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6. AUTHOR(S)
J. A. Meyer, S. J. Stranick, J. B. Wang, and P. S. Weiss

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Department of Chemistry
152 Davey Laboratory
The Pennsylvania State University
University Park, PA 16802

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FIELD EMISSION CURRENT–VOLTAGE CURVES AS A DIAGNOSTIC FOR SCANNING TUNNELING MICROSCOPE TIPS

J. A. Meyer, S. J. Stranick, J. B. Wang, and P. S. Weiss
Department of Chemistry
The Pennsylvania State University
University Park, PA 16802
USA

ABSTRACT

The current–voltage (I–V) characteristics of a low temperature ultrahigh vacuum scanning tunneling microscope (STM) tip positioned >100 Å from a planar surface have been recorded. We find curvature in the Fowler–Nordheim plots ($\log_{10}\frac{I}{V^2}$ vs. $\frac{1}{V}$) due to the tip–plane geometry as has been predicted theoretically. Additionally, oscillations and sharp breaks in these I–V curves are observed over a wide voltage range, 50–1000 V. These I–V curves are used to characterize the STM tips prior to tunneling.

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I. Introduction

Except for the few scanning tunneling microscopes (STMs) equipped with field ion microscopes [1] or scanning electron microscopes [2], no *in situ* measurement short of recording STM images has given information as to the geometry, particularly the sharpness of the STM tip. In order to have a simple diagnostic for determining tip sharpness without the necessity of approaching the surface to within tunneling range, we have measured field emission current–voltage (I–V) characteristics. It can be shown that the slope of a Fowler–Nordheim plot of the field emission I–V curve is related to the tip sharpness [3]. This is understood in terms of the higher field emission due to the increased electric field for a sharper emitter.

II. Experimental

The experiments were carried out in an ultrahigh vacuum chamber equipped with an STM. This chamber can be kept at room temperature, or at 77K or 4K by immersion in liquid nitrogen or liquid helium, respectively. The apparatus will be described in detail elsewhere [4]. Of relevance here is our ability to switch rapidly between two samples both mounted on a piece of single crystal sapphire. This allows us, for example, to prepare and to measure our field emission I–V curves on a Mo{100} crystal, and then use this same STM tip to image a Si{111} surface.

Our STM tips are prepared by cutting a tungsten wire to the proper length and then either installing these tips directly in our STM, or else first electrochemically etching them to a sharpened point following the procedure of Colton and coworkers [5]. We typically sharpen our tips *in situ* using field–induced sharpening [6]. In order to do this we apply a bias to our tips V=−200–−1000V and pass a field emission current of up to 10^{-5} A to the surface. The bias voltage is under feedback control to provide the field emission current requested [7]. Alternatively, we sputter our tips *in situ* using Ar^+ or other ions, using field emission electrons to ionize the atoms. Since the peak of the electron bombardment ionization cross section for Ar is ~100 eV [8], the field emission electrons are in a convenient energy range for this purpose. The negative bias applied to the STM tip then accelerates these ions towards it. As discussed below, we monitor field emission I–V curves in order to determine our tip sharpness for both tip preparation techniques.

In order to measure field emission I–V curves, we ramp the bias voltage applied to the tip, and record the field emission current by monitoring the current to our crystal surface (held at virtual ground) using a Balzers QME311/EP511 electrometer. Using a Data Precision 8200 Voltage Calibrator as our bias voltage supply, we can record I–V curves up to ±1000V. Essentially all our I–V characteristics are recorded with a negative tip bias. We plot Fowler–Nordheim curves — $\log_{10}\frac{I}{V^2}$ *vs.* $\frac{1}{V}$ — of our field emission I–V curves. Recall that to a reasonable approximation such a plot should yield a straight line for a planar field emitter.

