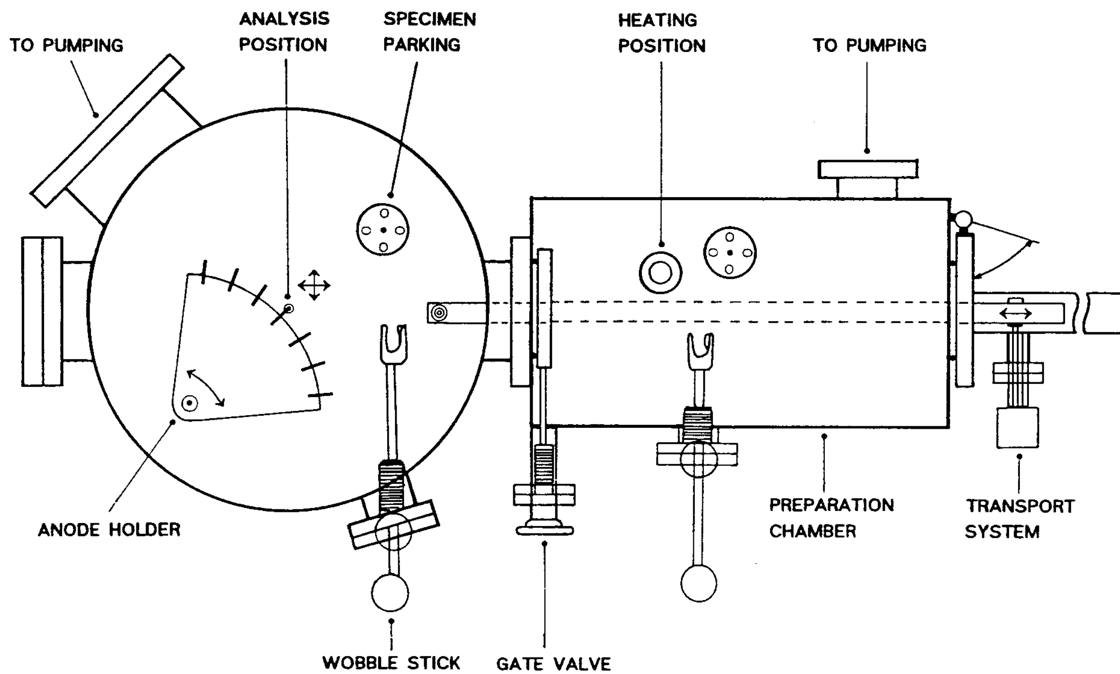

Figure 2: The analysis and preparation chamber with sample transport system and fast entry airlock.

In the preparation chamber samples can be heated up to 2000 °C by electron bombardment. For FE measurements an UHV field emission scanning microscope (FESM), which is similar to the Niedermann's type, was built. Inside the vacuum 3 linear tables with 25 mm range perform the x, y and z motions. These tables consist of a stainless steel body, austenitic stainless steel guiding surfaces and tungsten carbide (WC) rollers without lubrication. Two special levers (stainless steel, WC shaft, titanium nitride (TiN) covered rollers), actuated via linear motion feedthroughs, perform the x and y motions. The z table is directly driven by a feedthrough. In order to make automatic raster scans, all three motions are driven by microprocessor controlled stepper motors which permit single step movement of the sample in µm size. The resolution and reproducibility of the displacement will be measured with a SEM calibration grid.

The sample holder is mounted on a copper disc with a spheric diameter which is supported by a titanium counterpart. The specimen holder assembly can be fixed in the microscope by driving to a lower z position. Movements in x and y directions let this upper part of the microscope slide on a spherical surface. In this way the sample can be tilted in any horizontal axis by a maximum of 5 degrees. With this tilt mechanism it is possible to align the specimen surface parallel to the plane of the x-y-movement.

Seven anodes with different tip diameters for high and low resolution FE scans are mounted at a rotatable titanium-holder supported by a high voltage isolator. The micromanipulator is fixed on a CF 200-flange that can be tilted with respect to the analysis point of the vacuum chamber, in order to align the microscope with the electron gun of the SEM. In the design of the FESM any lubrication, especially MoS_2, was avoided. Special care was taken in the choice of material for the manipulator, mainly stainless steel, titanium, copper, bronze, gold, TiN and WC was used. A rigid construction of the anode holder and all mechanical parts minimizes vibrations of the tip and the sample.

