1. Introduction

Field emitters are ubiquitous electron sources found in microscopy, medical, mass spectrometry, and high power high energy machines and systems [1–6]. However, utilization of field emission sources is a trade-off between utmost performance (highest current) and lifetime. At high output current densities, obtained at very high local electric fields, field emitter may rapidly fail to operate. Failures are often accompanied with vacuum arcs (also called vacuum breakdowns) when plasma forms in high and ultrahigh vacuum environment. During arcs, surface of the emitter irreversibly modifies with the net mass loss through volatilization. Vacuum breakdown is studied with highest attention at CERN and SLAC in the effort to increase the accelerating gradient, the accelerator figure of merit measured in MV per m [7], and to minimize the operational downtime of high gradient linear accelerators. Before going into operation, accelerating structures are conditioned for weeks, i.e. they are exposed to many millions of short exceptionally high power electromagnetic pulses to purposely destroy undesired field emission (dark current) centers. During conditioning, many hundreds of thousands of breakdowns take place leaving behind extensively dented and eroded surface [8]. In plasma-related disciplines, there is a common agreement that the field emission, commonly called cold emission, is a very complex phenomenon that can cause severe thermal load. Thermal load and material runaway could be the major factors causing vacuum diode deterioration, i.e. progressive increase in turn-on field, decrease in field enhancement factor, and eventual failure.

Keywords: glow and corona plasma discharges, field emission, plasma discharges in vacuum, carbon-based materials, amorphous semiconductors

(Some figures may appear in colour only in the online journal)
Kyritsakis et al [11] it was computationally found for a copper asperity that breakdown formation and plasma discharge are thermally activated via Nottingham heating/cooling and Joule heating mechanisms.

Accelerating structures are relatively large volume standing and traveling wave high vacuum devices in which breakdowns are extremely short lived (on the nanosecond timescale) and therefore are extremely hard to study. However, arcs and breakdowns are present not only in accelerator R&D but in many other disciplines [12–14], meaning that there are many other platforms that can provide insight into the vacuum breakdown problem. The vacuum diode, a configuration often utilized for electron source prototyping/testing, could be one of such platforms. Indeed, in recent years several reports appeared that discussed transition from field emission to stable long lasting plasma discharge regime in small volume microgap DC vacuum diodes [15, 16]. When such transition takes place, it was found that the current versus voltage relation switches from Fowler–Nordheim (FN) to DC gas breakdown/discharge dependence. The transition is also accompanied with visible light generation. It was proposed that the plasma formation was due to anode material evaporation, accumulation and ionization. This supports earlier findings on anodic plasma formation for sensitized carbon fibers cathodes operated in pulsed µs regime [17] but contradicts findings for accelerator cavities in which only cathodic plasma is possible (the entire inner surface of the cavity is the cathode). It also contradicts very recent results obtained for a high voltage µs pulse vacuum copper tip-copper plate diode [18]. Therefore, currently a second contradiction exists, whether the discharge is due to anodic or cathodic plasma formation.

In the present study we use a DC vacuum diode that makes use of nitrogen-incorporated highly conductive n-type ultra-nanocrystalline diamond (UNCD) cathode as a model platform to test the hypothesis of the hot cathodic breakdown. Unlike the common vacuum diode design that relies on sharp nanoscopic tips, convenience of using UNCD as a cathode material stems from that it has large emission area. Thus, electron emitting locations are easier to detect and characterize by simple means [19]. Another important advantage is that diamond appears to be a temperature sensitive material that has a diamond to graphite phase transition at 1500–2000 K. One more key point has to be emphasized—(N)UNCD is a two-phase material built of sp³ diamond grains lined with sp² grain boundaries. Rich sp² content in (N)UNCD enables low turn-on fields ~1 V µm⁻¹ which in turn allows for large cathode-anode gaps, 100–200 microns, which is enough to observe with ease processes that may evolve in the vacuum gap. By following this rationale, we were able to observe smooth and reversible transition from field emission to glow discharge formation in such nanodiamond vacuum DC diode. Our findings suggest that there is a cathodic plasma that forms due to thermal cathodic material runaway condition. Additional surface analysis with Raman spectroscopy revealed phase transformation from nanodiamond to graphon (N)UNCD and their laterally resolved emissivity. Electron emission micrographs (a)–(c) correspond to I₀/I₀G ratio 1.40 (red line), 1.20 (blue line) and 1.05 (black line), respectively.

