# INVESTIGATIONS ON FIELD EMISSION FROM BROAD AREA Nb-CATHODES

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## INTRODUCTION

Fieldemission (FE) from broad area cathodes is known to take place at field levels which are a factor of 100 to 1000 lower than the predicted value for clean, ideal metal surfaces. This emission puts a limit to the operation of high field devices such as superconducting cavities. The physical mechanism of this emission is subject to many speculations and experimental investigations in the literature, but a detailed understanding is still lacking. For recent reviews, see refs. 1 and 2.

The FE at low fields is always found to come from certain emitting sites on the surface, and a few reports exist on localisation of such sites on metal surfaces by various techniques (3-6). No unique feature has been identified with the emitting sites but one has often found foreign particles or microcracks in the area where the emission comes from.

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X-ray microprobe analysis of these features showed in some cases foreign metals at FE sites. Since, however, this method is not sensitive to low-Z elements, it was suggested that oxides are present in reality. From model considerations it was inferred that the size of the emitting structure be of the order of 1000 Å up to microns. More recently, Latham and co-workers were able to deliberately create FE-sites which emitted at very low field levels by depositing carbon particles onto a metal surface (7).

In this paper, we shall describe an experiment where we localise FE-sites and correlate the observed SEM features with elemental concentrations measured in-situ by scanning Auger and micropoint Auger analysis. The emission was found to be frequently related to the presence of a particle containing either Nb or a foreign element (C,Ag,Al). However we also found sites where no particular feature was discernible both with SEM and micro-Auger analysis.

## EXPERIMENTAL

The experiments were done in a commercial Vacuum Generators "ESCALAB" UHV system with a base pressure of $5 \times 10^{-11}$ mbar. The upper part of Fig. 1 schematically shows the arrangement of the electron gun with 0.5 µm beam diameter, the 157° spherical sector electron analyser with its input lens system, the SE detector and an argon ion gun. The lower part shows the purpose-built manipulator capable of cathode x-y-z movement and tilt about two axes. It is also possible to interchange anodes by a rotation and to align both anode and cathode with respect to the electron beam and the analyser axis. The 25 mm x- and y- and the 12 mm z-movements are guided by linear tables which are in the vacuum, and are driven by standard linear motion feedthroughs (via levers with precision slide bearings in the case of the x and y movements). The achieved resolution of the movements is ca. 1 µm. The tilt motions are actuated by the same x and y movements with the sample holder held in position on a moving spherical surface by a

mechanical coupling that acts only at low z positions. The anode holder takes up several anodes that can be brought into the vacuum like the cathodes by the standard VG sample transport system which includes a fast entry airlock.

Samples were 14 mm diameter disks of 99.93 % purity niobium. They were chemically or mechanically polished or electropolished and ultrasonically rinsed in distilled water or ethanol. Once in vacuum, each sample was scanned with a 1 mm dia. flat anode at E $\cong$ 40 MV/m. This usually resulted in the detection of about half a dozen FE sites. The Fowler-Nordheim characteristics of the strongest of these sites were measured. We varied the z position and the voltage while keeping the current constant. This invariably resulted in a linear relationship that was used to determine the electrode separation.

Sites with $\beta > 200$ were selected for further study. The anode was exchanged for a tungsten tip that had been electrolytically etched to micron- size tip radius. After a fine scan, the site was then localized within 2-4μm by gradually reducing the gap down to 7-10 μm. Micro-Auger analysis could then be performed without change of geometry, alternating with sputter ion etching.

## RESULTS

The main results of the 12 stable sites found on 8 Nb samples are listed in table 1.

For convenience, and in order to compare our results with literature, we characterize our sites with the field-enhancement factor $\beta$ and the emitting surface obtained from standard Fowler-Nordheim analysis (8) neglecting, however, the image force correction. The real meaning of these quantities is unclear since the emission is almost certainly not coming from metallic protrusions with a static field enhancement.

Out of the detected sites, four appear as particles clearly visible in the SEM but compositionally not significantly distinguishable from the base metal. Two sites are not accompanied by any irregularity to be seen in our SEM and micro-Auger analysis. Other particles at the emission location consist mainly of aluminium [in 2 cases], silver [1], carbon [2] and possibly oxide of niobium [1].