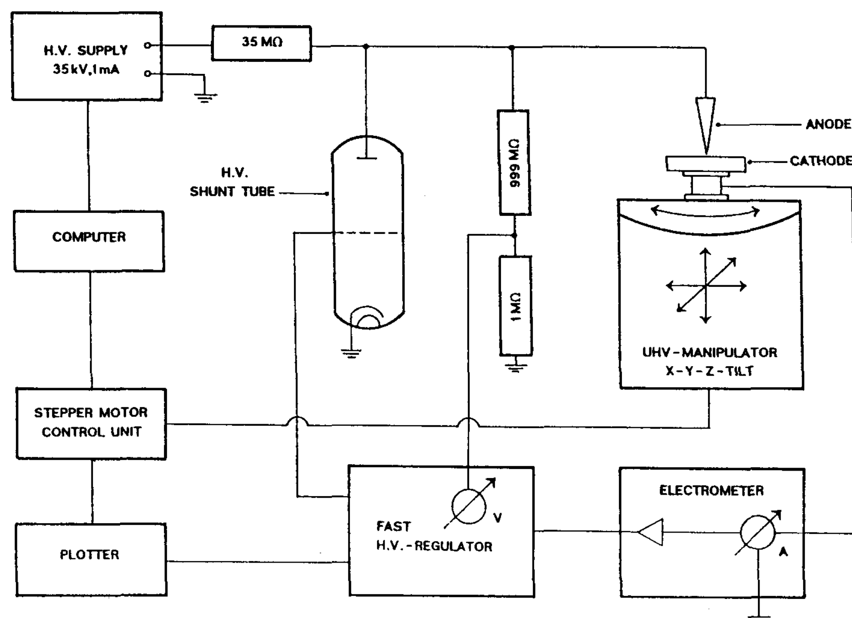

Figure 3: The high voltage regulation circuit which is used to keep either the field emission current or the high voltage constant. The main components are the H.V. supply, fast H.V. regulator, 35 kV shunt tube and the electrometer.

For the FE measurements we designed a special electronic circuit (Fig.3). The anode voltage can be set between any value U_O and the maximum of 35 kV. During a scan the fast high voltage regulator and a shunt tube holds the FE current I at a constant chosen value I_O of typical 10 nA. If a field emitter is moved under the anode, the tunnel current measured by the electrometer increases. In that case the regulator reduces the anode voltage U_O to a value U until $I=I_O$ is reached. The resulting $\Delta U=U_O-U$ and I_O characterizes the FE strength. The FE current remains constant with an accuracy of about 1 %. The regulation time lies in the order of 1 ms.

3. EXPERIMENTAL

The first step of a FE experiment is the tilt of the specimen. Therefore we use an electrolytically etched tungsten microtip which we observe with the SEM. By switching off the collector bias of the secondary electron detector, we see the shadow of the tip on the niobium surface. This procedure is done at four different points of the sample. Until now we reached an accuracy of about 5-10 µm in z-positioning. The gap between tip and sample is controlled by an optical long distance microscope which has a resolution of 1.8 µm, at a working distance of 23 cm. Another possibility is to center the anode above a FE site by measuring the maximum of the tunnel current. Then we reduce the gap while the high voltage regulator keeps the FE current constant at a chosen value (typical 1 nA). In most of the experiments the resulting voltage versus cathode-z-position is linear (Fig.4). By extrapolating to zero voltage we determine the gap. Both gap-measurements yield to corresponding results. To calculate β and S of a field emitter, Fowler-Nordheim plots can be made by measuring the I-V characteristic at a fixed gap.

