## RAPID COMMUNICATIONS

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## In situ cleaning of microfabricated field emitter cathodes

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(Received 5 April 2000; accepted 12 June 2000)

The use of microfabricated field emission cathodes in applications of technological importance has been hindered by difficulties in obtaining acceptable emission characteristics in the vacuum environment of the device. We have investigated *in situ* surface cleaning and annealing using high pulsed electron emission. Extraction of high current densities provides a reliable means to shape and clean microfabricated field emitter tips, thereby enhancing their temporal stability and emission spatial uniformity. © 2000 American Vacuum Society. [S0734-211X(00)00605-3]

Microfabricated field emission cathodes are being developed for use in devices such as field emission displays and microwave tubes.<sup>1</sup> In some cases, present cathodes can suffer from emission current noise, poor emission spatial uniformity from individual tips and tips in an array, and degradation of the *I-V* characteristics (i.e., increases in voltage required to extract a given current with operational time). These issues are related to the physical structure and contamination of the emitting surface.<sup>2</sup> A method for shaping and periodic, *in situ* cleaning of field emission cathodes to induce temporally stable and more spatially uniform emission characteristics would clearly be useful in many applications.

The procedure required to clean the surface of the cathode depends on the tip material and the contaminants. Elemental materials such as molybdenum and tungsten can be cleaned of adsorbed and chemisorbed residual gases under ultrahigh vacuum by heating to high temperatures.<sup>3</sup> This treatment also removes common refractory metal surface contaminants such as chlorine, sulfur, and oxides.<sup>3</sup> Unfortunately, many conventional surface cleaning approaches are not compatible with microfabricated field emission cathodes because of the combination of the materials used in their fabrication and their geometrical structure.<sup>2</sup>

Joule heating of field emission cathodes was identified as a precursor to cathode-initiated vacuum breakdown by Dyke and co-workers<sup>4,5</sup> and by Swanson *et al.*<sup>6</sup> and related studies continue.<sup>7</sup> In this communication we report on Joule heating of the emitter tip, by pulsed field electron emission, to initiate thermal desorption cleaning and thermal field surface self-diffusion.

The experimental chamber is a metal and glass field ion

Figure 1 shows the morphological changes induced by pulsed field electron emission from a field evaporated W(110) etched wire tip. Figure 1(a) is a He ion image of the field evaporated end form. The ion image in Fig. 1(b) was formed after evacuation of the image gas and electron emission current pulsing for 20 min. An important feature in the image is the enlargement of all the low index planes such as the (110)s, (100)s, and (211)s, a clear indication of thermally activated, field assisted, surface self-diffusion.<sup>8</sup> Several atomic layers were field evaporated from the (111) planes and surrounding areas as the original imaging voltage was approached. Due to this local field enhancement, the original imaging voltage is well above the best imaging voltage (BIV) for the (111) planes as evidenced by the poor resolution. A noticeable loss of material is seen near the (100) planes. Similar results were observed with molybdenum etched wire tips.

For etched wire tip experiments, the extracted current densities ranged from approximately  $10^7$  to  $10^8$  A/cm<sup>2</sup>, near the values that initiate a vacuum arc using microsecond duration pulses.<sup>4</sup> The electron emission current density, *J*, was calculated by first determining the tip radius, *r*, from the approximate field-voltage proportionality relationship, *F* = *V*/5*r*, where *F* is the applied field and *V* is the applied

microscope operating at less than  $10^{-10}$  Torr. Ion imaging was achieved using a channel electron multiplier array. The emitter tips were cooled to 77 K. Research grade He or Ne image gases were admitted to the system from 1 l glass flasks and further purified by a liquid nitrogen-cooled titanium sublimation pump. Conventional emitter tips were electrochemically etched from polycrystalline tungsten or molybdenum wire. Microfabricated field emitter tips were Spindt type, single-tip molybdenum cathodes fabricated at SRI International.

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FIG. 1. Helium field ion images of a W $\langle 110 \rangle$  etched wire tip. (a) The field evaporated end form, imaging voltage:  $V_i = 7880$  V. (b) The end form following emission current pulsing (20 min, 8 Hz repetition rate, ~1  $\mu$ s pulse width, 3.0 mA maximum electron current, total emission time,  $\tau$ , 10 ms).  $V_i = 7880$  V.

voltage.<sup>9</sup> The applied field is 4.5 V/Å at BIV for tungsten with He.<sup>9</sup> Since the electron emission extends to the outer four visible (110) planes, the emitting area is approximately hemispherical; therefore,  $J \sim I/2\pi r^2$ , where I is the emitted electron current.

As the critical emission current density was approached, sudden increases of  $\sim 30\%$  in the emitted current were observed during the voltage pulse. This sudden rise in current is referred to as "tilt"<sup>4</sup> and has been associated with the onset of thermally assisted field electron emission caused by an increase in tip temperature due to Joule heating. Ion imaging after the observation of several tilt events revealed no unusual tip structure,<sup>6,10</sup> only evidence of thermal field diffusion as shown in Fig. 1(b). Typically, our emission current densities of the structure densities of the several densities of the several densities of the several diffusion current densities of the several dens







FIG. 2. Helium field ion images of a W(110) etched wire tip. (a) The end form created by pulsed emission current heating (4.5 h, 8 Hz repetition rate,  $\sim 1 \ \mu$ s pulse width, 6.0 mA maximum electron current,  $\tau$ =130 ms),  $V_i$  = 10 990 V. (b) The end form in (a) contaminated by oxygen,  $V_i$ =10 990 V. (c) The end form in (b) cleaned by pulsed emission current heating,  $V_i$  = 10 990 V.



