**Limitations on the use of scanning probe microscopy for the measurement of field
emission from copper surfaces**

Y. Mizuno and R. E. Kirby

**The Stanford Linear Accelerator Center
Stanford University**

Stanford, CA 94309

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Abstract

High electric-field breakdown in cm-wave x-band radio frequency (rf) accelerating structures is an important phenomenon limiting attainable accelerating gradients. Linear collider development requires an accelerating gradient of, at least, 70MeV/m (150MV/m surface electric field). The observed breakdown sequence usually consists of field emission (FE) from an electrically-conducting surface feature which heats the point of emission, thereby releasing gas from the surface and nearby bulk. The ionized gas makes a plasma that regeneratively heats the metal and releases more gas and electrons (via secondary emission) until a flashover occurs. The FE, however, occurs at a much lower surface electric field in experiments than predicted by theory. Experimental measurements of FE from macroscopic surface areas require the use of a geometrical field-enhancement factor, β , to fit best the data to a Fowler-Nordheim (F-N) emission model with reasonable physical parameters. A newer, microscopic interpretation of the results proposes the existence of quasi-filamentary electrically-conducting channels between the metal bulk and the surface. These channels connect to emitting features near or on the surface, acting as microscopically field-enhanced electron emission sources. Analysis of emitter behavior suggesting that the macroscopic emission should primarily be due to classical geometrical-enhancement is, at least in some cases, due to the presence of these conducting nano-structures in the surface region. We describe an accelerator materials-related study of high electric field breakdown from polished copper (and other) surfaces, in dry-nitrogen gas ambient, using an atomic force microscope (AFM) modified to make F-N FE measurements.

In early periods of this work, the results showed that the instrument might be capable of making F-N measurements on small surface areas of mechanically-polished and natively-oxidized high-quality copper surfaces to the GV/m level. The conductive AFM is a topography-imaging instrument capable of extremely high magnification, making it possible to image the actual point of field emission. The technique appeared to be a powerful tool for in-situ measurement of the F-N current combined with imaging of the emitter itself. Following initially promising results, we began a systematic program of characterizing FE from natively-oxidized and oxide-coated polished copper surfaces. The results obtained were, however, not consistently repeatable and we believed that the difficulty was due to a lack of a mechanically-robust and electrically-conductive coating for the AFM tip surface. Similar problems have been recently reported by O'Shea et al for similar measurements. The metal-coated tips have been found to wear rapidly during image scanning and measuring current such that the conductive layer on the very end of the tip becomes insulating after minimal use. This difficulty is magnified by working in ambient because the sample and/or tip becomes quickly contaminated, producing chemical changes to the surface barrier heights in the high electric field region. Even in vacuum, wear of tip is found to affect tip-sample adhesion. In general, metal-coated tips are not reliable for obtaining repeatable FE data, although the problem is not completely resolved.

We present our data for conductive-AFM using Pt/Ir-coated Si tips generating FE from natively-oxidized Cu, and films of Au on mica, Al_2O_3 on Pt, Mg on Cu, Hf and W on Cu and, also, Hf ion-implanted into Cu, all in dry nitrogen atmosphere. Hardened surfaces, such as Hf-implanted, and oxide coatings on metals have historically exhibited higher breakdown thresholds and were chosen as sample surfaces for our investigation. F-N behavior was observed in all cases; however, the difficulties pointed out by O'Shea et al were readily observable.

1 Introduction

High-electric-field gradient accelerator structure development is focussed on achieving gradients of 0.5GeV/m ,¹ with an immediate goal of 70 MeV/m for the Next Linear Collider (NLC).² Radio frequency (rf) accelerators at this gradient will be operated around 100 GHz (30 GHz for NLC), fixing the manufacturing tolerance on the physical structure to the sub-micron level.³ Electron field emission (FE), which presages breakdown, is an important effect at these gradients and will need to be suppressed in order to prevent it from detuning the accelerating cavity structure. Coating metal structures with a uniform thin oxide has proven in the past to be a useful technique for raising the FE threshold.⁴ However, once a coated metal surface suffers a breakdown event, it is usually irreparably damaged. The phenomena responsible for breakdown events are very complex, only moderately well-understood, and not under good control.

The basic understanding of breakdown events is as following:

- (i) Electrons emitted from a metal surface strike other places in the structure and these, due to local heating, cause sufficient gas or metal vapor to be emitted for gas multiplication to occur. A stable vacuum arc can then be formed.⁵
- (ii) Another possibility places the source of initial instability on a metal surface. The electrons emitted from a localized site (assumed to be a protrusion or whisker) on the surface cause local resistive heating, leading to an instability due to emitted gas, melting, or migration. The mechanism predicts a critical electric field at which a breakdown will occur independent of the applied electric field.⁶

Figure 1 illustrates a schematic early stage of a breakdown event, and Figure 2 shows a crater on a metal surface, caused by a high voltage breakdown which completely destroys structures and information about the surface prior to the event.⁷ It would be useful to evaluate the effects that surface finishes and coatings have on breakdown behavior by measuring the FE characteristics and imaging the FE features in an rf structure. Such an instrument would be quite complex (if not impossible) and expensive to integrate into a real accelerating structure; however, if we focus on the earliest stage of the breakdown phenomena, electron emission, and measure only a representative piece of material, some useful knowledge can be gained from an examination of the DC, as opposed to the rf, case. Scanning probe microscopy (SPM) is a candidate technique that can study the electron emission processes at early stages of the breakdown phenomena.⁸⁻¹⁰ In the SPM technique, the tip is used to measure electrical properties of the localized surface region while the tip-sample spacing is controlled using the intensities of physical quantities, e.g., an atomic force, tunnel current and capacitance etc.¹¹⁻¹³ In this method, the local topographic and electrical properties of conducting or dielectric surfaces can be measured quasi-simultaneously.

On macroscopic metal surfaces subjected to a high electric field breakdown event, electron FE is observed at

fields which are much below the value predicted by the Fowler-Nordheim (F-N) law for a flat metal surface.¹⁴ This enhanced field emission (EFE) typically originates from a small number of localized spots (e.g., dust, particles, protrusions, whiskers, etc.) on the macroscopic surface.¹⁶ These sites can be described by a field enhancement factor, β , which is often of the order of 100 or more.¹⁷ It is well established by now that microscopic dust and particles, often of a composition different from that of the substrate, are at the origin of the EFE.¹⁹ However, the physical picture of EFE is not sufficient to explain the details of electron emission from metal surfaces (e.g., with Latham's models).²⁰ It is believed that EFE is in general not explained solely by the geometric field enhancement caused by sharp and complex metallic structures on the surface but, that at least part of the enhancement is due to an increased internal fields at localized insulating sections of the emitting micro- or nano-structures.²¹ A microscopic electric field, in a vacuum breakdown event, occurs in an electrically nano-structured surface region having quasi-filamentary conducting channels between the surface and bulk of the metal. These channels connect to emitting features near or on the surface, and act as electron emission sources themselves.²² The characteristics of the channel ensures that sufficient local field enhancement occurs at the emitting source to encourage tunneling. Analysis of the behavior of emitters suggests that the emission should primarily be due to geometrical or characteristic field enhancement, which at least in some cases arises from the conducting nano-structure inside the local surface region.²⁴ Such models with an internal (channel) field-enhancement hypothesis are able to explain qualitatively the difficulties with Latham's theory for describing field-induced emission surfaces.²⁶ For example, the electron emission is predicted, in Latham's theory, to occur via localized surface states. However, when the filled surface states screen the external applied field, the transition processes moves to a limited-current regime which is accompanied by the presence of anomalous electron states at the channel point. This is an important development for semiconductor devices because the extremely thin gate oxide films must support fields of a several V/nm.²⁸ In flat panel field emitter displays, bright emitters must perform at high fields with long life.²⁹ Clearly, better understanding of the emission phenomena at the nano-scale is needed for further understanding of the breakdown process itself.

