

In Situ Observation of Field-Induced Nanoprotrusion Growth on a Carbon-Coated Tungsten Nanotip

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Nanoprotrusion (NP) on metal surface and its inevitable contamination layer under high electric field is often considered as the primary precursor that leads to vacuum breakdown, which plays an extremely detrimental effect for high energy physics equipment and many other devices. Yet, the NP growth has never been experimentally observed. Here, we conduct field emission (FE) measurements along with *in situ* transmission electron microscopy (TEM) imaging of an amorphous-carbon (*a*-C) coated tungsten nanotip at various nanoscale vacuum gap distances. We find that under certain conditions, the FE current-voltage (*I*-*V*) curves switch abruptly into an enhanced-current state, implying the growth of an NP. We then run field emission simulations, demonstrating that the temporary enhanced-current *I*-*V* is perfectly consistent with the hypothesis that a NP has grown at the apex of the tip. This hypothesis is also confirmed by the repeatable *in situ* observation of such a nanoprotrusion and its continued growth during successive FE measurements in TEM. We tentatively attribute this phenomenon to field-induced biased diffusion of surface *a*-C atoms, after performing a finite element analysis that excludes the alternative possibility of field-induced plastic deformation.

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The application of intense electric fields on a metal surface can cause vacuum breakdown (VBD), also known as vacuum arc [1]. This severely hinders the performance of several devices, including vacuum interrupters [2], x-ray sources [3], fusion reactors [4], and particle accelerators such as the existing Large Hadron Collider [5] and the next-generation Compact Linear Collider [6,7]. These issues cannot be mitigated yet, due to insufficient scientific understanding on the mechanisms that cause VBD. Generally, it is widely accepted [1,8–11] that the initial vapor and ion population that leads to plasma onset is produced by the atom evaporation due to extreme heating, induced by a localized field electron emission spot that enters a thermal runaway process [8,9,11–14]. However, for such a process to occur, an extremely high geometric field enhancement (of the order of several hundreds) by localized sharp protrusions on the metal surface needs to be assumed [15], which has not been observed experimentally especially for the metal surface after prior conditioning, so it can be suggested that these sharp protrusions on metal surfaces are created spontaneously under high electric field [16]. Experience from industrial production suggests that the diffusion of the adsorbates or contaminants such as carbon compounds, etc., on the metal surface may play a dominant role in the extremely high geometric field enhancement [buildup of nanoprotrusions (NP)] and subsequent electrical breakdown under high electric field despite being in the vacuum

environment, which can cause the cathode craters [17–21]. Yet, the exact mechanisms that lead to this phenomenon have not yet been understood, and its relevance to VBD condition has not been demonstrated.

In terms of the exact mechanisms of the appearance of field enhancing protrusions, several processes have been proposed, including plastic deformations due to high electric field-induced stress [22–24], field-induced biased surface diffusion [25]. Yet, none of them has been proved experimentally or described accurately in theory, due to the high complexity of the field-induced modifications of the interatomic interactions. Several experiments have demonstrated that the diffusion of surface adsorbates or contaminants under high electric field can lead to the buildup of nanoprotrusions (NPs) under high electric field during field emission (FE) [19,26,27]. This process, apart from the possibility to lead to the vacuum arc, can be used to achieve extremely high-performance electron emitters [28,29]. Yet, the exact mechanisms that lead to this phenomenon have not yet been understood, raising the need for *in situ* studies of NP growth under high electric field.

In this Letter, we study *in situ* the morphological evolution of a field emitting tungsten (W) nanotip coated with an amorphous carbon (*a*-C) layer during the field emission process. We measure the evolution of its field emission characteristics *in situ* a transmission electron microscope (TEM) and record the growth of an *a*-C