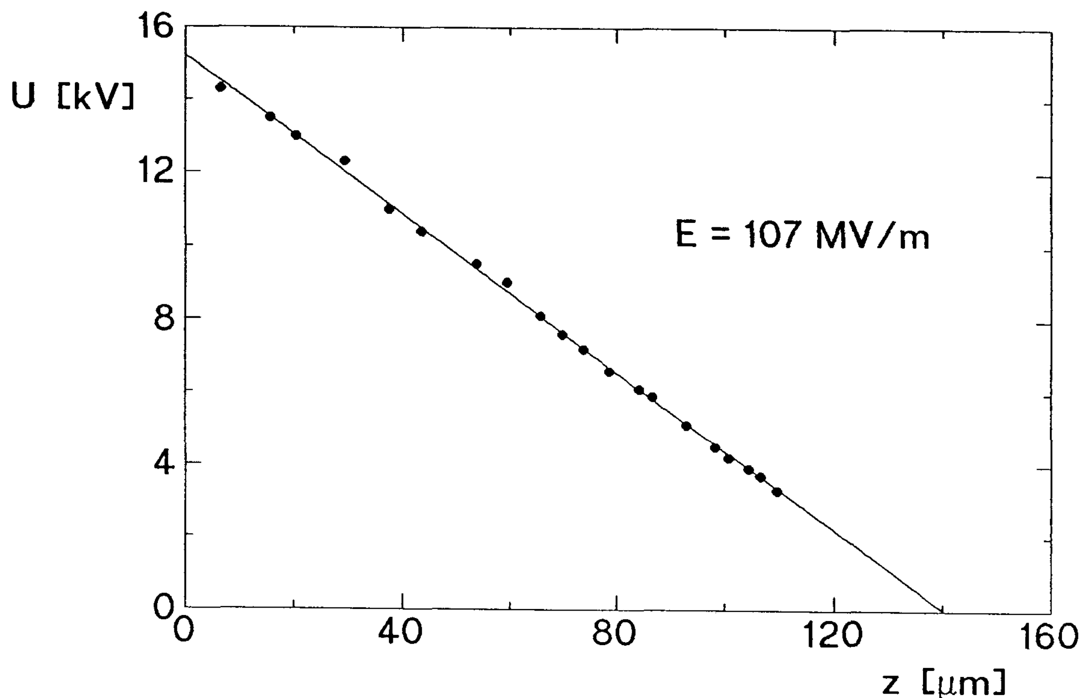

Figure 4: A typical plot of voltage versus z position of the cathode. The measurement was performed with a 0.5 mm diameter tungsten anode at 1 nA and 107 MV/m.

4. FIRST RESULTS

In order to test the manipulator, the high voltage regulator and the self-made heating station in the preparation chamber, we made FE scans on an electropolished niobium test sample with 16 mm diameter and RRR=800 [6]. The scans were done with a 0.3 mm diameter tungsten anode at a gap of 140±7 µm. We applied a high voltage to the tip and moved the sample in a chequered pattern. All measurements were performed in a constant current mode of 5 nA. As soon as the FE current increases, the voltage is regulated. FE sites on the sample are recognized by two-dimensional peaks. Their height is a measure of the emitter strength. In Fig. 5 the first results of FE scans as a function of applied field and heat treatment are shown. We observe that the density of sites between 57 and 85 MV/m decreases after HT at 820 °C (15 min.) and 1400 °C (10 min.). The sharp peaks in the diagram are associated with a rapid increase of the tunnel current. At this locations we sometimes recognized light emission due to sparks.

With these measurements we proved the correct working of our field emission scanning microscope. Detailed investigations of the FE behaviour of high RRR niobium samples are very promising and will follow.