In order to investigate the topographic specifics of field emitters, SPM is a powerful tool, because of the high image resolution (e.g., atomic force microscopy, AFM) and its measurement of the electron emission current (e.g., scanning tunneling microscopy, STM). AFM is more useful than STM for imaging because it can map dielectric surfaces where STM cannot. The focus of our work is to locate the high emission source and specify the emitter (Fig. 3). Measurements are done in dry nitrogen-gas ambient because of the ease of sample exchange. Obviously, improvements in breakdown standoff threshold would need to be confirmed in vacuum, ultimately with rf applied fields. In present research work, the emission currents from copper and gold surfaces were measured by conductive-AFM in contact mode with a Pt/Ir-coated silicon tip.

Reports, as far back as 1961, presented breakdown measurements on metal surfaces coated with thin dielectric

materials, showing a large increase in standoff potential compared to uncoated⁴ (**Opydo**). We prepared samples with Al₂O₃ as the coating, to repeat this work at the micro-scale. Another intriguing historical observation is the role of surface hardness in raising standoff, for example by ion-implantation hardening (**Xu et al**). We hardened copper samples by Hf-ion implantation and by Hf or W metal-coating. Finally, to investigate the role of work-function on standoff, we varied the work-function of our metal surfaces by using low (Mg), medium (Cu) and high (Pt) work-function base metals under our dielectric coatings. The complete sample list is 1) 150 nm Au film on cleaved mica, 2) mechanically-polished (250 nm diamond powder) OFE Cu, 3) Kou Enerugi Kasokuki Kenkyukiko (KEK) single-point diamond-machined Cu, 4) 2.5, 5.0, and 7.5 nm Al₂O₃ films deposited onto 20 nm Pt and 20 nm Mg films on Cu substrates prepared as in 2), 5) 50 nm Hf and W films deposited on Cu, and finally Hf-ion implantation into a Cu surface. The FE results show typical a F-N behavior³⁰ for electron emission from the surfaces. However, some difficulties were found in the experimental data with using the conductive-AFM technique for FE current measurements. The metal-coated tips were found to wear rapidly during image scanning and measuring current such that the conductive layer on the very end of the tip becomes insulating after minimal use.

In 1995, O'Shea's group reported³¹⁻³³ several problems with using the conductive-AFM measurement technique for FE. Those problems have been found to seriously affect our experimental results as well.

2. Theoretical approaches and emission models

2.1 Current-Voltage relationships

A variety of mechanisms can govern electrical conduction through dielectric layers on material surfaces. The current-voltage relationships are:

1) Schottky emission	$I \sim \exp(\alpha V^{1/2}/kT),$	... (1)
2) Fowler-Nordheim injection	$I \sim V^2 \exp(-\alpha/V),$	... (2)
3) space charge limited	$I \sim V(\text{low fields}),$	... (3)
current (SCLC)	$I \sim V^n (n>1, \text{high fields}),$	... (4)
4) space charge limited current		
with Poole-Frenkel effect	$I \sim V^2 \exp(\alpha V^{1/2}/kT),$	... (5)
5) Poole-Frenkel conduction	$I \sim V \sinh(\alpha V^{1/2}/kT),$	... (6)
6) Poole-Frenkel conduction with		
overlap of Coulombic potential (Hill's law)	$I \sim \sinh(\alpha V/kT).$	... (7)

Generally, there are the two basic models for a carrier injection at a metal-insulator interface, Schottky emission (the injection over a potential barrier corresponding to the energy difference between the conduction

band of insulator and the Fermi level of the metal)³⁴ and the Fowler-Nordheim (F-N) injection (tunneling through a potential barrier).³⁰ The F-N model will be discussed in detail later on.

The importance of SCLC in somewhat thicker insulating films on surfaces has also been recognized³⁵. The SCLC can be fitted with a function of the form $I = aV + bV^n$. The ohmic component dominates at low electric field ($E < 2 \times 10^6$ V/m), while the V^n term dominates for electric fields higher than about 2×10^6 V/m. The V^n dependence has been ascribed to the SCLC effect. If SCLC is really involved, then the magnitude of the current implies the existence of a band-tail with a high density of states increasing exponentially towards the valence band. The very high density of band-tail states could be explained with the SCLC provided that the conduction occurred in highly-disordered regions, for example, at the grain boundaries in polycrystalline materials, although such band-tails with high densities of states are also found in amorphous materials³⁶. More recently, the grain boundaries have been claimed to be the dominant conduction path in polycrystalline films.³⁷

Poole-Frenkel (PF) conduction³⁸ is a bulk process, which relies on there being a significant number of defect or impurity sites within the body of the insulator. The charge carriers reside on these defects sites and, if the sites are sufficiently close together, the wave functions of the charge carries may overlap and allow the carriers to “hop” from one site to another. However, if the defect sites are too far apart, the hopping mechanism cannot keep up with the applied electric field.

Hill-type conduction involves discrete energy levels E_i (at E_i from the conduction band) whose overlap of Coulombic potentials is sufficient to allow the hopping of carriers from site to site by effectively lowering the barrier. This hopping mechanism, which displays non-linearity at high electric fields, is basically Hill’s law.³⁹

Now, in order to obtain the characteristic effective parameters of electron emission from a surface, e.g., the work function, field enhancement factor, and emission area, we consider the F-N equation, which is the most commonly used model for ejection of electrons from a surface, when discussing field emission.

(a) Fowler-Nordheim emission phenomena³⁰

In the theory of electron emission phenomena, in addition to the electron gas model of conduction electrons and the effective potential model of surfaces, is made the additional physical assumption that electrons inside the surface remain essentially at equilibrium in spite of those that are escaping from the surface. Then the current of electrons is obtained by integrating over all the electron energies, and the equilibrium flux of electrons incident on the surface is given by multiplying this current by the probability that an electron penetrates the barrier. The transmission coefficient is calculated from a quantum mechanical discussion (Fig. 4) of the electron incident on the barrier. Corresponding to the electron energy connection with the potential, the time-independent Schrodinger equation for the motion of one of electrons is easily carried out through a parabolic WKB-type

approximation.⁴⁰ So, the integration over all the accessible energies of the product of the charge on an electron, the number per second per unit area incident on the barrier, and the penetration probability, yields the total electric current per unit area $j(F, T, \zeta)$,

$$\begin{aligned}
 j(F, T, \zeta) &= e \int_{-W_a}^{\infty} D(F, W) N(T, \zeta, W) dW \\
 &= \frac{4\pi m k T e}{h^3} \int_{-W_a}^{W_i} \frac{\ln\{1 + \exp[-(W - \zeta)/kT]\}}{1 + \exp[(4/3)\sqrt{2}(F\hbar^4/m^2 e^5)^{-1/4} y^{-3/2} v(y)]} dW \\
 &+ \frac{4\pi m k T e}{h^3} \int_{W_i}^{\infty} \ln\{1 + \exp[-(W - \zeta)/kT]\} dW.
 \end{aligned} \tag{8}$$