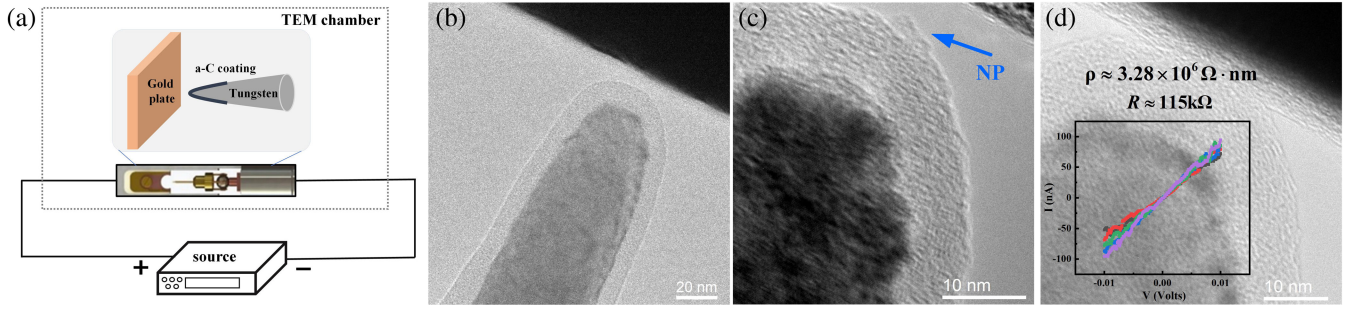


FIG. 1. (a) Schematic diagram of the *in situ* morphology characterization and field emission measurement system. (b) TEM image of the amorphous carbon-coated tungsten nanotip and the gold plate anode. (c) TEM images of *a*-C coated tungsten nanotip after the FE measurements in d_3 gap and the corresponding nanoprotrusion growth. (d) TEM image of the nanotip and anode in contact. Inset in (d): *I*-*V* curves during short circuit with the corresponding resistivity of the coating being $\sim 3.28 \times 10^6 \Omega \text{ nm}$.

nanoprotrusion, which is attributed to field-induced biased surface diffusion. This is the first *in situ* observation of field-induced NP growth on an *a*-C layer coated metal surface, demonstrating the possibility of such a process leading to protrusion growth and revealing the origin of enhanced field emission before vacuum arcs.

The *in situ* field emission experiments and corresponding electrode morphology recordings are carried out inside a JEOL-2010F TEM, where the pressure is on the order of 10^{-5} Pa, and the electrode gap is adjusted by an *in situ* electrically biased TEM holder (ZepTools Technology) as shown in the schematic of Fig. 1(a). The W tip was electrochemically etched down to ~ 20 nm radius in a NaOH/KOH solution [30]. Then an *a*-C thin layer was deposited onto the W nanotip by electron-beam-induced deposition. Figure 1(b) shows the TEM image of an as-prepared *a*-C coated W nanotip with about 29 nm radius. Figure S1 in Supplemental Material [31] presents the results of energy dispersive spectroscopy element analysis and electron diffraction (SAED) analysis for different selected areas. The energy dispersive spectroscopy result confirms that the tip core is W, and the shell is carbon.

A single crystal grain is observed at the W apex area based on the SAED result. Figure 1(d) shows the TEM image and *I*-*V* curves when the *a*-C coated tungsten nanotip is brought to contact with the gold-plate counterelectrode. The short circuit current shows a linear relationship as a function of the applied voltage, and the volumetric resistivity could be derived to be about $3.28 \times 10^6 \Omega \text{ nm}$, exhibiting graphitelike properties [32,33].

We took field emission *I*-*V* measurements for various gap distances d . Unfortunately, this particular setup does not allow for an accurate estimation of d from the TEM images, due to the high sensitivity of this measurement to even minute tilting of the anode plate (see Fig. S2 in Supplemental Material [31]). In several cases, we observed that the field emission process induced significant changes in the morphology and the emission behavior of the tip. Although the exact dynamic process of these morphological changes is extremely difficult to observe in real time by TEM, a careful analysis of the field emission behavior of the tip can give us significant insights.