2. Experimental details

(N)UNCD films were grown by microwave-assisted chemical vapor deposition system in a mixture of CH₄/Ar/N₂ (with small addition of H₂ for initial plasma ignition) on stainless steel cylindrical stubs 4.4 mm in diameter using a standard procedure which was established in our previous studies [6, 19].

2.1. Sample analysis. Correlation between sp² phase and emission characteristics

The films were analyzed and assessed using a combination of three microscopy and spectroscopy methods. The relative graphitic sp² content in the diamond sp³ matrix of the samples before and after field emission tests was evaluated by Raman spectroscopy by calculating the ratio between the intensities of the two major characteristic D and G peaks (labeled in figure 1). I_D/I_G—the smaller the ratio the larger the sp² content [20]. A Renishaw (probe laser 633 nm) spectrometer and a Horiba (probe laser 532 nm) spectrometer were used. Scanning electron microscopy was used to inspect surface of the samples.

Field emission properties of (N)UNCD were characterized and visualized using a field emission microscopy approach described in our previous work, see [19, 21]. In such experiments, field emission from planar (N)UNCD surface is laterally resolved by making use of complementary pair of imaging anode screens. YAG:Ce/Mo screen (yttrium aluminum garnet crystal doped with cerium and coated with molybdenum) [21] directly transfers the electron emission center distribution from the emitter surface through the gap, and ITO (semimetallic
An increase in the blue/white light emission in (a) can be related to micro-plasma formation near the cathode surface. The white circles in (a), (b) depict diamond-to-graphite ratios [23]. Extending this correlation in (b) was stable and reproducible during multiple ramp-up and ramp-down experiments. Therefore, this sample allowed for imaging with both YAG and ITO screens. Unlike CNT, (N) UNCD produced intense blue light. Most importantly, from the side by side comparison presented in figure 2, it is clear that the strongest electron emission locations imaged by YAG overlap precisely with blue light emission locations imaged by ITO. After a series of ramp-up/ramp-down experiments, the sample was examined ex situ by SEM. Extensive amount of circular dark spots was detected. The spot distribution across the surface, in turn, precisely overlapped with the YAG and ITO emission maps. To answer the question of the nature of the light generation, a few pictures were taken through a side view port to look into the gap between the cathode and anode, thanks to the large inter-electrode gap.

2.2. Formation of a plasma jet

The result shown in figure 2(d) was interpreted as an evidence for an early stage of plasma formation when plasma was confined to the cathode surface and laterally distributed around the strong electron emission locations, figures 2(a) and (b).

Field emission from the sample figure 1(b) ($I_D/I_G = 1.20$) was stable and reproducible during multiple ramp-up and ramp-down experiments. Therefore, this sample allowed for imaging with both YAG and ITO screens. Unlike CNT, (N) UNCD produced intense blue light. Most importantly, from the side by side comparison presented in figure 2, it is clear that the strongest electron emission locations imaged by YAG overlap precisely with blue light emission locations imaged by ITO. After a series of ramp-up/ramp-down experiments, the sample was examined ex situ by SEM. Extensive amount of circular dark spots was detected. The spot distribution across the surface, in turn, precisely overlapped with the YAG and ITO emission maps. To answer the question of the nature of the light generation, a few pictures were taken through a side view port to look into the gap between the cathode and anode, thanks to the large inter-electrode gap.

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vacuum, the discharge is self-formed and self-supported. Summarizing the results, unlike in previous studies [15, 16], our experimental evidence supports the cathodic plasma scenario. In this scenario, the cathode serves as a vehicle delivering atomized material for the discharge.

3. Discussion

The results discussed above call for pressure estimation inside the discharge. Since Paschen curves for atomized carbon discharges are not available, we referred to the book ‘Gas Discharge Physics’ by Raizer [28]. The book (see figures 7.2 and 7.3 on page 134) illustrates that mono-elemental or multi-elemental gases, well heavier than hydrogen, show similar discharge characteristics. By making use of those plots, and by knowing that the gap was set to 100 \( \mu \)m and that the discharge formed at 1kV, we find a rough estimate for the local pressure in the discharge zone as 10–1000 Torr.