where D is the probability of penetration through the barrier, NdW is the number of electrons with x -part of their energy within dW incident on the surface cross section per second per unit area, W is the energy for the motion normal to the surface, F is the applied external field, T is the absolute temperature, ζ is the Fermi energy, W_a is the effective constant potential energy, e is the charge of electron, m is the electron mass, k is Boltzman's constant, h is Planck's constant, $\hbar$ is $h/2\pi$, and

$$W_i = -\frac{1}{2}\sqrt{2}(e^3 F)^{1/2}, \tag{9}$$

$$y = (e^3 F)^{1/2} / |W|, \tag{10}$$

$$v(y) = 2^{-1/2} (1+a)^{1/2} \{ E[(2a)^{1/2}/(1+a)^{1/2}] - (1-a)K[(2a)^{1/2}/(1+a)^{1/2}] \}, \tag{11}$$

Here E and K are the complete elliptic integrals of the first and second kinds, and a is defined by

$$a = (1 - y^2)^{1/2}. \tag{12}$$

The extensions of Richardson-Schottky and Fowler-Nordheim theory have also been developed by Murphy and Good.⁴¹ The current density is

$$j = j(0) \left[\frac{\pi k T / D}{\sin(\pi k T / D)} \right], \tag{13}$$

where

$$D = \frac{\hbar e F}{2\sqrt{2m\phi}t(y)}. \quad \dots (14)$$

Here ϕ is the work function and

$$t(y) = v(y) - \frac{2}{3}y \frac{dv(y)}{dy}. \quad \dots (15)$$

$j(0)$ is the Fowler-Nordheim formula, is then obtained from Eqn. (8) as

$$j(0) = \frac{e^3 F^2}{8\pi\hbar\phi^2(y)} \exp\left[-\frac{4\sqrt{2m\phi^{\frac{3}{2}}}v(y)}{3\hbar e F}\right]. \quad \dots (16)$$

Numerically, $\left[\frac{\pi k T / D}{\sin(\pi k T / D)}\right]$ is found near unity for a metal at room temperature.

In semi-logarithmic form, Eq. (16) becomes,

$$\ln\left(\frac{I}{V^2}\right) = \ln\left(\frac{e^3 \beta^2 S}{8\pi\hbar\phi^2(y)}\right) - \frac{4\sqrt{2m\phi^{\frac{3}{2}}}v(y)}{3\hbar e \beta} \frac{1}{V}, \quad \dots (17)$$

where,

$$I = j(0)S, \quad \dots (18)$$

$$F = \beta V, \quad \dots (19)$$

where S is the effective local emitting area, β is defined as the field enhancement factor, and V is the applied voltage.

Having established the basics, it is possible to sketch out a method for determining the work function, effective local emitting area, and field enhancement factor, using the basic input data that are set up as the intercept, R ,

and slope, P , on the F-N graph, at a specific applied potential, V , and the corresponding current, I , at that voltage. SI units will be used in the present work except for energy, 'eV'. From Charbonnier's calculations with $t(y) = 1.044^{42}$, the F-N graph is seen to have the slope

$$P(\beta, \phi) = \left| \frac{d \left[\ln \left(\frac{I}{V^2} \right) \right]}{d \left(\frac{I}{V} \right)} \right| = \left| \frac{4\sqrt{2m\phi^{\frac{3}{2}}}}{3\hbar e\beta} s(y) \right|, \quad \dots (20)$$

where

$$s(y) = v(y) - V \frac{dv(y)}{dV}, \quad \dots (21)$$

and, for $s(y) = 0.956^{42}$ $P(\beta, \phi)$ yields

$$P(\beta, \phi) = 6.532 \times 10^9 \frac{\phi^{\frac{3}{2}}}{\beta}. \quad \dots (22)$$

The intercept $R(\beta, S, \phi)$ is obtained as

$$R(\beta, S, \phi) = \left[\ln \left(\frac{I}{V^2} \right) \right]_{V=\infty} = \ln \left(1.416 \times 10^{-6} \frac{\beta^2 S}{\phi} \right). \quad \dots (23)$$

A linearized F-N form is

$$\ln \left(\frac{I}{V^2} \right) = R(\beta, S, \phi) + P(\beta, \phi) \frac{1}{V}. \quad \dots (24)$$

If the F-N graph is plotted as a straight line of Eq. (24), giving a value for one of β , S , and ϕ , then the other two parameters can be calculated in straightforward manner. The field enhancement factor, β , is calculated from geometrical measurements of the surface.

(b) Geometrical field enhancement at the surface

Electrons are emitted from a few isolated microscopic sites having the form of metallic micro-protrusions (Fig. 10(a)) that can give rise to local electric field enhancement. We will consider in basic detail how such local field

enhancement can be related to the specific geometry of the micro-protrusion. Field emission of an electron from a surface in a vacuum at an inter-electrode field ($\sim 10^8 \text{ Vm}^{-1}$) can only be explained by the occurrence of a locally-low work function or a locally-high field intensification site. A combination of these is also possible, but observation of suitably-localized sites surfaces shows that they are sharp micro-protrusions or whiskers, approximately conical.⁴³ In addition, it is possible that such whiskers can be grown under the action of a high electric field. It has also been noted that cylindrical emitters (Fig. 5) behave quite differently at high current density.⁴⁴ Therefore, the two configuration micro-protrusion models considered here are a truncated cone and cylinder with having a hemispherical top.⁴⁵ The basic geometries, with the assumption that the micro-protrusions have an idealized symmetrical geometry, such as semi-ellipsoidal microstructure orientated normal to a planar electrode, are shown in Fig. 5. A relevant parameter in the theory is the field enhancement factor, β' , defined as the ratio of the true electrical field F to the mean field $F' = v/d$, where v is the voltage applied across the electrode gap d . The magnitude of β' depends on the geometry and dimensions of the micro-protrusion. In the calculations of β' for a truncated cone from its dimensions, it is possible to obtain a solution of sufficient accuracy by comparison with similar shapes having a calculable β' . Two such shapes are the spheroidal and cylindrical micro-protrusions, for which β' can be calculated.⁴⁶ The spheroidal micro-protrusion, illustrated in Fig. 5, consists of a sphere on the tip of an infinitesimally thin wire.⁴⁵ In the range of β' values ($20 < \beta' < 300$), Chatterton gives the approximate representation as⁴⁴

$$\beta' = 0.5 \frac{h}{r} + 5, \quad \dots (25)$$

where h is the height of the protrusion and r is the radius of curvature of its tip. The expression for the cylindrical micro-protrusion is given by Vibrans as⁴⁵

$$\beta' = \frac{h}{r} + 2. \quad \dots (26)$$

The β' -factor is also given by a more detailed expression⁴⁷

$$\beta' = \frac{(\lambda^2 - 1)^{1.5}}{\lambda \ln \left[\lambda + (\lambda^2 - 1)^{1/2} \right] - (\lambda^2 - 1)^{1/2}}, \quad \dots (27)$$

where $\lambda = h/b$ is the ratio of the semi-major to semi-minor axes of the ellipse in Fig. 5. For needlelike geometries, for example $\lambda \gg 10$, the above expression can be approximated by

$$\beta' \approx \frac{\lambda^2}{\ln \lambda - 0.3}. \quad \dots (28)$$

Alternatively, β' may be expressed in this form⁴⁸

$$\beta' = \frac{2l^3}{(1-l^2) \ln \left(\frac{1+l}{1-l} \right) - 2l}, \quad \dots (29)$$

where $l = [1-(b/h)^2]^{1/2}$ is the eccentricity of the ellipse. Other idealized micro-protrusion geometries have been analyzed and particular models,⁴⁷ based on a spheroidal emitting surface, are summarized in Fig. 6.