III. Results and Discussion

With our STM tip out of tunneling range (>100 Å), we record the I–V characteristics over voltages in the range $^{-}50$–$^{-}1000$ V within the range of currents measured by our electrometer ($10^{-12}-10^{-5}$ A). While processing our tip by field–induced sharpening or by sputtering, we monitor the field emission I–V characteristics over a smaller voltage range, typically a 10–40V range. Examples of curves taken while performing electric field induced sharpening are shown in figure 1. Note that a deviation from Fowler–Nordheim behavior as discussed in the calculations of Cutler and coworkers [9] is observed. That is, due to the tip–plane geometry, there is curvature in the Fowler–Nordheim plots ($\log I/V^2$ *vs.* $1/V$). Within the narrow range, we take the first derivative of the Fowler–Nordheim plot to be an indication of tip sharpness. The lower the first derivative, the sharper the tip. The sharper the emitter, the higher the electric field, the lower slope of the Fowler–Nordheim plot. This is also consistent with the discussion of Saenz *et al.* and is shown nicely in figure 12 of reference [3]. Note that since we have no measure of absolute tip–surface distance with our STM walker, we cannot record the field emission as a function of (measured) tip–surface distance. If this were possible, this would give us another indication of tip sharpness.

We note two interesting features that appear in our field emission I–V measurements. We characterize the first of these features — a sudden change in the emission current — as a "break" in the I–V curve. An example is shown in figure 2. Note that the slope within the break region is substantially larger than the slope elsewhere. We infer from these features that part of our field emission tip has moved and that the tip is duller during the break region. These breaks are often reproducible and can occur at the same voltages from scan to scan until the tip changes further. The breaks are consistent with what is observed due to the motion of atoms during field–induced sharpening. During field–induced sharpening, the current jumps rapidly back and forth between two or three values at rates faster than our feedback loop can respond (>1 kHz).

A second feature that we occasionally see is one or a series of "oscillations" in Fowler–Nordheim plots of the field emission I–V characteristics as shown in figures 3 and 4. For the microtips that Vu Thien Binh and coworkers fabricate using their pseudo–stationary profile technique, they observe a single reduction in current from a linear Fowler–Nordheim plot of their field emission I–V measurements which they attribute to space charge effects [10], or to effects due to the tip geometry [3]. Current densities in excess of 10^6 A/cm^2 are required for these space charge effects [11]. Here, some of these oscillations may be due to multiple emitters on the STM tip which reach the space charge density required to observe a decreased field emission current at a variety of voltages. However, in addition we observe both decreases and increases in the field emission current. A possible explanation of these features is motion of atoms on the tip. Evidence for this is seen in figure 5 in which what appears as a deviation from a linear plot "breaks" back to the original (lower bias) curve. We therefore speculate that these oscillations are due to motion of atoms on the tip and perhaps also to multiple emitters.

We find that when preparing our STM tips, the magnitude of the first derivative of the Fowler–Nordheim plots of the field emission I–V characteristics decreases. The particular values of the first derivative which we believe are the result of having a sharp tip vary according to tip–sample distance and the voltage range for which field emission is recorded. For this reason it is still difficult to obtain an absolute measure of sharpness with this technique.

IV. Conclusions

We have measured field emission I–V curves of our STM tip–surface system. These curves may prove useful as a simple diagnostic of the apparent sharpness of STM tips. These measurements can be made *in situ*, during tip preparation. In addition, we observe oscillations in the field emission current over ranges of 1–50V. We attribute these to geometry changes of the tip.

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[11] W. P. Dyke and W. W. Dolan, *Adv. Electron. Electron Phys.* **8**, 89 (1956). Note a typographical error in reference [10] — the correct onset current density for space charge effects is $J>10^6$ A/cm^2, rather than $J>106$ A/cm^2.