We will now discuss three typical sites in more detail. Fig. 2a shows the SEM-image of site 3-5 with the probe tip at 10 μm distance. The site coincides with an aluminium particle which probably stems from an accident during spot welding that deposited a number of molten splashes onto the surface. Several of these particles were found to emit strongly. This sample was subsequently taken to a conventional SEM of 1000 Å resolution. In the micrograph obtained (fig. 2b), no sharply peaked feature was seen capable to explain the enhanced FE by a classical protrusion model.

Fig. 3a shows the site 5-1 with $\beta = 237$. The separation from the probe to the emitting feature is 6 μm; the horizontal bars indicate the approximate vertical position of the site on the figure. On the right of fig. 3a, a non-emitting silver particle is seen. After FE-measurements and localisation, the system was vented with nitrogen and baked together with the sample at 150°C for 12 hrs, but $\beta$ changed by less than 20 per cent. Subsequently, roughly 100 Å of material was sputtered off and a point Auger spectrum was taken on the particle (fig. 4b). For comparison, an Auger spectrum on the neighbouring surface was taken as well (fig. 4a). There is a marked increase of the carbon peak on the particle. In order to measure the distribution of the elements, Auger line scans were performed along the bars in fig. 3a. These scans (fig. 3b-d) show strong increase of carbon concentration at the site location and some enhancement of oxygen concentration at the right of the white particle in fig. 3a. Due to an unintentional manipulation, the particle disappeared and after that no field emission could be measured at up to six times the original field. However, the dark spot in the background of fig. 3a emitted at

the fourfold of the original field strength.

An example of a FE-site where no particular feature was visible in our system is shown in fig. 5a (site 3-6). The Auger spectra taken in the region of the site and at a small distance did not show any significant variation in concentration of niobium and its usual surface pollutants, carbon and oxygen. Of this site, fig. 5b shows a higher resolution SEM image showing a rather high number of submicron particles on the surface, one of which might possibly be associated with FE. Fig. 6 shows the Fowler-Nordheim characteristics of the three discussed FE sites.

In summary, the field emission sites localised on a total of 10 $cm^2$ Nb surface area were in majority associated with micron-size particles sitting probably rather loosely on the surface. Their elemental composition is not unique. These particles mostly contain a metal: either Nb or others, sometimes also a high carbon concentration which was persisting to hundreds of angstroms in depth. The almost general white appearance of these particles in the SEM in our opinion does not necessarily mean that they are insulating but this could rather be due to their shape and small size. In a minority of cases no particle was seen down to 1μ resolution and only in one instance the results point to an oxide particle. At present, we are investigating effects of anodizing and high-temperature vacuum firing of Nb on field emission.

## ACKNOWLEDGEMENTS

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TABLE 1

| Sample No | Preparation[1] | Site No | β | S[cm²] | E[MV/m] at 1nA | SEM-feature | Size [µm] | Elements[2] detected |
|---|---|---|---|---|---|---|---|---|
| 1 | CP | 1-1 | 190 | $7 \cdot 10^{-9}$ | 13 | white dot in grain boundary | 2 | Nb/C/O[3] |
| | | | 250 | $2 \cdot 10^{-11}$ | | | | |
| | | | 227 | $1.3 \cdot 10^{-10}$ | | | | |
| 2 | MP | 2-1 | 290 | $2.8 \cdot 10^{-10}$ | 11 | white particle | 4 | Nb/C/O[4] |
| | | 2-2 | 202 | $1.7 \cdot 10^{-11}$ | 16 | white particle | 2x10 | Nb/C/O[4] |
| | | 2-3 | 280 | $2.6 \cdot 10^{-12}$ | 13 | white particle | 7 | Ag/O/C/Nb |
| 3 | CP | 3-2 | 207 | $1.2 \cdot 10^{-11}$ | 16/20 | splashed metal particle | 100 | Al/O |
| | | 3-5 | 251 | $5 \cdot 10^{-11}$ | 13.5 | dark dot with white rim | 3 | Al/O/C/Nb |
| | | 3-6 | 303 | $6 \cdot 10^{-15}$ | 27 | no irregularity | | Nb/O/C[4] |
| 5[5] | CP | 5-1 | 237 | $4 \cdot 10^{-10}$ | 12 | white particle | 7 | C/Nb/O |
| | | 5-2 | 590 | $4 \cdot 10^{-10}$ | 4 | white particle | 3 | Nb/C/O[6] |
| 7 | EP | 7-1 | 206 | $9 \cdot 10^{-10}$ | 11.7 | no irregularity | | Nb/O/C[4] |
| 8 | MP | 8-2 | 190 | $9.4 \cdot 10^{-8}$ | 12.0 | white dot | 2 | C/Nb/O |
| | | 8-3 | 278 | $4.9 \cdot 10^{-10}$ | 10.4 | white dot | 4 | O/Nb/Ag |