The cactus-like emitter shape may be thought of as a number of tiny spines with a height of h_2 and sharpness of r_2 on the micro-protrusion (Fig. 7 and 10(a)). The minimum separation, s , between the spines is assumed to be equal to h_2 because, if $s < h_2$, then a screening effect will take place and decrease the effective height of the spine. Such a two-step field enhancement mechanism on a surface can be considerable in this case. The electric field on a blunt protrusion is equal to

$$F_1 = (h_1/r_1)F_0 = (h_1/r_1)(v/d), \quad \dots (30)$$

where F_0 is the macroscopic mean electric field. Then, the electric field on the very end of macro-protrusion can be written as

$$F_2 = (h_2/r_2)F_1 = (h_1 h_2 / r_1 r_2)(v/d). \quad \dots (31)$$

Furthermore, the field enhancement factor dealing with a nano-scale feature has been reported recently.⁴⁹ Theoretical difficulties for a field enhancement factor are known to arise in the context of extremely sharp tips, which have radii $r < 10$ nm, because the F-N theory has built-in the assumption that the theory is predicated on a planar or semi-infinite medium emitting surface. An additional conceptual difficulty with such an extremely sharp tip, for the field-enhancement approach, is that generally the electric field is dealt with as a constant over the entire emission surface. The principal difficulty of utilizing the field enhancement concept in the F-N analysis is that β is often conjoined with the work function, ϕ , the estimation of which is complicated by nano-scale structures and the presence of adsorbates, such as chemisorbed oxygen, in the localized surface area. Zuber's group has proposed a theoretical model to deal with such difficulties.⁴⁹ The model relies on an exact and fully three-dimensional treatment for an electric field in a micro-tip/base geometry expressed in the

prolate-spheroidal coordinate system,⁵⁰ the solution of which is obtained for several electrostatic quantities due to the presence of a free charge in the tip/base junction. Fig. 8(a) shows a tip modeled as a constant-radius structure by the dotted line and a tip modeled as a hyperboloidal surface by a solid line. In Fig. 8(b), a transmission electron micrograph (TEM) of a fabricated silicon microtip is reproduced, with an overlay of the two models. The prolate-spheroidal coordinate system shown in Fig. 9 is defined by the orthogonal coordinate parameters ξ , η , and φ . Other parameters in this geometry include the foci distance of $2a$, the distance d between the tip and surface, the tip half-angle θ , and the apex radius on axis as defined by $r = a \sin \theta \tan \theta$. These parameters are related by the expressions as $\cos \theta = d/a$ and $\cos^2 \theta = d/(d + r)$.

In order to obtain the electric field F , Laplace's equation $\nabla^2 V = 0$ is solved with the boundary condition that the microtip is held at ground whereas the anode potential is fixed at V_0 . In the prolate-spheroidal coordinate system, the Laplacian is given by

$$\nabla^2 = \frac{1}{a^2(\xi^2 - \eta^2)} \left\{ \frac{\partial}{\partial \xi} \left[(\xi^2 - 1) \frac{\partial}{\partial \xi} \right] + \frac{\partial}{\partial \eta} \left[(1 - \eta^2) \frac{\partial}{\partial \eta} \right] + \frac{(\xi^2 - \eta^2)}{(\xi^2 - 1)(1 - \eta^2)} \frac{\partial^2}{\partial \varphi^2} \right\}. \quad \dots (32)$$

The voltage potential at the surface is independent of ξ and φ because of the cylindrical symmetry. The solution of Laplace's equation will be reduced to the Legendre equation in the coordinate η with a degree of $n = 0$ and boundary conditions of $V = 0$ at the tip and $V = V_0$ at the anode. The solution yields

$$V(\eta) = V_0 \frac{\ln \left[\frac{(1 + \eta)(1 - \eta_1)}{(1 - \eta)(1 + \eta_1)} \right]}{\ln \left[\frac{(1 + \eta_2)(1 - \eta_1)}{(1 - \eta_2)(1 + \eta_1)} \right]}, \quad \dots (33)$$

where η_1 corresponds to the hyperboloid surface associated with the tip, η_2 corresponds to the anode surface, and the range of η is $\eta \in [\eta_1, \eta_2]$. The electric field vector $F(\xi)$ at any point on the tip surface is perpendicular to the surface. The magnitude and direction for the microtip are determined from the negative gradient of potential V , and are given by

$$F(\xi) = -\text{grad}V(\eta) = \frac{V_0}{r} \frac{\tan \theta}{Q_0(\cos \theta) \sqrt{\xi^2 - \cos^2 \theta}}, \quad \dots (34)$$

where Q_0 is the Legendre polynomial of the second kind. Using this exact analytical solution for the electric field, the expression for the field enhancement factor β along the surface of the emitter frequently used in an

empirically based model of $F = \beta V$ is

$$\beta(\xi) = \frac{1}{a \sin \theta Q_0 (\cos \theta) \sqrt{\xi^2 - \cos^2 \theta}} \quad \dots (35)$$

When obtaining local surface topography, the field enhancement factor will be frequently predicted with the above formulas without complex numerical calculations.

2.2 Emission models

From a theoretical point of view, the highest possible electric field breakdown threshold requires that the electrodes have perfectly clean and microscopically-smooth surfaces. However, achieving such a perfect surface depends on many factors including, but not limited to, material fabrication and finishing, in-situ “conditioning” and the continued state of the vacuum environment itself. Ultra-clean preparation and surface treatment, borrowed from the semiconductor fabrication industry, have proven to be useful in obtaining the highest thresholds to date.^{51,52} Further progress requires the recognition that the metal electrode surface is really composed of peculiar kinds of microstructures of varied chemical composition. In the previous Section, we discussed the various geometrical field-enhancement factors. We now concentrate on connecting these physical ideas with the micro-irregularities found on technical surfaces.

Micro-protrusions generally appear as a result of surface treatments, operation of emission centers at the local sites, and/or from impact of fast microscopic particles on surfaces, etc. Dielectric inclusions result from abrasive surface treatment or from fabrication-embedded impurities.^{19,53} Oxide layers are formed within a rather short time following completion of surface treatment. Adsorbed layers of gases and vapors (especially, water and hydrocarbons) are held on the electrode surfaces by adsorption forces. The surface of an electrode is usually covered with chemically stable carbon-containing areas, that result in patch surface potentials. The surface also contains crystalline irregularities formed by impurities and defects, for example the segregation of dislocations and oxide inclusions to the grain boundaries in a polycrystalline material.^{16,54} In mechanical fabrication and chemical treatment, micro-cracks, pits and voids may appear on the surface. These fill with gas and change the characteristics of the surface. Finally, micro-emissions occur from foreign micro-particles (including dielectric ones), which reach the surface at any stage of the material’s history and are often difficult to remove. Thus, some features of the microstructures on electrode surfaces, which are regarded as plausible emission centers, can be listed as 1) geometrical micro-irregularities (micro-protrusions, whiskers, particles, craters, grooves, cracks, and so on) and, 2) electro-physical micro-irregularities related to the presence of foreign inclusions (interfaces of metal-dielectric, metal-semiconductor, and metal-insulator-metal types, etc.). See Fig. 10(a)-(g) for a summary.