FIG. 3. Change in the *I*-V characteristics of a microfabricated single tip molybdenum emitter due to emission current pulsing. The *I*-V characteristic before current pulsing and 2 h following the cessation of current pulsing ( $\bigcirc$ ). The *I*-V characteristic immediately after current pulsing ( $\bigcirc$ ) (70 pulses, 1 Hz repetition rate, 100  $\mu$ s pulse width, maximum electron current 200  $\mu$ A,  $\tau$ =7 ms). Fowler–Nordheim parameters: ( $\bigcirc$ )  $a=2.70\times10^{-6}$ A/V<sup>2</sup>, b=925 V; ( $\bigcirc$ )  $a=6.65\times10^{-6}$  A/V<sup>2</sup>, b=886 V.

sities approximately 30% below where the onset of tilt was observed.

When current pulsing was stopped for several hours or more, we found that the emitted current was initially 30%-50% lower when reapplying voltage pulses of the same magnitude. As pulsing continued, the current gradually increased to its value measured before the cessation of pulsing. Similar results were obtained when the tip was dosed with reactive gas species. Figure 2(a) is a He ion micrograph of a W $\langle 110 \rangle$ etched wire tip after pulsed emission current heating. The tip was then dosed with 200 langmuirs of research grade oxygen and imaged in He [Fig. 2(b)]. The presence of a chemisorbed oxygen layer or initial surface oxide is evidenced by the bright emission spots decorating the axial (110) plane and the step edges surrounding the low index planes. Following evacuation of the He, emission voltage pulsing was initiated at its previous level. During pulsing, the emission current rose nearly monotonically from 1.8 mA to a plateau at 6.0 mA in 50 min ( $\tau$ =24 ms). The ion image in Fig. 2(c), taken after this pulsing treatment, shows the return to the clean surface image. The changes in emission current over time are associated with an increase and a decrease in the average tip surface work function due to, respectively, the adsorption of oxygen and then its gradual thermal desorption by emission current pulsing. The desorption rate depends exponentially on the temperature and is therefore sensitive to the extracted current density.5

Emission current noise has been associated with the presence of physically and chemisorbed gases on microfabricated cathodes;<sup>11</sup> thus, thermal surface cleaning should decrease this noise. Spindt type microfabricated tips have an overall length, *l*, of approximately 1  $\mu$ m, which is less than that of typical etched wire emitters (>1 mm). In addition, Spindt tips are attached to a massive substrate having good thermal conductivity. However, steady-state heat transfer equations<sup>5</sup> show that such considerations, given similar tip cone half angles, are not important in correlating the tip temperature to the pulsed emission current until the tip's length approaches the dimensions of the tip radius. In the case of typical Spindt cathodes  $l \gg r$  as  $r \sim 200$  Å.

Figure 3 shows the change in the *I*-*V* characteristics of a microfabricated single tip before and after emission current pulsing. The I-V curve slowly reverts from the post- to the prepulsing characteristic during 2 h of cathode operation at  $1 \times 10^{-10}$  Torr and emission levels of 10  $\mu$ A. The change between the pre- and postpulsing I-V characteristics could be reproduced by again pulsing the emission current at 200  $\mu A$  and then operating the cathode. We associate these changes with adsorption and desorption of residual gas (probably hydrogen) from the cathode surface and the respective increases and decreases in the average surface work function. Because the tip radii and cone half angles of the microfabricated tips investigated were similar to those of the etched wire emitter tips, currents on the order of several milliamperes will be required to reach tip temperatures sufficient to desorb well bound contaminants, activate surface self-diffusion, and thereby induce a *permanent* change in the I-V characteristic relative to that observed before any pulsing. Clearly, emission current levels of several hundred microamperes cause no permanent changes in the tip surface due to surface self-diffusion. Similar results were observed with all 12 microfabricated tips tested.

Microfabricated cathode gate-to-base oxide thicknesses of  $\sim 0.6 \ \mu m$  were used in the present experiments. This oxide thickness must be increased to allow for the application of the voltages required to draw current densities that initiate significant surface self-diffusion without gate-to-base surface voltage breakdown. At the proper pulse length-temperature (i.e., current) combination, annealing of the surface of the microfabricated emitter tip should occur with currents on the order of several milliamperes and time scales of several milliseconds. Surface tension forces will result in net smoothing of the emitter tip surface,<sup>2,8</sup> thereby enhancing the spatial uniformity of the emission from the tip. We have shown that pulsing to even the modest emission levels discussed above can dramatically decrease microfabricated cathode emission current noise.<sup>12</sup> Finally, we note that with the development of "thick oxide" cathodes there is the possibility of using field evaporation<sup>9</sup> as a tip cleaning, shaping, and surface analysis<sup>13</sup> technique.

The authors gratefully acknowledge many useful discussions with Capp Spindt of SRI, and the support of SRI's Vacuum Microelectronics Staff: C. E. Holland, S. L. Shepherd, D. A. Thibert, and M. Simkins. This research was funded by Ion Diagnostics, Inc., of Santa Clara, California.

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