Figure 2 presents four consecutive sets of *I*-*V* measurements, with various gap distances. We see that for d_1 and

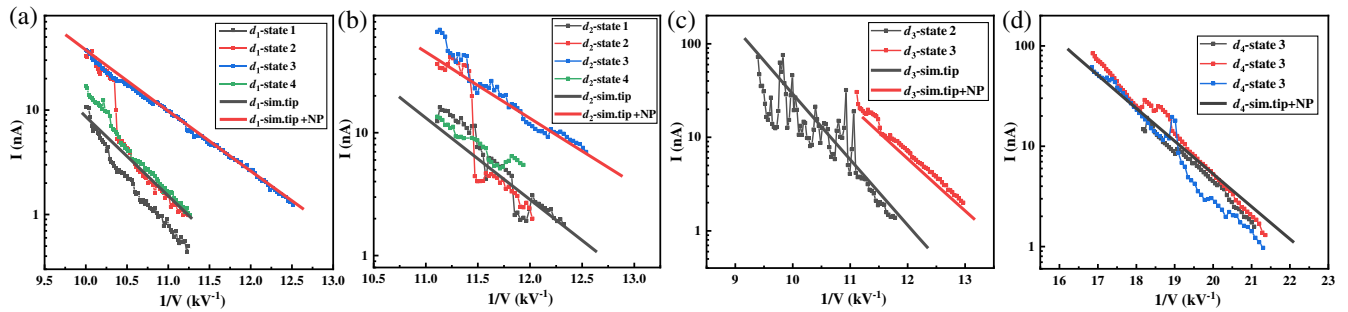


FIG. 2. Measured field emission *I*-*V* curves (dotted lines) from the *a*-C coated tungsten nanotip under different nanogaps. The corresponding simulated curves are given in solid lines, with the fitted gap distances found to be $d_1 = 50$ nm, $d_2 = 37$ nm, $d_3 = 41.5$ nm, and $d_4 = 17$ nm. The field emission current ($d_{1,2}$) exhibits four states: initial stable low current state 1, low-high current transitional state 2, stable high current state 3, and final stable low current state 4. In the d_3 case, the field emission current shows two states: low-high current transitional state 2 with an intensively fluctuating *I*-*V*, and stable high current state 3. In the d_4 case, only the stable high-current state 3 is observed, together with a permanent NP outgrowth visible in TEM.

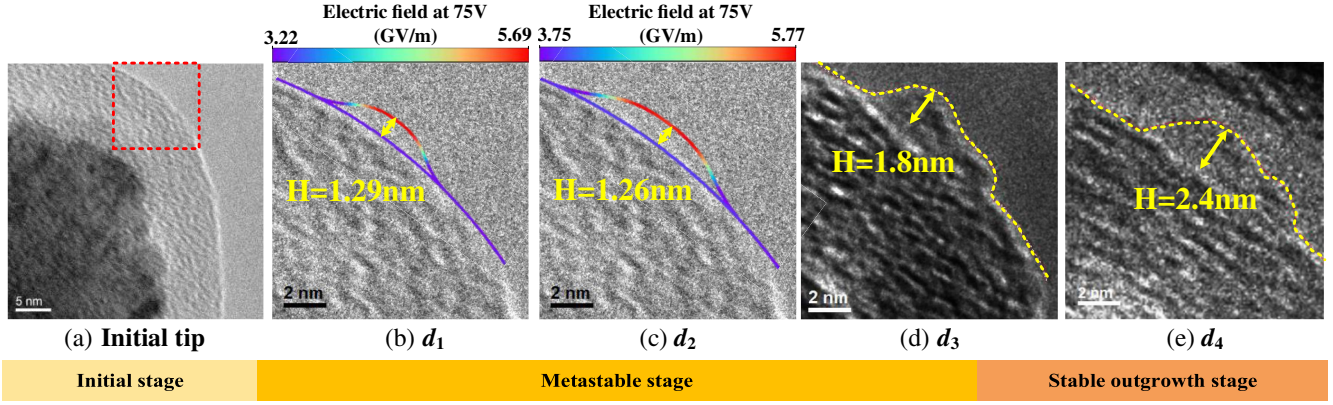


FIG. 3. Schematic representation of the tip evolution. (a) the initial nanotip before FE measurements. (b),(c) the simulated NP (colored lines) for $d_{1,2}$. The color coding gives the calculated surface electric field distribution, with the NP exhibiting a significant local field enhancement. (d),(e) the stable NP outgrowth visible in TEM after the FE experiments in d_3 and d_4 .