FIGURE CAPTIONS

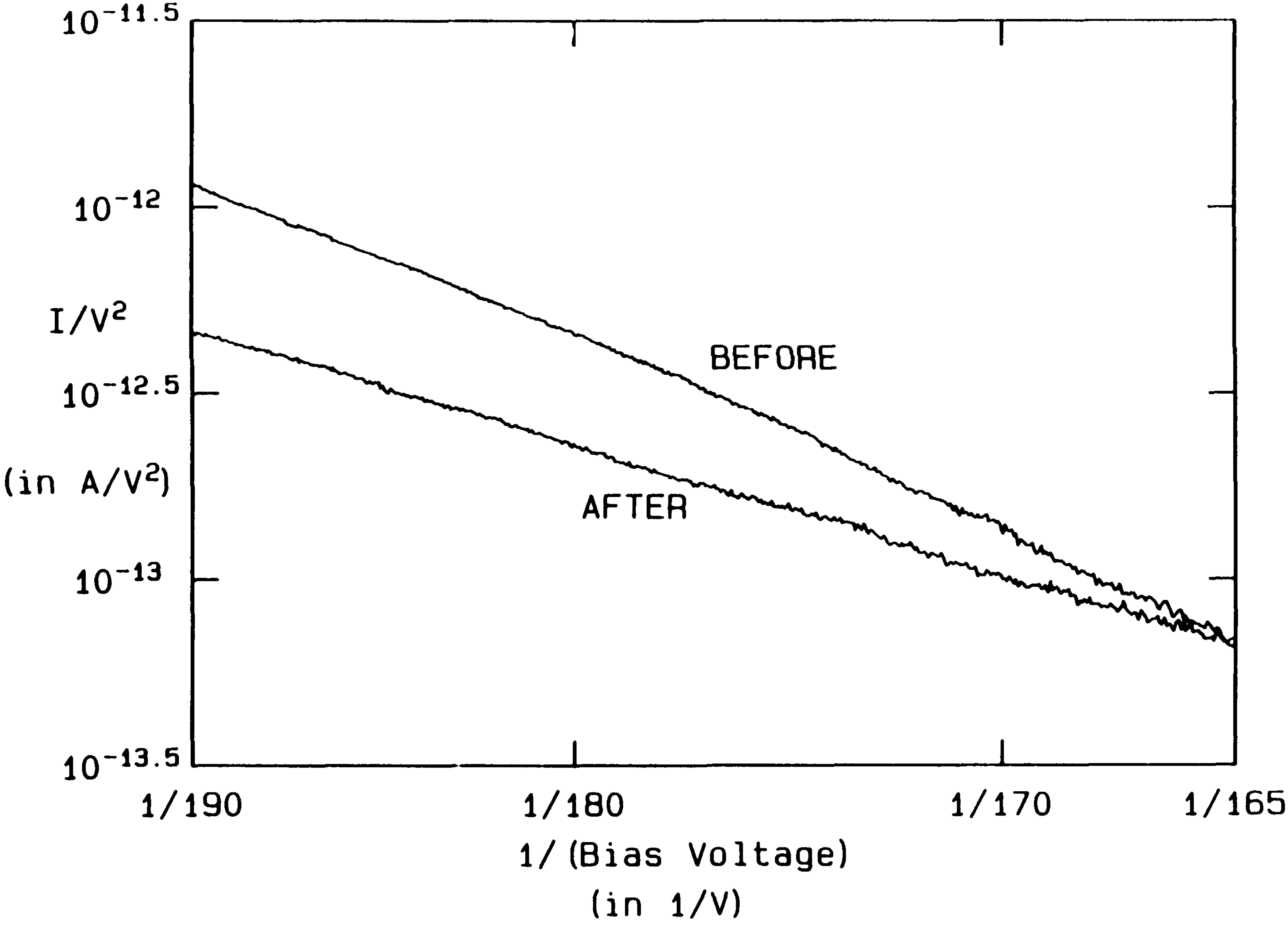
1. Fowler–Nordheim plots of field emission I–V characteristics recorded before and after field–induced tip sharpening.

2. A Fowler–Nordheim plot of a field emission I–V characteristics showing sudden changes in the field emission current. As the voltage is scanned to higher bias, there is first a break, then the emission current returns to the original curve, and then there is another break. Note that the slope in the central "break" region is substantially higher than elsewhere in the plot.

3. Oscillations sometimes appear in the Fowler–Nordheim plots of the field emission I–V characteristics as discussed in the text. A series of seven consecutive measurements all show a feature at the same bias voltage.

4. A series of oscillations appear in two of several consecutive I–V characteristics showing these features.

5. The feature that appears to be an "oscillation" ends abruptly with a break, and the I–V characteristic follows its low bias curve.