More recently, several possible structures and models have been reported for field emission from electrically nano-structured heterogeneous (ENH) surfaces. These are generally described as ‘conducting particles’ within a less-conducting matrix layer which can act as a localized leaky dielectric (Fig. 10(h)).²⁰ Filamentary conducting channels can exist (or are formed by activation or conditioning) between the substrate and particles, between particles, and between a conducting particle in near-front surface and a vacuum (Fig. 10(i)).⁵⁵ Structures, which are ‘thick pin-like’, conducting cylindrical particles with a conducting channel that stretches from the substrate to the vacuum or protrude directly into the vacuum, are also possible. In addition to carbon-based films on surfaces, field emitting ENH materials are known to include (a) an oxide film and semiconducting inclusion on the surface,¹⁹ (b) a manufactured composite material in which is deliberately-dispersed as small conducting or semiconducting inclusions in resin, glass, or other dielectric,⁵⁶ (c) small conductive clusters embedded in a carbon matrix⁵⁷ and, (d) small carbon particles embedded in a betain matrix.²⁰ Consideration of the ENH hypothesis suggests that field-induced electron emission (FIE) from ENH materials occurs because there are conducting nano-structures inside the surface layer that cause local geometrical field enhancement to take place, either at the film/vacuum interface, or near this interface but inside the layer (Fig. 10(j)).^{58,59} As a result, the local field at the site of the nano-structure becomes sufficiently high for ordinary field electron emission to take place. In some cases, the emission electrons may be slightly hot in moving through the potential barrier and, in other cases, may assist in emission by other mechanisms.

Such compound nano-scale structures, which include defects, voids, dislocations, and strains of metal atoms and oxides, have been observed in the oxide and selvedge layer of chemically-treated titanium surfaces by scanning transmission electron microscopy (STEM) and high resolution transmission electron microscopy (HRTEM) in Fig. 11.⁶⁰ Fig. 11(a) shows a native oxide layer, mostly TiO_2 , covering a titanium single crystal surface. However, the oxide layer includes carbon contamination and the structure itself not a uniform and perfect insulator layer (Fig. 11(b)), and Fast Fourier transform (FFT) image analysis shows deflection spots from metallic titanium in the oxide layer. The FFT image analysis and the irregular surface structures are visible in Fig. 11(c).

The overall field enhancement is a combination of the macroscopic external field enhancement, resulting from superficial surface irregularities, plus the internal field enhancement below, but near the surface, a combination known as hybrid field enhancement. As an example of internal field enhancement (Fig. 10(j-1)) if, at the outermost surface, an emitter having a 5 nm height and 2 nm diameter sits on a floating conducting cluster of 5nm radius and 20 nm height from the interface, then the total field will be enhanced by a factor of 20 over that of a single 30 nm-tall conducting feature. In addition, because of external geometrical field enhancement of the surface emitter, the work function may also be reduced in this small sub-surface region, which further enhances the total emission. These sub-surface structures are rarely observed in the usual surface-sensitive

morphological investigations.

These ideas are not new in themselves, with the general idea of field emission from ENH materials derived from the work of Latham.¹⁹ Further, with respect to carbon films, several groups (in particular Refs. 61-63) have independently reported similar conclusions about similar field enhancement mechanisms.

The field enhancement factor, β , which might be conjoined with the local work function in such a nano-scale surface region, is now the important parameter hindering the development of accelerator technology at high accelerating gradients, about 0.1 GeV/m for NLC². As mentioned in Sec. 2.1, β can be numerically-calculated from the microscopic geometry of the protrusion or measured experimentally, and the validity of F-N emission established by data fitting with a suitable local work function and effective emission area. SPM measurement and F-N analysis are useful for the measurement of I-V characteristics from small emitters on a surface, making it possible to determine how much the microscopic electric field is increased, how much the local work function is decreased, or both. The purpose of the present work is focussed on finding the emission source, then to build on past work, correct some deficiencies, and synthesize relevant ideas into a broad picture applicable to FE generally.

3. Experiments

3.1 Sample preparation

Experimental samples included gold, copper, coated- and ion-implanted copper. The substrate non-annealed copper samples were prepared at SLAC used ASTM (American Society for Testing and Materials) Class 1 material with surface treatments as follows:

- Mechanical polishing

All copper samples, except the KEK diamond-machining, were mechanically polished using successively smaller grits of diamond paste, finishing at 1/4-micron, until the surface had a mean roughness of less than 50 nm (by AFM).

- Al₂O₃-film depositions

Some copper samples were electron beam-coated with a 20 nm Pt or Mg films and then e-beam-over-coated with dielectric Al₂O₃ films of varying thicknesses at a rate of about 0.1 nm/s in a background pressure of 9.3×10^{-4} Pa. The metal and Al₂O₃ film depositions were done sequentially, without venting the system between the metal and oxide depositions. The Al₂O₃ film thicknesses were 2.5, 5.0, and 7.5 nm.

- Hf- and W-film depositions⁶⁴⁻⁶⁶

The Hf and W films were formed on Cu surfaces by using a filtered vacuum arc deposition set-up with substrate pulse-biasing of about 2 kV applied during the early stages of deposition to ion-mix the interface into the substrate, for good adhesion. The repetitively-pulsed vacuum arc plasma gun was operated to form Hf and W plasma in pulses several milliseconds long at a rate of 1 pulse per second. The depositions were continued until the 50 nm thickness was built up. The vacuum arc has an inherent ion drift energy of 100 eV for Hf and W. Therefore, the deposition processes, even after the initial high ion mixing phase, are relatively energetic depositions. The vacuum pressures in the deposition chamber was a base of 1.3×10^{-4} Pa and an operating pressure of 4.0×10^{-4} Pa.

· Hf-ion implantation⁶⁷

The ion implantation into Cu was done using the LBL Mevva ion source-based metal ion implantation facility.⁶⁸⁻⁷⁰ The system base pressure was 1.3×10^{-4} Pa and the background pressure was 4.0×10^{-4} Pa during the operation. A vacuum arc ion source was used to form a broad beam of Hf ions at an acceleration energy of 100 keV and a pulse ion beam current density 5 mA/cm^2 at the target. The ion source was operated in a repetitively-pulsed mode with 250 microsecond pulses at a rate of several pulses per second, and the pulsed beam current density of 1 mA/cm^2 transform to a time-averaged Hf ion current density was $0.5 \mu \text{ A/cm}^2$. The total Hf-ion implantation dose was $5 \times 10^{16} \text{ cm}^{-2}$.

