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CATHODE BOMBARDMENT STIMULATED MICROSTRUCTURE GROWTH - AVERAGE ION ENERGY

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Abstract - Surface microprotrusions have been associated with cathode initiated vacuum breakdown processes and some interpretations of their formation include surface migration and field enhanced growth. Microstructure growth on field emitters has been observed in association with low temperature (5.0K) adsorption of hydrogen or helium and electron emission currents of 200 to 1000 nA. The presence of adsorbed films (multilayer films for hydrogen) at 5.0K and the general surface damage without stimulated growth at liquid nitrogen temperature suggest that a high dose of low energy ions formed near the emitter surface may activate the surface diffusion. The nucleation of the growth site may result from a number of different events : i) Surface density fluctuations which achieve a critical local field enhancement, ii) Surface damage resulting from the impact of a high energy ion, iii) Threshold surface etching by ion sputtering. A model calculation of the ion energy indicates that at 5.0K, $\approx 86\%$ of the hydrogen ions have a kinetic energy less than 100 eV (0.35 V/Å Applied Field, 3300 V). Similar effects are demonstrated for He cathode bombardment. Low energy helium ion bombardment activated surface self diffusion in the presence of an applied field provides several technical advantages for high brightness field ion source development.

Introduction

The field ion microscope has been applied primarily exploiting its capability of imaging surface atoms. For practical application as an ion beam source, however, emission current dependencies upon single surface atoms is a severe limitation. Atomic surface rearrangement processes appear as beam current fluctuations¹ as well as angular distribution fluctuations. The relative fluctuation is inversely proportional to the square root of the number of surface atoms¹ imaged by the beam. During long term source operation, single surface site events such as negative ion impact cannot be eliminated. Localized emission from molecular size surface features² are inherently very difficult to control on rounded, well annealed field emitter endforms. Such emitter morphology constraints and other practical considerations of alignment, emitter processing, emission distribution stability and emitter lifetime determine the usefulness of gaseous field emission ion sources.

For the field ion microscope, the axial ion current is low. As the total emitter current increases, the source emittance also increases, and the brightness remains relatively low. The distribution of ion emission in the I-V regime above threshold is dominated by surface supply mechanisms^{3,4} with a length scale of an emitter radius. Emitter surface regions which have reached threshold for ionization due to higher field enhancement factors act as sinks of the mobile surface supply and sources of field ions. A surface structure of low field enhancement factor, 1.2-1.5, acts as an ion beam source. Angular magnification factors⁵ show that the image of a 0.05 μm diameter surface feature is compressed by a 0.5 μm radius emitter into a beam with a half-angle of 0.02 radian. Such a surface area consists of $\approx 2 \times 10^4$ surface atoms and produces hydrogen ion current densities of $\approx 2 \times 10^3 \text{ A/cm}^2$. This ion image, compared with the large angle ion distribution of the FIM, is emittance matched for beam forming systems which require 1-10mr acceptance half angles. The successful application of such an emission microstructure as an ion source relies upon techniques to make and stabilize this emitter topography.

Caraille and Drechsler,⁶ and Dranova and Mikhailowski⁷ have demonstrated the production of field stabilized growth of protrusions. The authors interpret this phenomenon in terms of ion bombardment activated surface atom transport. These experiments were conducted with substrate temperatures 77-100K and apparent doses, (electron current) $\times$ (time) $\times$ (gas pressure), which are significantly larger than those required if a base gas and emitter temperature of 5K is used.^{8,9} This work at 5K by Schwoebel and Hanson^{8,9} primarily with hydrogen cathode bombardment, is limited in its application for ion sources because of the embrittling effect of the implanted hydrogen in the surface. Further investigation of the 5K growth is warranted because the initial hydrogen results did not distinguish clearly the relative importance of ion bombardment, dielectric effects or work function-electron emission effects. It is suggested that enhanced growth at 5K is dominated by the temperature dependence of the ion bombarding energy distribution; a preponderance of low energy ions (25-500eV) increases the rate constant for surface self diffusion relative to that for sputtering and surface layer amorphization. Due to the electrostatic stress on the emitter surface, the last two effects are to be minimized for a successful application of such microstructure growth as a field ion source for beam application. Observation of microprotrusion growth using helium ions and tungsten or iridium substrates support the above interpretation and add data to that for hydrogen ion bombardment at 5K.