Remarks

1) CP = chemically polished, MP = mechanically polished, EP = electropolished
2) elements are listed in the order of decreasing Auger peak-to-peak height
3) slightly enhanced concentration of C and O
4) no variation of composition found compared to the surrounding surface
5) sample 4 (CP) and 6 (CP) did not show any sites with $\beta > 200$
6) enhanced C concentration beside the particle.

## FIGURE CAPTIONS

Fig. 1 experimental apparatus

Fig. 2 SEM-images of site 3-5; (a) with the probe tip; the site is within the encircled region, (b) 1000 Å resolution image.

Fig. 3 site 5-1 shown by (a) SEM-image with lateral position of site determined by the probe tip, vertical position indicated by horizontal bars along which Auger line scans (b-d) were taken

Fig. 4 (a) Auger spectrum in the vicinity of site 5-1, (b) point Auger spectrum of a white portion of the particle in fig. 3a. The 10 keV electron beam had $I \cong 20$ nA.

Fig. 5 SEM images of site 3-6. The position of the site in (b) is indicated by bars outside the micrograph

Fig. 6 FN-plots of sites 3-5 (Δ), 3-6 (⊕) and 5-1 (*,+)

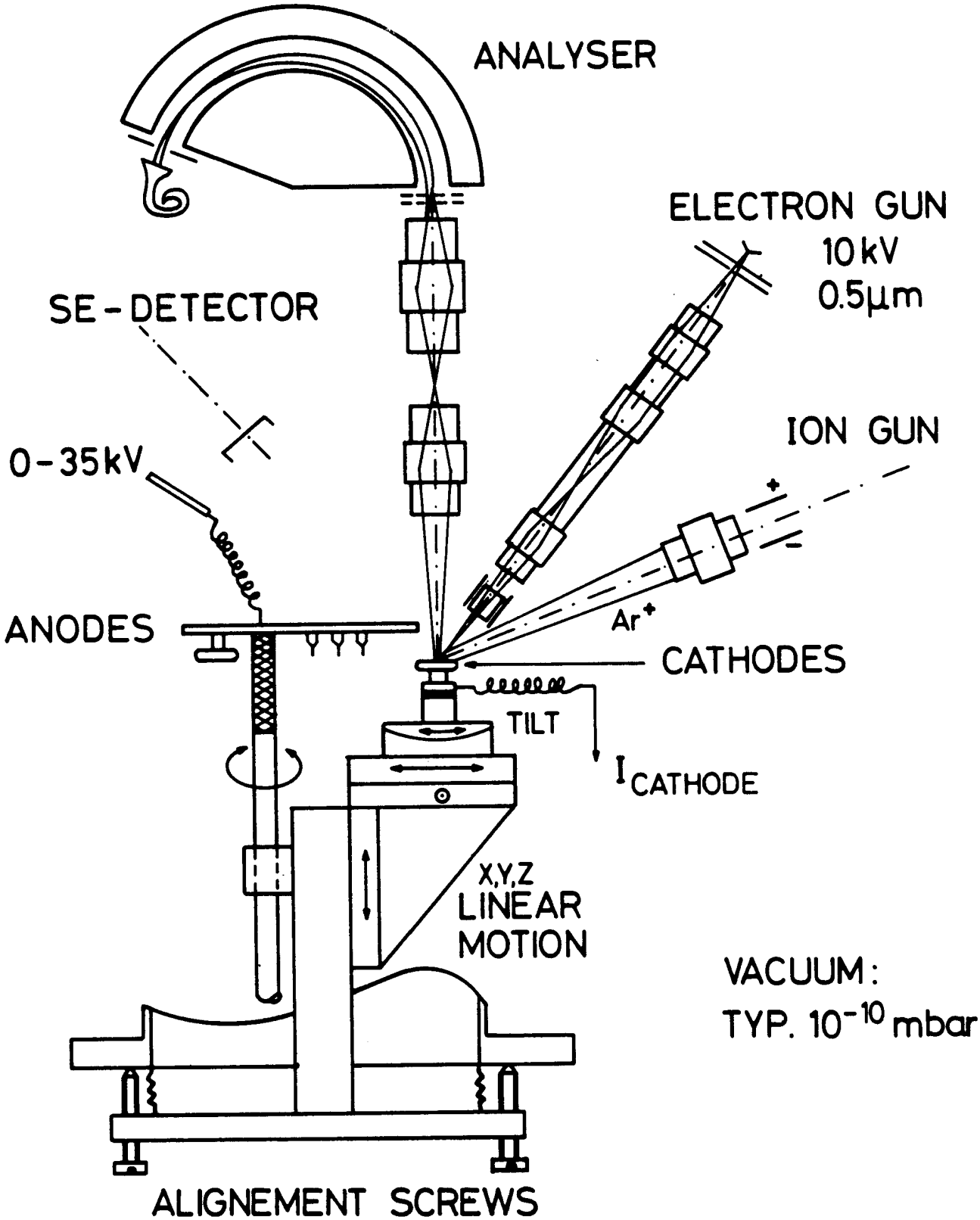
ANALYSER
ELECTRON GUN
10 kV
0.5µm
SE-DETECTOR
ION GUN
0-35kV
ANODES
Ar+
CATHODES
TILT
I CATHODE
X,Y,Z
LINEAR
MOTION
VACUUM:
TYP. 10-10 mbar
ALIGNEMENT SCREWS