d_2 , the I - V curves exhibit a metastable behavior, evolving through four distinct states. In state 1, the tip emits a low current (I_{low}), following an approximately stable Fowler-Nordheim (FN) type curve. In the following state 2, the I - V starts with a similar characteristic at low voltages, but when the current reaches the 10 nA scale, it abruptly jumps to a significantly higher current ($I_{\text{high}} > 4I_{\text{low}}$). Then, in state 3, the I - V continues following the high current behavior, in a line consistent with the formation of an NP that slightly increases the local electric field at the tip. Finally, in state 4, the emission returns to I_{low} , following a similar characteristic as in state 1. Here we use state 4 instead of state 1 to illustrate the temporal evolution, indicating that the FE-enhancing features that cause the high-current state (probably an NP) can appear and disappear, which indicates a metastable process.

For the d_3 gap, the I - V curves have only two distinct states. One is similar to state 2, i.e., the current starts at a low level and then is enhanced up to four times at higher voltages, but in this case the I - V curve has significant fluctuations at higher voltages rather than jumping to a stable high-current state, indicating an extremely unstable α -C layer. The other is similar to the stable enhanced-current state 3. Then, with progressive FE measurements at a reduced nanogap d_4 , an NP was observed to be formed firmly near the tip apex, and only the stable high-current state 3 was obtained afterward.

To test our NP growth hypothesis as an explanation for the transitional FE behavior of Fig. 2, we simulated the FE process fully, using our multiphysics software FEMOCS [9,12,13], which is coupled with the general field emission calculation software GETELEC [34]. We simulated a static tip with a hemisphere-on-cone geometry, which matches the TEM images of the emitter. The tip radius was set to $R = 29$ nm and the shank cone full-aperture angle to 2° . The electric field is calculated around the tip by solving the Laplace equation using the finite element method (FEM). Then the electrostatic potential near the tip surface is passed

to GETELEC, which calculates the FE current density distribution around the tip and the corresponding total emitted current (see Refs. [9,34] for details), taking into account the local emitter curvature [35]. The work function of the emitter was set to the one of graphite, $\phi = 4.62$ eV [36]. The total current is multiplied by a fitted preexponential correction factor of 0.025, which accounts for various uncertainties in the field emission theory (e.g., barrier shape, tunneling probability, real band structure, etc.) [37].

For the measurements at $d_{1,2,3}$, we simulated two geometries. One with the regular smooth tip and one with a small NP at the apex, as illustrated in Figs. 3(b) and 3(c). For the measurements at d_4 , only the geometry with NP was simulated. The value of the anode-cathode distance was fitted for the smooth tip case to the experimental low-current curve, i.e., state 1, being found $d_1 = 50$ nm, $d_2 = 37$ nm, $d_3 = 41.5$ nm, $d_4 = 17$ nm. The resulting simulated I - V for are plotted together with the experimental ones in Fig. 2. We see that the curves without the NP are in good agreement with the low-current curves (states 1 and 4), while the one with the NP matches very well with the enhanced-current state 3. The height of the NP is chosen to fit the third I - V curve for state 3, being $H = 1.29$ nm for d_1 , and $H = 1.26$ nm for d_2 . The simulated NP shape for these cases is shown in Figs. 3(b) and 3(c). An important characteristic of the simulated I - V , that agrees with the experimental measurements, is that the one with the NP corresponds to a slightly higher field enhancement factor and a significantly lower effective emission area, obtained upon fitting the simulated curves to the standard Murphy-Good equation using GETELEC [34] and its on-line interface [38]. The obtained values are shown in Table I. Here the enhancement factor is defined as $\beta = Fd/V$, with F being the electric field entering the Murphy-Good equation. Furthermore, based on the simulated field enhancement factors, the maximum electric fields during the I - V measurements can be obtained, as Table I shows.

TABLE I. Extracted effective enhancement factors for the tip without NP (β_{tip}) and tip with NP (β_{NP}), and emission areas for the tip without NP (A_{tip}) and tip with NP (A_{NP}) from the simulated I - V curves of Fig. 2.