In addition to the SLAC-prepared copper substrates, a copper sample was prepared at KEK¹ as follows:

· KEK diamond machining

KEK diamond machining used for preparing the surface of Hitachi Densen Class 1 copper (Hitachi Cable. Ltd.). Stock was annealed in a vacuum at 500°C for hour; then diamond-turned with a natural single diamond-tipped cutting tool at 2800 rpm, $8 \mu \text{ m/rev}$ feed rate, $1 \mu \text{ m}$ cutting depth, 0° rake angle, 5° clearance angle, using kerosene lubricating fluid at 23°C in a class 1000 clean room. After machining, the samples were degreased by dipping in alcohol, then processed in an alcohol ultrasonic bath for 30 sec, followed by acetone ultrasonic for 30 sec and grade A nitrogen gas blow dry.

· Au(111) film on mica

This is our gold film reference sample (150 nm thickness) obtained from Molecular Imaging, Inc., Tempe, Arizona.

Fig. 12 shows scanning electron microscope images (RJ Lee Instruments Ltd., PSEM 75) for the Cu surface with diamond polishing and for a 2.5 nm Al_2O_3 film on a 10 nm Pt film on Cu. The AFM topographical images for each test sample will be shown in the Results Section.

3.2 Experimental setup

The experiments were performed using a commercial contact-AFM (Burleigh Instruments, Ltd. ARIS-3310) to measure morphologic images with a Pt/Ir-coated silicon probe (Digital Instruments, SCM-PIC, 13 kHz resonant frequency and 448 μm length, pictured in Fig. 13). In addition to its imaging capability, the conductive-AFM tip can measure the electrical behavior on the surface under the tip locally, with prior and subsequent mapping of the topography. The experimental set-up and schematic diagram are shown in Fig. 14. The sample is placed on a piezoelectric x-y-z scanner and is electrically-isolated. After the laser is focused onto the tip and centered in the position sensitive photodiode, tip and sample are brought into contact for normal Van der Waals-type AFM imaging. The imaging and electrical measurements were made in a dry nitrogen gas atmosphere during experiments.

In addition to the normal imaging mode, we can stop the image scan and measure local I-V curves by ramping the voltage and measuring the current flowing from sample to the positively-biased tip. The bias voltage scans were done under computer control. The voltage step size was 0.025 V/0.5 sec., and the current limit was set at 10 pA. It is important to limit the current through the tip to avoid irreversible damage. Therefore, software control resets the applied voltage to zero as soon as the limit current of 10 pA is reached. A 100G Ω ballast resistor also acts as an additional safeguard against excessively high current flow. SEM images were taken of the tip before and after each set of experiments.

The control software was created with LabView,⁷¹ because of the availability of pre-packaged libraries of application functions and developmental tools designed specifically for acquisition and instrument control, e.g., VXI, GPIB and serial instrument control, data analysis, data presentation, and data storage, and a multitasking system capable of running multiple execution threads and multiple virtual instruments (VIs) in parallel, etc.

It was important to protect the measurement system environment from vibration and electromagnetic interference (EMI). Isolation from building vibrations was accomplished by suspending the AFM module from long rubber cords. Residual modes from the piezo stage were passively-damped with a diamagnetic copper disk attached to the module base and suspended between the poles of a 6 kG magnet. For EMI shielding, a noise-cut transformer was used to electrically isolate the module and metal foil shields covered it (Fig. 15). The effects of the EMI shielding are displayed in Fig. 16.

4. Results

The as-received conductive AFM tips often have contaminated surfaces (Fig. 17),⁷² which produce unstable emission, current surges, and sparks (Figs. 18,19⁷³). In order to obtain proper data, all I-V measurements were

performed after a period of “cleaning” of the tip surface, which consists of ramping the tip voltage up and down repeatedly until reproducible emission is measured. The maximum current during the cleaning procedure was limited to less than 10 pA to prevent undesirable effects such as essentially catastrophic damage to either the tip or the sample.

Figures 20-31 show the I-V data (a) characteristic of electron emission from the sample surface, (b) the F-N plot of the data, and (c) the AFM topographic image of the sample surface being measured, except for Fig. 25 which has the (a) I-V curve and (b) topography. The data are displayed as an average value of, at least, two measurements. The F-N plots were fitted for the extended field emission region.⁷⁴

The data are shown for a 150 nm Au(111) film on a mica substrate (Fig. 20), ASTM Class 1 Cu mechanically polishing with diamond paste (Fig. 21), Cu surfaces with KEK diamond machining (Fig. 22), 20 nm Pt film on Cu both over-coated with 2.5 nm Al₂O₃ (Fig. 23), 5 nm Al₂O₃ (Fig. 24), and 7.5 nm Al₂O₃ (Fig. 25), 20 nm Mg on Cu both over-coated with 2.5 nm Al₂O₃ (Fig. 26), 5 nm Al₂O₃ (Fig. 27), and 7.5 nm Al₂O₃ (Fig. 28), 50 nm Hf on Cu (Fig. 29), 50 nm W on Cu (Fig. 30) and, finally, Hf ion-implanted into Cu (Fig 31).

The F-N model was found to provide the best fit to the data, except for the 7.5 nm Al₂O₃ film on 20 nm Pt on Cu because of excessive surface roughness, when compared to the other models of Eqns. 1-7. See Fig. 32 for data fits to each of the emission models. The F-N analysis could now be discussed further by adding in details for each sample involving local emission area, surface work function and field enhancement in the emitting region. However, problems arise with that approach, posed by the requirement of unrealistic values for these various parameters. The cause of these difficulties will be discussed in the next Section.

5. Problems with conductive-AFM measurements

Conductive-contact AFM measurement was used above to measure FE as an early stage of electrical breakdown, from metal and oxide surfaces in a nitrogen gas atmosphere. The experimental results exhibit the behavior of F-N field emission, but the data was difficult to correlate with realistic physical parameters of the emitting surface. In this section, these problems with some new approaches to their solutions are discussed.

In an ohmic conductive-contact AFM measurement on a metal surface, the data should display a linear I-V characteristic, even in weak repulsive contact with the surface (Fig. 33).⁷⁵ However, our conductive AFM measurements often show a no-current-flow region at low bias voltage values (Fig. 18). In these situations, the contact portion of the tip and/or the surface must be insulating.

There are two possible reasons in which the end of the tip can be insulating (predicated on the metal sample surface being clean and non-insulating, e.g., Au in Fig. 33). First, the conductive material may be worn off the tip (as in Fig. 33), revealing the semiconducting Si base below and, second, insulating materials (e.g., thick oxides, particulates, and contamination) may appear on the tip surface, forming a barrier to conduction between the tip and the sample surface. In both of the cases, the tip-surface junction appears as an illustration in Fig. 34 where the I-V measurement depends on the height of the insulating region and material properties of the junction between the conducting part of the tip and the metal surface. O'Shea's group has reported that for insulating-behavior metal-coated tips, the coating material is changed or missing on the very end of tip.³¹⁻³³

The metal tip-metal sample AFM contact is characterized by large adhesive and friction forces. The probable causes of conductive tip failure are mechanical wear, electrical damage and melting, or a combination of these mechanisms. Fig. 35 shows a scanning transmission electron microscope (STEM) image taken of a worn tip, confirming that the metal coating has been lost from the end of the tip. Evidently, the contact mode imaging damages the metal coating, making the tip unsatisfactory for further use for conductive-AFM. This problem of wear is a problem common to all metal-coated tips where the wear of the coating has been reported to be most severe when good electrical contact is established. Scanning any surface in ambient air contact mode, even at low loads, can lead to tip contamination (Fig. 36). Wear and contamination affect the adhesive forces acting between the tip and sample. Wear also occurs in other environments (e.g., UHV) but contamination is certain to be more pronounced in ambient atmosphere. In addition, the water meniscus which forms between the tip and the surface provides an alternate conduction path when the very end of the tip is worn and the metal-coated tip side is bridged to the surface by the meniscus. In summary, an insulating tip surface increases the tip impedance, independent of the local electrical properties of the sample surface, while oxidation of the sample surface produces repeatability problems.