$10^{-11.5}$
10^{-12}
I/V^2
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(in A/V^2)
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$10^{-13.5}$
BEFORE
AFTER
1/190
1/180
1/170
1/165
1/(Bias Voltage)
(in 1/V)

Fig. 1

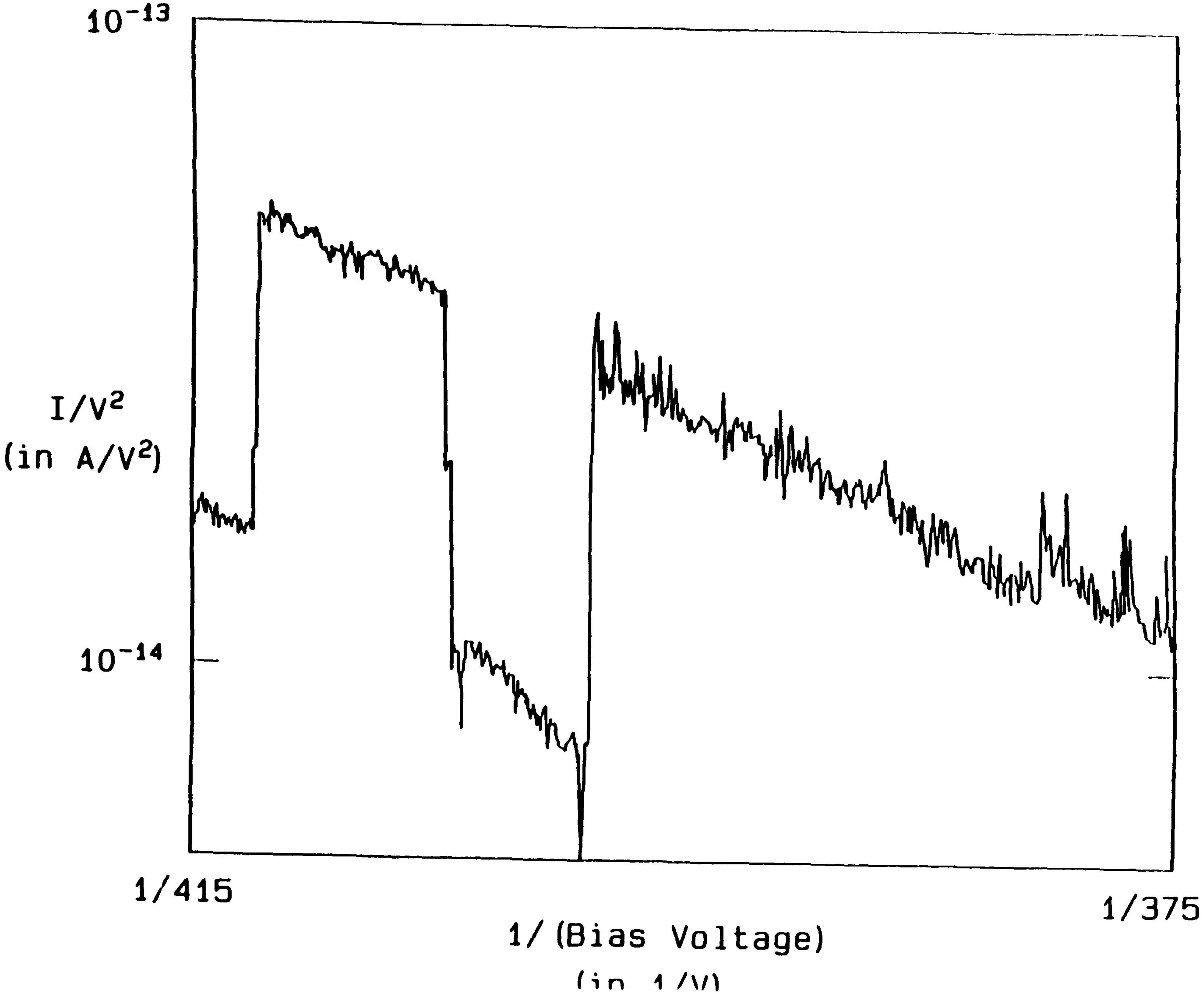


Fig. 2

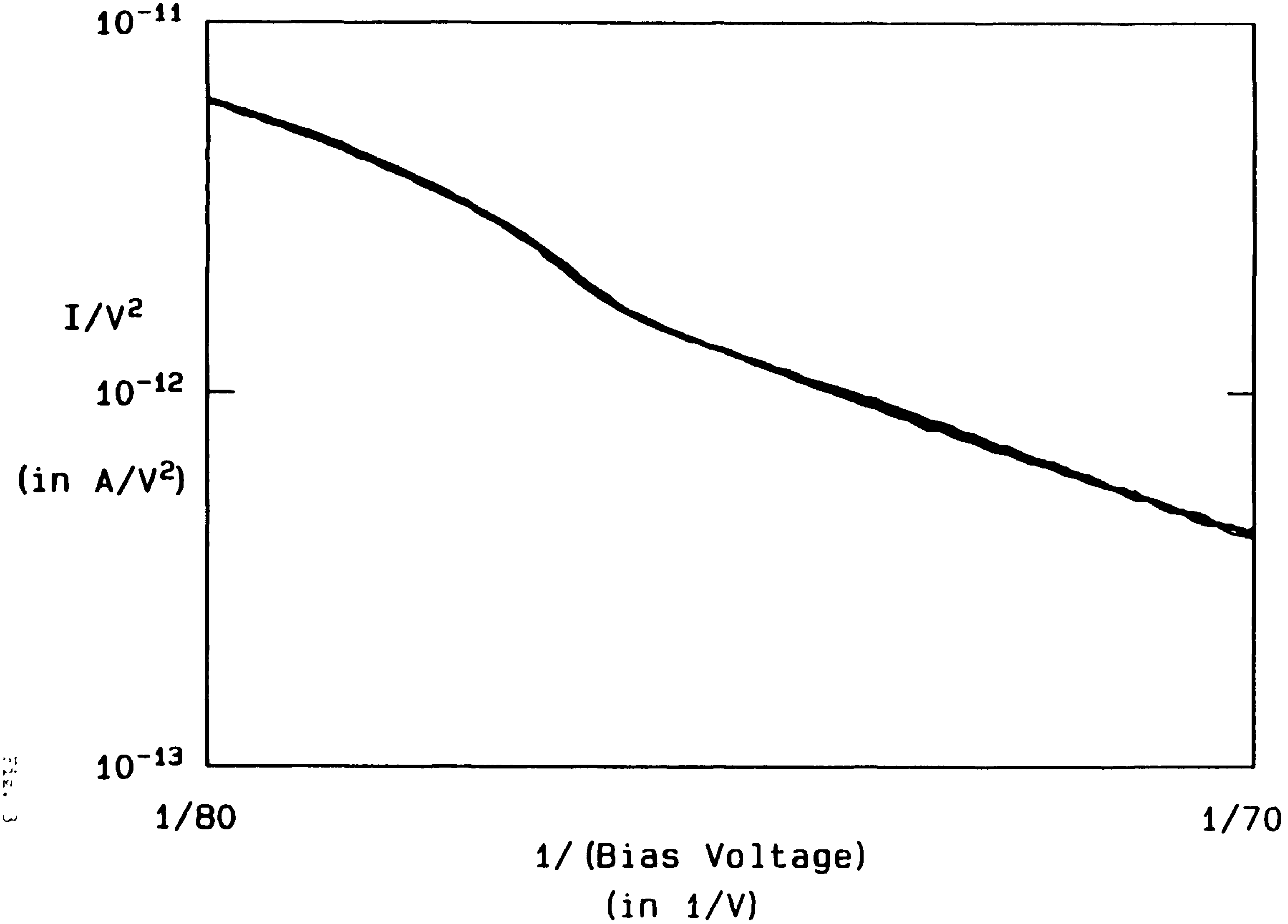
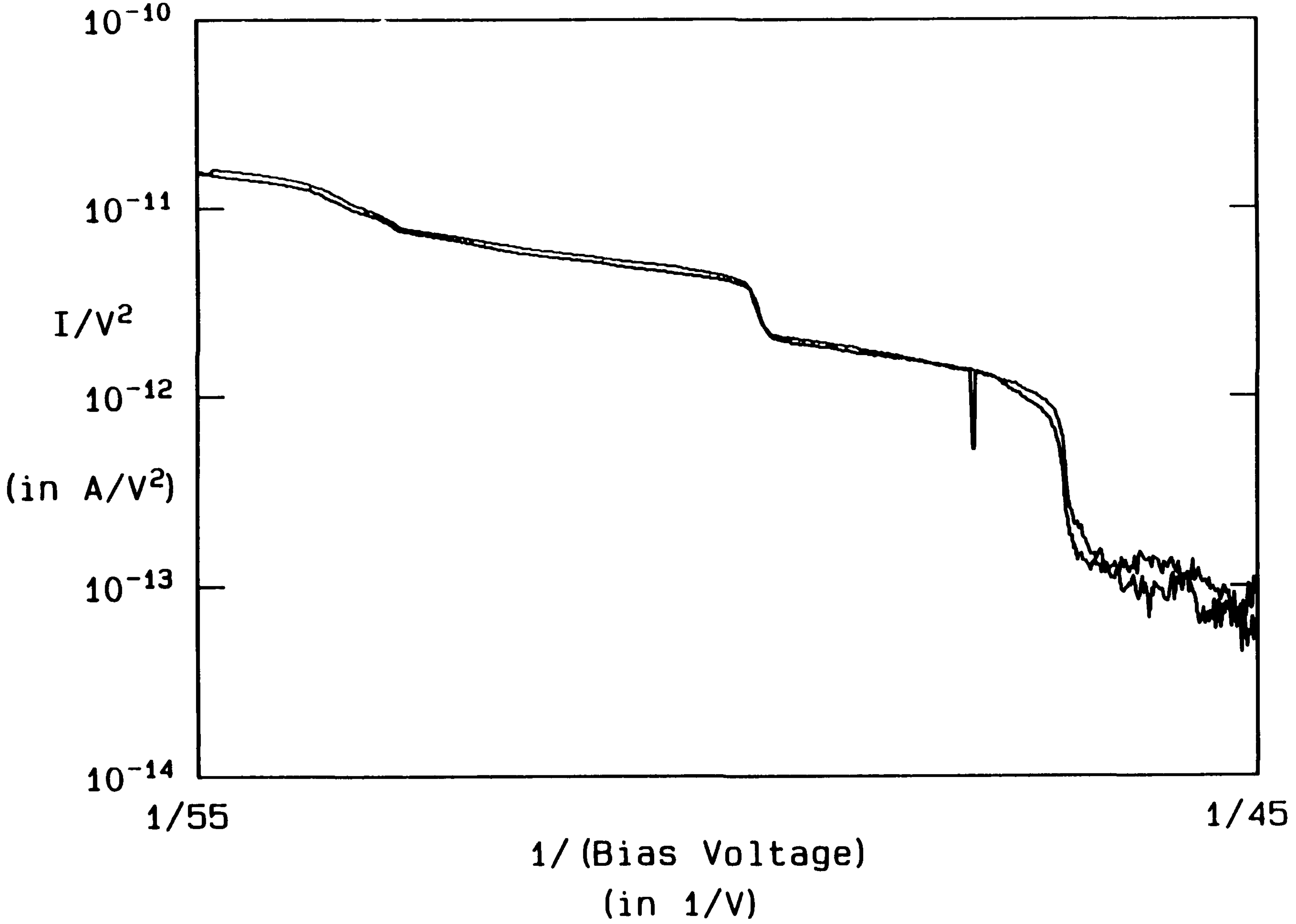