Gap distance (nm)	β_{tip}	β_{NP}	$A_{\text{tip}}(\text{nm}^2)$	$A_{\text{NP}}(\text{nm}^2)$
$d_1(50 \text{ nm})$	1.975	2.6	3743	237
$d_2(37 \text{ nm})$	1.67	2.1	2980	315
$d_3(41.5 \text{ nm})$	1.77	2.26	3305	266
$d_4(17 \text{ nm})$...	1.58	...	159

The height of the NP first observed experimentally is about 1.8 nm [Fig. 3(d)]. With further field emission at d_4 , the NP height increased slightly to 2.4 nm [Fig. 3(e)]. Our results indicate that the simulated NPs for $d_{1,2}$ are metastable, with a height of about 1.3 nm, thus they cannot be observed in TEM. The exact physical process of NP growth is not clear yet. However, we can assume its basic characteristics based on the observed FE dynamics. The I - V curves in state 2 exhibit a quick transition upon the application of high field that draws high FE current, while in other circumstances they transit to a relatively stable condition. This implies that the physical process that causes these transitions possibly involves the effects of high electric field on the surface (e.g., Maxwell stress and modification of the interatomic interactions [39]). Additionally, an important role can be attributed to the local heating due to the field emission current, caused by the Nottingham and Joule effects. Yet, the temperatures cannot reach near the melting point of a -C because this would probably induce an extremely rapid and violent thermal runaway process [9]. There are two known mechanisms that involve the above processes. One is plastic deformation due to the Maxwell stress and the second is surface diffusion, biased by the modification of the interatomic interactions due to the high electric field [25,40]. One could also consider deposition of adsorbents from the vacuum or from electron-induced degassing of the anode [20], but this mechanism was ruled out by additional experiments on clean W tips under the same setup that remained clean after a long stable emission (see the Fig. S7 in Supplemental Material [31]).

To test the validity of the plastic deformation hypothesis, we conducted FEM simulations utilizing our previously developed electrostatic-elastoplastic model [16,41] based on the constitutive modeling approach for continuum mechanics [42]. Continuum methods rely on the homogenization of the material behavior over large volumes. Although this is not an accurate approach if only a few atoms are involved, reliable order of magnitude estimations can be made [43]. Here, we consider the elastoplastic deformation of the a -C tip coating under the electric field-induced Maxwell stress. To parametrize the elastoplastic properties of a -C, we fitted them against the nanoindentation measurements of Ref. [44] and the simulation results of

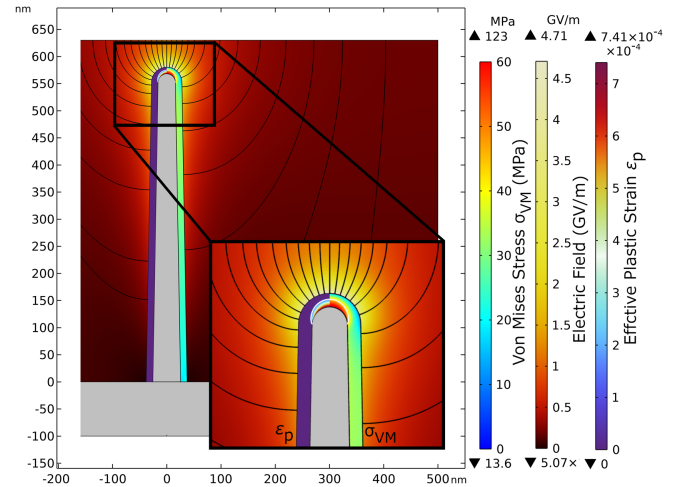


FIG. 4. Results of the electrostatic-elastoplastic model for the a -C coating under Maxwell stress.

Ref. [45]. The aim of our study was to evaluate if the Maxwell stress can cause yielding and plastic deformation consistent with the observed surface modifications.