Occasionally, it was observed that conductive behavior can be restored by applying voltage bias pulses between tip and sample (Fig. 18). This suggests that adsorbed contamination is removed, until a stage is reached where the pulsing can no longer improve the conductive behavior. At this point, the tip and surface are presumably clean. However, it is possible that the pulsing treatment can cause current-induced damage when the tip surface is initially covered with a "hard" (e.g., carbide vs. a more weakly-bound hydrocarbon) contamination. Further, if the tip is covered with a tenacious insulating layer, the application of a relatively large bias voltage is required to achieve an appreciable current. If the insulating material is then removed during the higher voltage biasing, the resistance will decrease dramatically and the tip can be damaged by the sudden increase in current. Another mode which can occur is conduction through surface contaminant that surrounds the tip, as mentioned above.

Metal coating is usually lost from the tip apex during the I-V measurements, resulting in a rapid decrease in the

current intensity at each successive measurement. The decreasing of current intensities is shown before and after I-V experiments in Fig. 37. Alternatively, the current density can be huge, even for small bias voltages, during a cleaning operation causing the metal coating to be melted or damaged at the contact point. This phenomenon, where the coating appears to have melted, is shown in Fig. 38 in a STEM image of a Pt/Ir-coated tip. Actually, the Burleigh AFM probe mount with a lateral size, parallel to the sample, of $5\text{mm} \times 5\text{mm}$ has a capacitance of 0.22 pF, giving a stored energy of 2.75×10^{-12} J. The discharge of this energy is quite enough to melt or damage the film of 20 nm thickness on the end of the tip (Fig 33).

When working in air or even in dry nitrogen gas ambient, there is always an uncertainty about the tip-sample distance (Fig. 34). Oxides and other film growth underneath the tip can be enhanced by the high electric field and current density used during an AFM I-V scan. Contamination (e.g., a hydrocarbon adsorbed layer) may also be present. Both of these conditions lead to change of the effective insulator thickness, leading to changes in the effective barrier height. Further complications may arise from the field distortion caused by charge trapping at surface defects, dislocations, or contamination in the interface region. The emitted current depends on both the applied and distorted field in the emitting region. One of the emission characteristics observed when working in air or dry nitrogen is that, while relative changes in current intensities are straightforward to observe, the details of the experimental condition in the emitting region make it difficult to convert the results to meaningful quantitative data.

In 1995, O'Shea's group reported³¹⁻³³ several problems with using the conductive-AFM measurement technique for FE. One of the major problems reported was electrical and mechanical degradation of the tip during measurement, i.e., the conductive properties of the tip changed during the course of the experiment. Drawing excessively high current densities led to destruction of the coating film on the tip while the mechanical wear or topographic scanning could be severe, even at zero applied bias. In general, the problems affecting the reliability of conductive-AFM measurements are oxidation, contamination, and wear of the tip. Oxidation of a tip can result in a gradual increase of the tip impedance, independent of the local electrical properties on the sample surface. Similarly, the oxidation of the sample itself introduces repeatability problems. Wear of the tip coating can result in a total loss of conductivity while, on the other hand, if an insulating oxide layer forms on the surface of the end of tip, the wear of this adventitious oxide can result in an increase of the tip conductivity over the course of experimentation. The contamination on a tip and sample can distort the repeatability of experimental data, while too thick a water meniscus that forms between the tip and sample surface in a air environment provides an alternate conduction path and leads to false topographic images. These problems can be very serious if the conducting material is completely worn off the tip apex.

The above mentioned problems and difficulties have been also observed by other groups,⁷⁶ who have analyzed in detail these effects in their data. Their analysis also shows that proper FE measurements require that the tip

must not change characteristics over the course of an experiment. Therefore, UHV conditions are essential if hygroscopic effects, hydrocarbons, and ionic contamination are to be either eliminated or systematically characterized. Unfortunately, tip wear is more serious in UHV because of the absence of a lubricating water layer, leading to an increase of adhesion between the now UHV-clean surfaces of tip and sample. Nevertheless, STM measurements in UHV have demonstrated that nano-scale all-metal (as opposed to metal-coated) tips can sustain much higher currents than those used in our conductive-AFM study, and it seems likely that mechanical stress arising from the large adhesive forces is, therefore, responsible for the removal of metal from the metal-coated tip apex.³³

Recently, new SPM techniques developed for the semiconductor industry now can measure the local surface potential or work function without adhesive contact with the sample, e.g., micro-Kelvin-probe microscopy (micro-SKP),⁷⁷ scanning Maxwell-stress microscopy (SMM),⁷⁸ scanning-capacitance microscopy (SCM),⁷⁹ and so on. Use of micro-SKP microscopy, combined with UHV, could be an effective probe for our FE work. The removal of surface contact removes the various tip-sample-ambient problems. More importantly, mapping the sample surface potential shows those areas of low potential that are likely to be good field-emitting locations. Returning to these areas for I-V scans and F-N plots should prove productive as we search for the intrinsic sites that lead to breakdown on metal surfaces, particularly Cu. SLAC has begun a cooperative research project, "Nano-scale Field Emission and Surface Potential Measurements on Metal Surfaces", with the National Institute for Material Science, in Japan,⁸⁰ which has a modified UHV STM combined with micro-SKP.

6. Conclusions

Since 1996 the Physical Electronics Group at SLAC has worked on high electric field breakdown from polished copper surfaces, with application to accelerator construction materials, using an AFM modified for F-N FE measurements.

The experiments performed so far measured emission currents with a applied electric field range of 0 to a few tenths of a V/nm from diamond-polished Cu, KEK diamond-machining Cu, Al₂O₃ films of 2.5, 5.0, and 7.5 nm thicknesses on 10 nm Pt and 20 nm Mg films on Cu, 50 nm Hf and W films on Cu, and finally, Hf-ions implanted into Cu, using a conductive-AFM technique in a dry nitrogen ambient. The F-N analysis has shown a good fit to F-N theory except for the 7.5 nm Al₂O₃ film on 10 nm Pt film on Cu (caused by surface roughness). However, serious problems, previously reported by O'Shea et al and other groups, have been encountered by us as well. Oxidation of tip and sample surfaces, contamination of both surfaces, tip wear, electrical damage and melting have compromised interpretation of the measurements. It appears that conductive-AFM is useful for comparing the FE behavior of surfaces to each other, but not for quantitative measurements.