Fig. 1

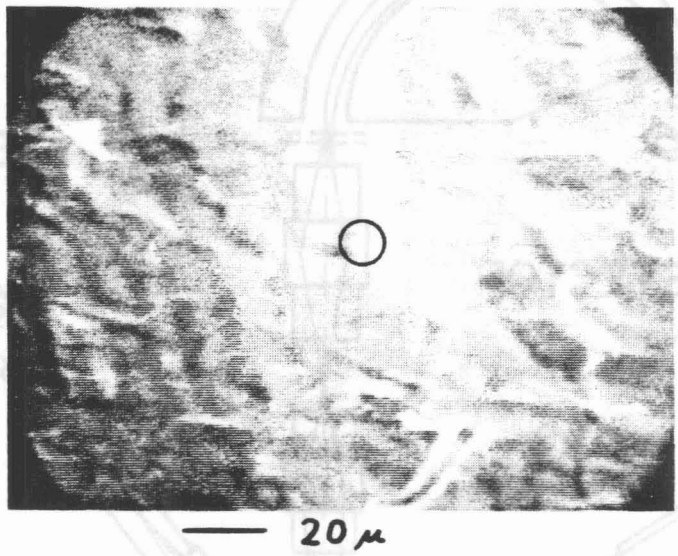


Fig. 2a


Fig. 2b

Fig. 3

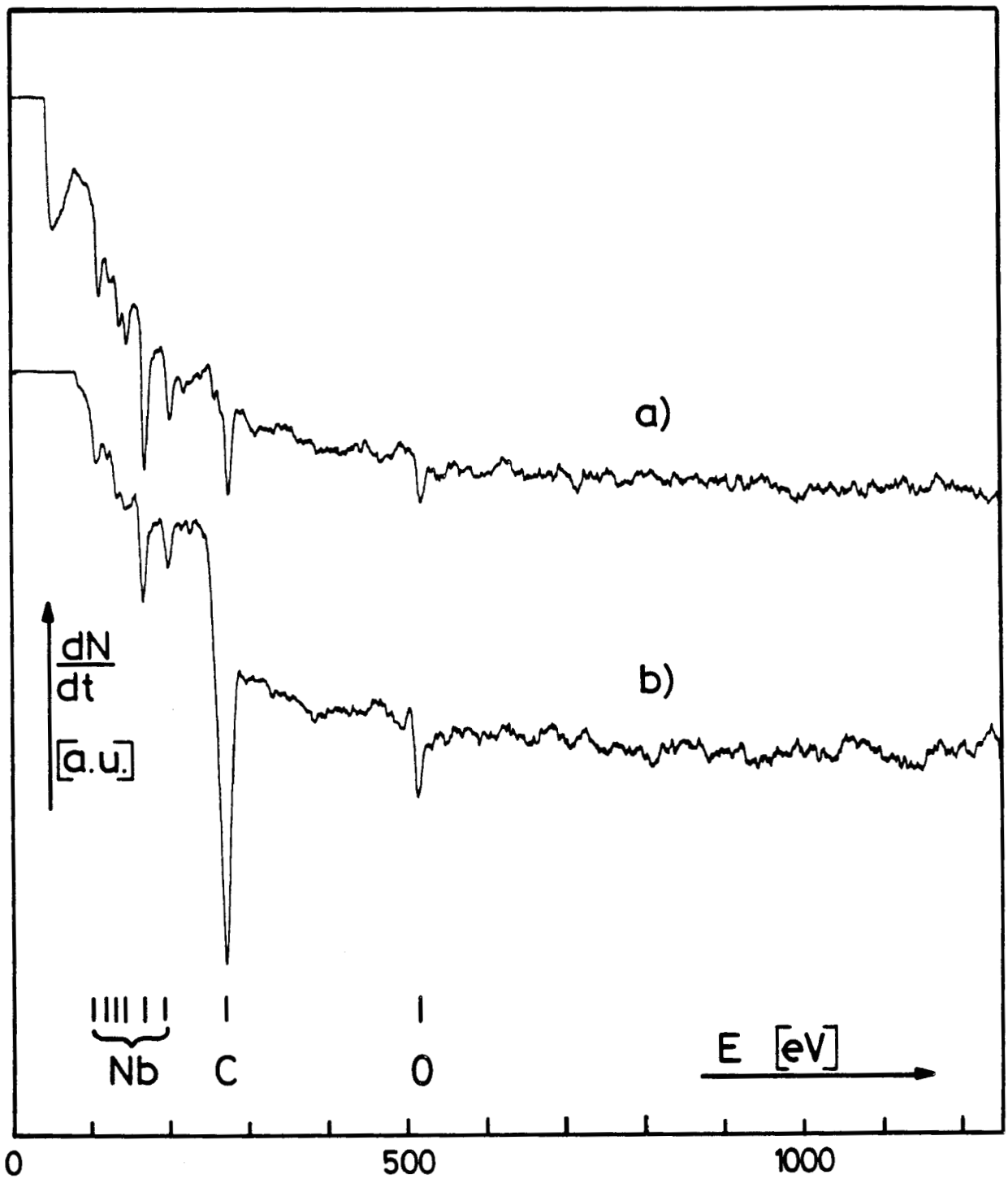