The results of our model are summarized in Fig. 4. We first calculated the electric field distribution, which is shown in the “hot” color scale around the tip. The anode-cathode distance was set to $d_1 = 50$ nm and the applied voltage was ramped from 0 to 170 V to cover the theoretical range of 8 GV/m maximum local field on the tip, which is an upper limit for the field magnitude of our FE simulations. We then calculated the distribution of the total strain and stress in the coating layer at 100 V applied voltage, which exceeds the maximum of the experiments and corresponds to apex field of 4.71 GV/m. The stress distribution is depicted with the “rainbow” color scale, while the “jet” one gives the percentage of the plastic component of the total strain. We see that the contribution of the plastic deformation on the total strain is less than 0.001, with the stress being significant only at the inner part of the a -C layer, where the high-stiffness high-curvature W core acts as a stress concentrator. The apex area of tip becoming plastic (nonzero plastic strain) around $V \sim 150$ V or $F_{\text{max}} \sim 7$ GV/m, which far exceeds the voltage applied in our experiments. This means that the a -C coating is expected to behave almost entirely elastically within the range of stresses exerted to it by the electric fields involved in FE experiments. We repeated the same calculation for various distances (see Fig. S8 in Supplemental Material [31]), yielding similar results. The above results effectively exclude pure plastic deformation as the cause of at least the initiation of the NP growth we observed. It would be very hard to consider a plausible scenario by which the field-induced stresses within the FE range can cause significant plastic deformation to the a -C coating.

This implies that the most plausible mechanism behind the observed surface modifications is field-induced biased

surface diffusion. The high local electric field significantly modifies the migration barriers of surface atoms for any metal [39,40,46–48], causing the diffusion of surface atoms to be biased toward higher field values. This effect has been shown to induce significant modification on metal surfaces, although typically very high fields (beyond the FE range used here) and temperatures (close to melting) are involved [25,49,50]. However, it is plausible to assume that the atoms on the surface of the *a*-C coating are more loosely bound (especially π bonds) than those of a metal, leading to an increased mobility of the surface atoms that allows the biased surface diffusion to become significant at moderate temperatures.

The surface diffusion mechanism is also compatible with the observed instability of the NP growth. The FE dynamics of Fig. 2 imply that the NP grows under certain conditions when a significant FE current is drawn, but then flattens back to its original state after the FE experiment ceases, which is compatible with surface diffusion dynamics. When no field-induced bias is present on the surface, diffusion typically promotes the flattening of any asperity, as it seeks to minimize the surface energy and maximize the volume to surface ratio. However, upon application of high field and possibly moderate local heating caused by FE, the biased diffusion can induce NP growth due to the diffusion bias toward a positive field gradient [39,40,46].

This diffusion hypothesis cannot yet be confirmed by computational models. Moreover, it remains an open question what is the mechanism that stabilizes the NP after the measurements at d_3 , making it visible on the TEM image. We can hypothesize that under specific circumstances the atoms on the NP might take metastable configuration, needing to overcome a relatively high barrier to flatten out. Yet, we currently lack the tools to test this. Although previous attempts for kinetic Monte Carlo simulations that include the field effects have been made [25], their application to a non-metal surface such as *a*-C is very complex. Furthermore, even for simple metals, the exact modification of the interatomic interactions by the field is highly dependent on the local atomic environment, which further complicates their consideration in kinetic Monte Carlo simulations [51]. It is therefore essential and timely to develop such models, test the biased diffusion hypothesis and understand its dynamics quantitatively.

Furthermore, in addition to the observation of NP under *I*-*V* conditions (ramping voltage measurement) in this Letter, we have also observed the dynamic growth of NP during a constant voltage measurement in other experiments, which was accompanied by the enhanced field emission current and induced a following electrical breakdown (see the Figs. S3 to S6 in Supplemental Material [31]).

In conclusion, we demonstrated a nanoprotrusion growth on the amorphous carbon (*a*-C) coating layer of a tungsten nanotip during field emission, by *in situ* field emission experiments and accompanying field emission simulations.

We attribute the nanoprotrusion growth to field-induced biased surface diffusion, after excluding the possibility of field-induced plastic deformation by FEM simulations. This Letter provides the experimental confirmation of amorphous-carbon nanoprotrusion growth on an electrode surface exposed to high electric field, which is a plausible explanatory mechanism of the appearance of field enhancing features necessary to initiate electrical breakdown in vacuum.

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