Recently, several new electron-emission models for nano-structures have been proposed, and also several new measurement techniques have been developed to analyze surface electric characteristics on the sub-micron scale, in a semiconductor field picture in which the solid state or film breakdown field is in a range of a few V/nm. These ideas are applicable for metal-vacuum breakdown in our range of 0.1GV/m, and we will use a modified-STM in UHV with combined micro-SKP for the investigation, in a collaboration with the National Institute for Material Science in Japan on a project “Nano-scale Field Emission and Surface Potential Measurements on Metal Surfaces”.

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Fig. 1. Schematic illustration of an early stage of breakdown phenomena.

Fig. 2. Crater on a metal surface caused by a high voltage breakdown in vacuum.

Fig. 3. Cartoon of a field emission measurement using SPM.

Fig. 4. Schematic of the modification of the surface potential energy by an external electric field that permits electrons to tunnel through the narrowed barrier.

Fig. 5. Geometrical illustrations of protrusions.

Fig. 6. Field enhancement factor β vs. various micro-protrusion geometries.

Fig. 7. Two step field enhancement for electron emission from a protrusion shaped like a cactus.

Fig. 8. Schematic tip illustration and fabricated micro-tip TEM image. The dotted line in (a) shows the tip model as a constant-radius structure (like an idealized floating-sphere model), and the solid line shows a tip model as hyperboloidal surface. In (b), a TEM image of a silicon micro-tip is overlaid with the prolate-spheroidal and constant radius tip geometries of (a).

Fig. 9. Prolate-spheroidal coordinate system.

Fig. 10. Possible micro-emission sources are depicted. (a) shows micro-protrusions or whiskers.

Fig. 10(b). Metal-insulator-vacuum emitting insulating particle in (b) (from Ref. 19).

Fig. 10 (c). Metal-insulator-metal-insulator-vacuum (MIMIV)- type emission. Schematic shows field- induced electron emission via a two stage switch-on mechanism associated with substrate-resin-graphite-resin-vacuum (from Ref. 53).

Fig. 10 (d). MIM electron emission associated with metal particle and conductive channel (from Ref. 19).

Fig 10 (e). Oxide and contamination surfaces.

Fig 10 (f). Electron emission from grain
(from Ref. 16).

Fig. 10 (g). Electron emission from a conductive channel in a defective oxide (from Ref. 54)

Fig. 10 (h). Field emission from nanostructures, She's model (from Ref. 20)

Fig. 10 (I). Field emission from nano-structures, Robertson's model (from Ref. 55).

Fig. 10 (j). Field emission models for conducting nano-structuresl,
 (a) Latham model (from Ref. 58) and
 (b) Forbes model (from Ref. 59).

Fig. 11. TEM images of a Ti single crystal with a chemical polishing. (a) Scanning TEM image and XPS analysis on surface, (b) HRTEM image in circled part of Ti sample, and (c) HRTEM image and FFT analysis.

Fig. 12. SEM images of Cu and Al₂O₃ deposition samples. (a) Cu surface with diamond polishing. (b) Al₂O₃ deposited on a Pt film on a Cu substrate.

Fig. 13. SEM image of Pt/Ir-coated Si AFM probe.

Fig. 14. Shielded AFM in (a) and schematic diagram of measurements in (b).

(a)

(b)

Fig. 15. AFM vibration and electromagnetic isolation systems. (a) Rubber cords and EMI shield. (b) Magnetic damping system under AFM module.

Fig. 16. External electric noise. Solid line is background signal without EMI shield and circles show measurement of background signals with EMI shield.

Fig. 17. TEM images of carbon fibers and contamination on tip surfaces (from Ref. 70). (a) The arrangement of fibers at the end of tip. (b) A protrusion at the end of tip (from Ref. 72).

Fig. 18. Irregular spike-like electron emission during cleaning operations.

Fig. 19. HRTEM shadow images of W tip covered with Ga-In-Sn liquid alloy. (a) Tip voltage $V = 0$ V, (b) $V = -2.5$ kV, (c) $V = -3$ kV (from Ref. 73).

(a)

(b)

(c)

Fig. 20. Characteristics of electron emission and AFM topographic image of Au(111) film on mica. (a) The I-V curve, (b) the FN plot, and (c) the topography.

Fig. 21. Characteristics of electron emission and AFM topographic image of Cu sample with diamond mechanical polishing. (a) The I-V curve, (b) the FN plot, and (c) the topography.

Fig. 22. Characteristics of electron emission and AFM topographic image of KEK single-crystal diamond-machined sample. (a) The I-V curve, (b) the FN plot, and (c) the topography.

(a)

(b)

(c)

Fig. 23. Characteristics of electron emission and AFM topographic image of 2.5 nm Al_2O_3 film deposition on Cu coated with 20 nm Pt film. (a) The I-V curve, (b) the FN plot, and (c) the topography.

(a)

(b)

(c)

Fig. 24. Characteristics of electron emission and AFM topographic image of 5.0 nm Al_2O_3 film deposition on Cu coated with 20 nm Pt film. (a) The I-V curve, (b) the FN plot, and (c) the topography.

(a)

(b)

Fig. 25. Characteristics of electron emission and AFM topographic image of 7.5 nm Al_2O_3 film deposition on Cu coated with 20 nm Pt film. (a) The I-V curve and (b) the topography.

Fig. 26. Characteristics of electron emission and AFM topographic image of 2.5 nm Al_2O_3 film deposition on Cu coated with 20 nm Mg film. (a) The I-V curve, (b) the FN plot, and (c) the topography.

Fig. 27. Characteristics of electron emission and AFM topographic image of 5.0 nm Al_2O_3 film deposition on Cu coated with 20 nm Mg film. (a) The I-V curve, (b) the FN plot, and (c) the topography.

(a)

(b)

(c)

Fig. 28. Characteristics of electron emission and AFM topographic image of 7.5 nm Al_2O_3 film deposition on Cu coated with 20 nm Mg film. (a) The I-V curve, (b) the FN plot, and (c) the topography.

(a)

(b)

(c)

Fig. 29. Characteristics of electron emission and AFM topographic image of 50 nm Hf film deposited on Cu. (a) The I-V curve, (b) the FN plot, and (c) the topography.

Fig. 30. Characteristics of electron emission and AFM topographic image of 50 nm W film deposition on Cu. (a) The I-V curve, (b) the FN plot, and (c) the topography.

Fig. 31. Characteristics of electron emission and AFM topographic image of Hf implantation onto Cu with a total dose of $5 \times 10^{16} \text{ cm}^{-2}$ at 100 keV. (a) The I-V curve, (b) the FN plot, and (c) the topography.

Fig. 32. Comparison of current-voltage relations with best fit curves as abscissa and ordinate for 7.5 nm Al_2O_3 deposition on 20 nm Mg film on Cu. (a) Schottky emission. (b) FN plot. (c) SCLC high field. (d) SCLC with Poole-Frenkel. (e)

Fig. 33. Linear I-V characteristic of Au film on mica with Pt/Ir coated tip by AFM contact mode.

Fig. 34. Schematic illustration showing the wear of the conductive tip apex during measurements.

Fig. 35. STEM backscatter image of a conductive AFM tip apex after scanning (Photograph from Ref. 33).

Fig. 36. SEM image of contamination on the end of Pt/Ir-coated Si AFM tip.

(a)

(b)

Fig. 37. Difference between electron emissions from 150 nm Au (111) film on mica before and after measurements. (a) I-V curves and (b) FN plots.