Fig. 4

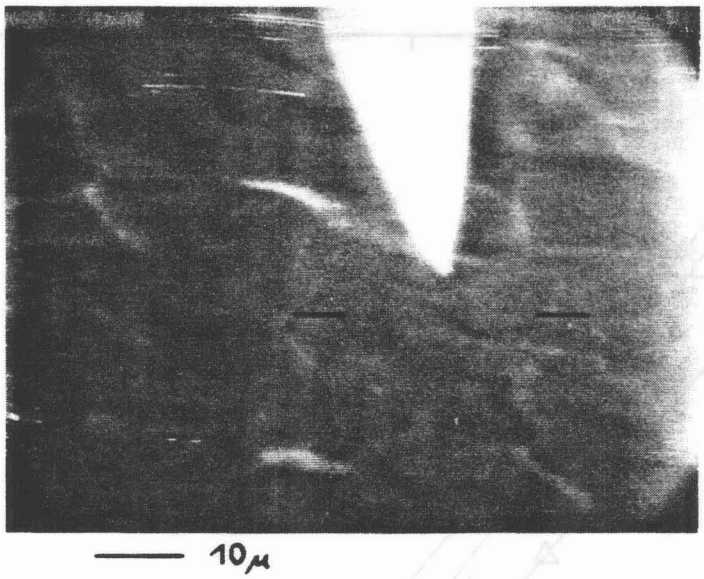


Fig.5a


Fig. 5b

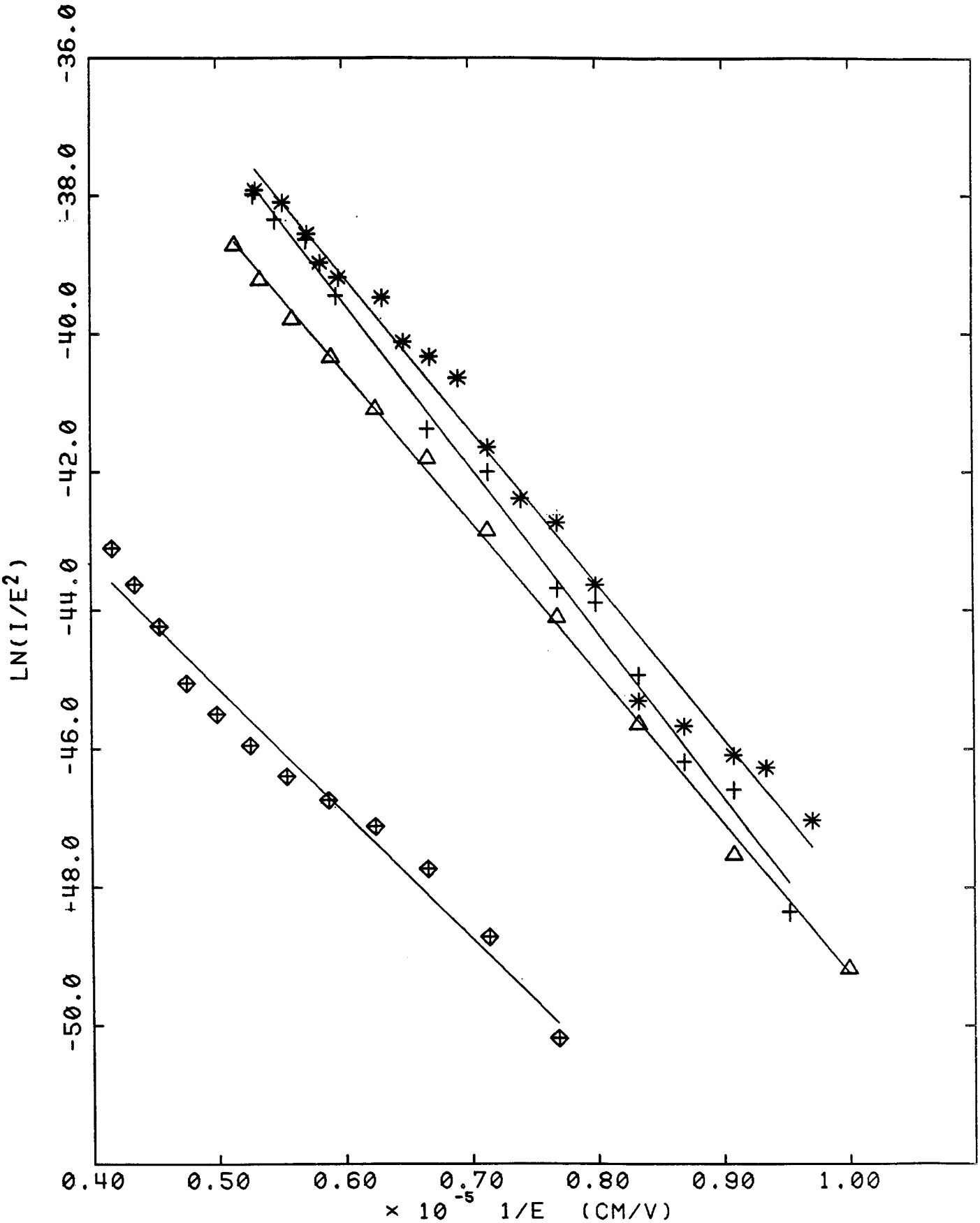
LN(I/E²)
-36.0
-38.0
-40.0
-42.0
-44.0
-46.0
-48.0
-50.0
0.40
0.50
0.60
0.70
0.80
0.90
1.00
× 10⁻⁵ 1/E (CM/V)

Fig. 6