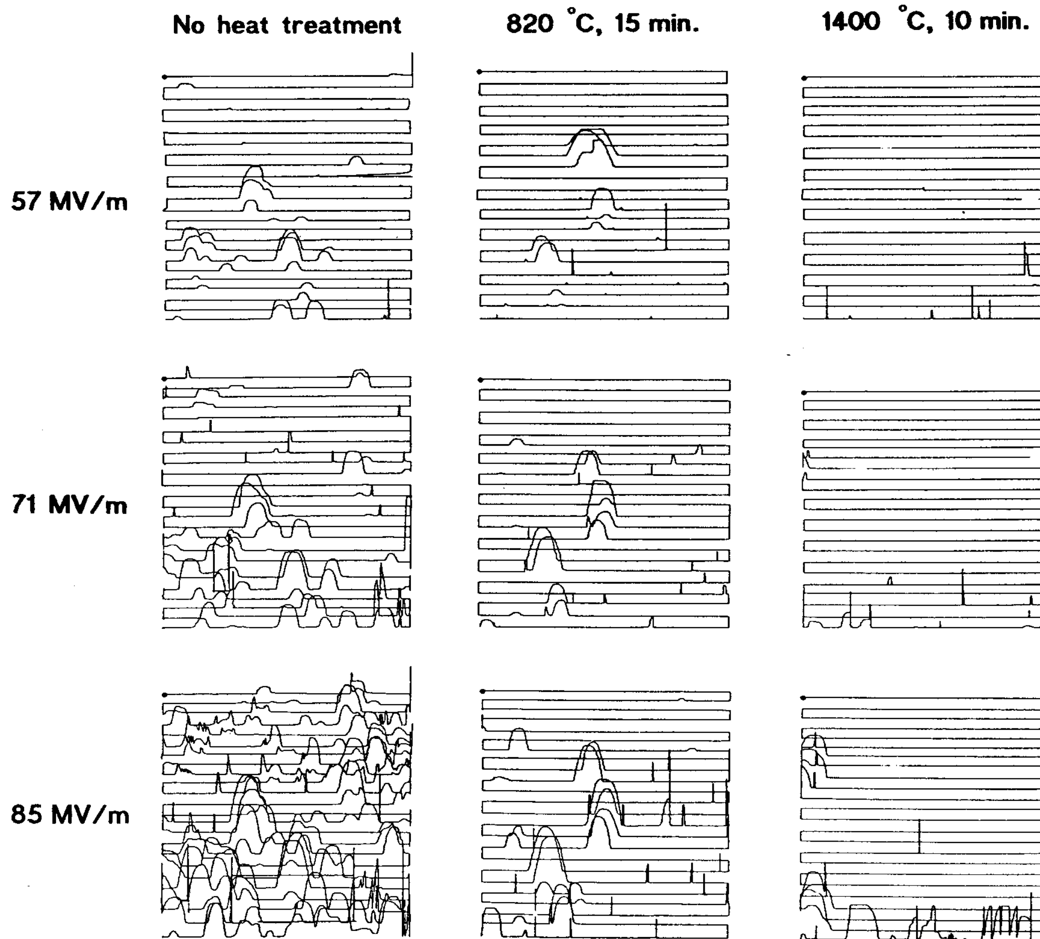

Figure 5: First field emission scans of a high RRR niobium sample as a function of heat treatment. The scans (7.6*7.6 mm) were taken at 5 nA and $p=5*10^{-10}$ mbar.

5. THE SCANNING TUNNELING MICROSCOPE

The construction of our STM is based on the idea of searching for field emitters of submicron size with high spatial resolution in the Escalab chamber. Therefore we built an UHV compatible test device with a large scan area. The main parts of the STM are a conventional "louse", a "piezoelectric car", a single "tube scanner", and a vibration damping system [2,7]. To control the movement of the STM, the used piezo elements are driven by special power supplies.

The louse consists of a macor body and 3 piezoelectric tubes with integrated sapphire balls. This device performs the coarse x-y-movements of the microscope. Until now the stepsize can be varied from 2000 Å up to 2 μm. Horizontal steps of the piezocar either lowers or raises a platform on which the STM is positioned. This can be done in steps of the order of 300 Å. So this car enables a coarse z-positioning of the microscope. An approach of the STM to an arbitrary point of a large specimen surface (16 mm diameter) is possible with these two devices. After zooming to parts of interest under control of the SEM, we want to use the single tube scanner with integrated tungsten microtip. This piezo tube with 20*20 μm scan area and 3 μm lift for the fine z position will be used for high resolution field emission scans. The first measurements will be done with a calibration grid.

6. CONCLUSION

An UHV microscope was built in order to study field emission sites on large niobium cathodes (16 mm diameter) of high purity. We scanned an electropolished niobium test sample, consisting of 2 single crystals, in a constant current mode (5 nA) at $p=5*10^{-10}$ mbar. Inside a scan area of 7.6*7.6 mm we observed that the density of sites between 57 and 85 MV/m was reduced after a heat treatment at 820 °C (15 min.) and 1400 °C (10 min.). For high resolution FE scans a scanning tunneling microscope with a large scan area of 20*20 μm was constructed and tested. This STM is optimized to localize FE sites on a large surface under SEM control. Until now the stepsize of the STM can be varied between 2000 Å and 2 μm. A coarse z-positioning is possible in steps of about 300 Å. In the future we want to study high quality niobium samples (single crystals of large size) with the main focus on the influence of artificial emitters, heat treatment, and grain boundary effects. The STM will be transfered to UHV.

ACKNOWLEDGEMENTS

We gratefully acknowledge the fruitful help of Ph. Niedermann and Ø. Fischer at the beginning of the construction of the apparatus. It is a pleasure to thank L. Sevryukova for the high purity niobium samples. We are grateful to R. Clasen and R. Schüngeler for technical support. This work was funded in part by the German Federal Minister for Research and Technology (BMFT) under the contract number 05 5WT85 I.

[1]: Ph. Niedermann, Ph.D. thesis, University of Geneva, 1986.

[2]: Ph. Niedermann, Ch. Renner, A.D. Kent, Ø. Fischer, Paper presented at 2nd Int. Conf. on Vac. Microelectron., Bath, 1989.

[3]: N. Sankarraman, Ph. Niedermann, R.J. Noer, Ø. Fischer, J. Physics (Paris) 47, C7-133, 1986.

[4]: H. Padamsee, J. Kirchgessner, D. Moffat, R. Noer, D. Rubin, J. Sears, Q.S. Shu, Proceedings of the 4th Workshop on RF Superconductivity, Vol.1, Aug.1989, KEK Japan.

[5]: Proceedings of the 1st TESLA Workshop, July 1990, Cornell University.

[6]: The sample was prepared in the group of L. Sevryukova, IHEP, Protvino, USSR.

[7]: Ph. Niedermann, Ch. Renner, A.D. Kent, Ø. Fischer, J. Vac. Sci. Technol. A, Vol.8 No.1 Jan.-Feb. 1990 and references in it.