Fig. 3


10^{-10}
10^{-11}
I/V^2
10^{-12}
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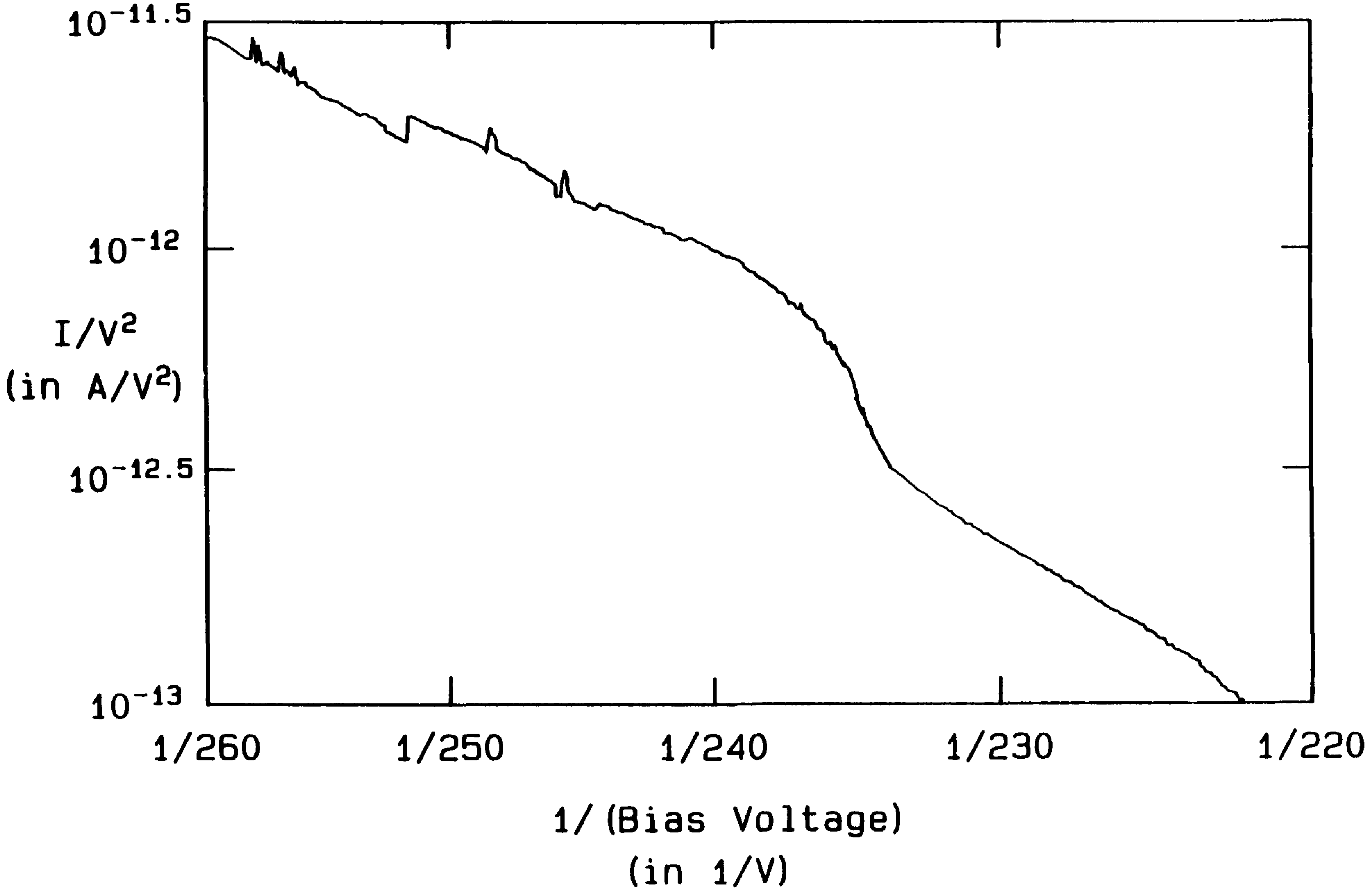
1/(Bias Voltage)
(in 1/V)

Fig. 4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10^{-13}
1/260
1/250
1/240
1/230
1/220
1/ (Bias Voltage)
(in 1/V)

Fig. 5