Experimental

Using a helium cooled ion source assembly,⁸ helium ion imaging of helium cathode bombardment induced microstructure growth has been done on tungsten and iridium by standard field emission techniques. Figure 1 is a sequence of helium ion, threshold emission patterns from a tungsten microstructure, applied average field-F=2.6V/A. The diameter of the structure is $\approx 200\text{\AA}$. Figure 2 shows a field electron pattern of an iridium microstructure grown by 5K helium bombardment. In this case, growth occurred at very low total apparent dose ($\approx 100\text{nA}$ for 1 minute at 5×10^{-5} torr helium). This occurred at one of the four field enhanced surface points which are in proximity to both the (100) and (211) facet edges. Figure 3 shows a sequence of helium ion patterns before and after growth on the (100) regions of a field evaporated tungsten emitter by helium cathode bombardment at 5K. The emitter was field evaporated (7021V) before surface self-diffusion was activated by helium bombardment at 5K. Figure 3a shows the surface before bombardment. Figure 3b shows helium ion emission concentrated in the regions of enhanced field after ion bombardment. Figures 3c and 3d show the ion image after partial desorption of the microstructure with surface atoms in high free energy sites across the emitter surface. Figure 3e is an image after field evaporation at 6978V. There is no evidence of large scale surface damage or blistering. The experiment showed a surface layer which is scrambled by the ion bombardment. This is made apparent by a lowering of the evaporation field (4.5-5V/A) of redistributed surface atoms. Helium must also be present in the substrate surface layers as a result of the bombardment.

The region which is field-enhanced by the ion bombardment shows a high degree of crystallinity under the surface selvedge referred to above. For the magnification available, no change of emitter radius is detectable after growth and removal of the field enhanced region.

Discussion and Theory

Work function and dielectric effects of helium on iridium are small compared to those of hydrogen on tungsten (5K). Therefore, current changes during cathode bombardment experiments using helium-iridium at 5K are more easily interpreted in terms of ion-solid interactions. Adlayers have the effect of decreasing ion-substrate collision energies and scattering incident ion-momentum into directions transverse to the substrate surface. Heavy helium adlayers cannot exist at 5K¹⁰ particularly under cathode bombardment and the neutral gas distribution must be considered as the only dependent variable of temperature. Although the series in Figure 3 was observed using a highly curved emitter, and therefore low voltage, the energy distribution of the bombarding ions ranges from 25-790eV. For helium ions to create a bulk interstitial, 480eV ions are required. Surface diffusion activation energies span values from ≈ 1 to 3.1eV¹¹ and therefore require much lower threshold ion energies, ≈ 37 eV He⁺ ion. Figure 4 shows that the sputtering threshold is ≈ 110 eV for a sputter activation energy of 8.8eV. The damage mechanisms operative for large radius emitters to be used in source research are operative in this case.

At liquid nitrogen temperature (77K), the required apparent dose (e⁻ current x time for growth x chamber pressure) for growth of single microstructures using hydrogen or helium is sufficiently high that within the normal operating constraints, 10⁻⁶-10⁻⁴ torr chamber pressure and emission currents of 100-1000nA, only generalized surface damage is found. Previous workers report results with higher total apparent dose at pressures where ion-neutral collisions may affect the incident ion energy distribution.

Ions with energies below the sputter threshold will activate surface diffusion without a sputter loss fraction. The density of these ions is strongly temperature dependent through a boltzmann factor of the polarization. It is suggested that single microstructure growth occurs when the relative dose of low energy transfers which activate surface self diffusion without sputtering is greater than the rate of loss of surface material for all impinging (ion energy range, 25V to applied emitter voltage) ions. This relative dose is greatest if the total energy distribution is peaked at low values. For continued growth, the time required to grow a microstructure must be less than the time for direct impact at the microstructure by a high energy event which obliterates the cluster nucleus or changes its shape. With increasing chamber pressures, the dose of incoming ions having a kinetic energy equal to the ion potential energy at the instant of electron impact would become less frequent because of increasing multiple ion-neutral collisions which distribute the field energy of the ion.

At low pressure and low temperature (5K), the distribution of the neutral gas particle concentration is strongly weighted to the high potential energy region of the emitter polarization field. The electron impact cross section is peaked at electron energies of ≈ 100 eV. These two factors determine the ion energy distribution function. Investigations of the ion backbombardment dose have been given by several authors,^{6,7,12,13} but without consideration of the neutral concentration effects induced by the emitter. An axial electron current, I, expands as depicted in Figure 5 where Z is the axial distance from the emitter and V is the potential. For this model calculation, the deviation of the bombarding ion trajectory¹³ from the field lines will be neglected; an approximation appropriate only for the low energy ions. Ions formed far from the surface do not follow the electron trajectory back to the emission point. For such an approximation to the real case, the surface ion bombardment rate produced by ions originating at Z is:

$$\frac{dn}{dt} = \frac{I}{e} C(Z) \sigma dZ \quad (1)$$

where $C(Z)$ is the concentration of neutrals and σ is the electron impact ionization cross section. This equation does not consider multiple ionization or ion-neutral collisions. The kinetic energy of the bombarding ion is determined by its initial distance from the surface when the electron impact occurs.

$$K_{ion} = eV(Z) = \int_0^Z eF(Z) dZ \quad (2)$$

The electron impact ionization cross section is therefore expressible in terms of the ion energy using Eqn. 2; $\sigma = \sigma(K)$. The factor $C(Z)$, assuming equilibrium is given by the boltzmann factor:

$$C(K) = C_0 \exp \frac{\alpha F^2(K)}{2kT} \quad (3)$$

The axial distance dependence of the electrostatic field is approximated by:

$$F(Z) = \frac{V_0}{\tanh^{-1} \frac{D}{D+R}} \frac{1}{D+R} \frac{1}{1 - \left(\frac{D-Z}{D+R} \right)^2} \quad (4)$$

where D is the emitter-cathode distance and R is an emitter curvature parameter (the focus of the hyperboloid representing the emitter).

Figure 6 shows the relative ion dose rate as a function of ion kinetic energy,

$$\frac{dn}{dtdK} = \frac{I}{e^2} \frac{\sigma(K) C(K)}{F(K)} \quad (5)$$

The distribution for a relatively large emitter radius (Figure 6) divides into two distinct regimes: the low energy (25-500eV) regime dominated by the boltzmann factor and the high energy regime which in the one-dimensional approximation is dominated by the inverse field dependence. It is apparent from Figure 6 that ion bombardment at very low temperatures significantly increases the low energy ion dose rate which activates surface migration without sputtering. The 77K distribution in Figure 6 is not significantly different from the 294K distribution function. Its shape is primarily determined by the energy dependence of the electron impact ionization cross section.¹⁴ For small emitter radii, the rapid field decrease with axial distance decreases the effect of the concentration factor; however, for small emitter radii, the low electron emission voltages inherently make a low energy ion distribution.

Conclusion

Activation of surface self diffusion by He^+ impact with resulting field stabilized growth can occur at 5K without extensive radiation damage. Gas blisters are not observed and bulk damage such as microcrystal formation can be avoided. Possibilities for ion beam source application of diffusion limited emitter morphological changes is greatly enhanced by helium ion activation (5K He gas) which minimizes chemical effects of the implanted gas species; 5K hydrogen ion activated structures are subject to implanted hydrogen embrittlement. Surface selvedge or surface damage is observed and must be minimized. Recrystallization of the surface layer and protuberance by thermal-field techniques will further increase the structural integrity of the processed endform. Successful

protuberance growth on iridium has the advantage of increased resistance to chemical effects which degrade long term source performance and complicate emitter operation.

Investigation of the surface-self diffusion activation by very low energy ions, from 10-100eV, is warranted because such processing would eliminate sputtering and benefit the growth process in several technical ways: i) Decrease large energy transfers which limit the total growth time for a single microstructure, ii) Decrease the implanted fraction, iii) Decrease the depth and energy of the substrate damage distribution, and iv) Decrease the activation energy necessary for thermal desorption¹⁵ of the implanted helium.

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Figures

Figure 1. He^+ field ion image - localized emission from a surface microstructure grown on an annealed tungsten emitter by 5K helium cathode bombardment. The pattern was photographed every 15 minutes.

Figure 2. Field electron micrograph of $\langle 100 \rangle$ Iridium after the growth of a microstructure by 5K helium cathode bombardment.

Figure 3. He^+ field ion image of substrate effects due to 5K helium cathode bombardment. a) before growth: evap. voltage - 7021V, image voltage - 5673V. b) after cathode bombardment induced growth on the (100) faces: image voltage - 2676V. c) after some evaporation: evap. voltage - 5766V, image voltage - 5766V. d) after further evaporation: evap. voltage - 6700V, image voltage - 5812V. e) after returning evaporation voltage to the initial value: evap. voltage - 6978V, image voltage - 5927V.

Figure 4. Sputter yield measurements summary, H.H. Anderson and H.L. Bay, Sputtering by Particle Bombardment I (Springer-Verlag, Berlin, 1981) p. 145.

Figure 5. Schematic representing one-dimensional ion dose rate calculation.

Figure 6. Ion dose rate as a function of ion energy for a one dimensional calculation using a hyperboloidal endform.