Further, \( p–T \) diagrams [29–31] for carbon provide that the estimated pressure of 10–1000 Torr corresponds to a temperature of at least 3000–4000 K on the emitter surface. Indirect confirmation of extensive heating of the cathode comes from post mortem analyses of the samples. Multiple damaged locations (localized breakdowns), revealed by SEM in figure 5(a), consist of the re-melted metal substrate core surrounded by nanographite (labeled nanoG) which has a quite sharp transition into the rest of the cathode film that appears to be the pristine (N)UNCD film microscopically (SEM) and spectroscopically (Raman). We distinguish between nanodiamond and nanographite phases using micro-Raman. Diamond phase (\( sp^3 \)) contained in (N)UNCD completely converted into nanographite. A few minute conversion time implies fast kinetics, and therefore suggest a temperature \( \sim 2000 \) K (see chapter 13 in [20]). Micro-Raman measurements made inside the circular, thermally pre-damaged, spots on the surface of the sample figure 1(b) \( (I_D/I_G = 1.20) \) captures a state of nanodiamond transitioning into nanographite state—the recorded Raman spectrum suggests that the film state is neither of both but simultaneously contains highly separated nanographitic like D and G peaks (in the wrong ratio though) and nanodiamond \( \omega_1 \) peak near 1150 cm\(^{-1}\). Further, probably time resolved experiments would be needed to understand this non-trivial transition from nanodiamond to nanographite.

From analyzing the I–V curves in figure 3, the existence of visible light emission suggests thermal heating that may be dominated by Joule heating due to the diode-to-resistor
transition. At the same time, the extremely high temperature regime capable of evaporating graphite and diamond has to have an initial stage before the Joule heating comes into play. We believe that the precursor rise in temperature can be explained by the Nottingham effect [32]. According to analytical formulation, supported by the recent computational results [11], the inversion temperature should exist, that balances out the Joule effect and guarantees a stable emission within a certain $1 - E$ domain for classical emitters based on tungsten and molybdenum tip arrays. Another recent work [33] experimentally verified that the Nottingham effect dominates in large area field emitters, such as CNT fibers, and therefore should play the most crucial role in setting their operating point temperature. The inversion temperature is contingent on the local electric field $E_{loc}$, work function $\phi$ and electron mass $m$ as [32]

$$T_i = A \frac{E_{loc}}{\sqrt{m\phi}}.$$

By replacing the free electron mass in this definition with effective electron mass, which was found to be as small as $1/18 m_0$ from transport measurements [34], one can find that the inversion temperature is $4500 \text{K}$ which is close to the graphite vaporization/sublimation temperature. Thus Notingham heating, never inverting into cooling under our realistic conditions, and additional Joule heating drive the nanodiamond vacuum diode into thermal runaway regime. The use of the effective mass $1/18 m_0$ relies on extensive experimental evidence that field emission and photoemission take place from graphitic like grain boundaries or graphitic patches [23, 35, 36]. If this is the case, the effective electron mass in nanodiamond films is as small [34] as it is in graphite [37]. In comparison to UNCD, CNT materials are different. In CNT the effective mass is $1/2 m_0$ and the inversion temperature is then expected to be approximately $2000 \text{K}$—this is the maximal temperature reported in experimental literature [39]. Therefore, field emission is found not to be a cold process: CNT materials were found to be red hot [21, 39] and nanodiamond is found to be blue hot.

The observed plasma discharge stabilization for an extended period of time may be due to the fact that the surface temperature never reaches $4500 \text{K}$. A somewhat lower temperature is expected due to a cooling mechanism when heavy atoms carry away significant amount of energy, i.e. cool the surface. Radiation cooling should also play role, though its contribution is expected to be small.

Heats of vaporization $\Delta H$ for graphite and CNT, that are very similar $\approx 7 \text{eV}$ [38], effective masses of $1/18 m_0$ and $1/2 m_0$ respectively, and the Arrhenius formula relating evaporation rate $R$ and $\Delta H$ as $R \sim \exp(-\Delta H/kT)$ allow one to come up with a thermodynamical scenario of the lifetime difference between graphite-like and CNT emitters, i.e. enable an elementary interpretation of why some materials are stable during field emission and some are not. When exposed to the same elevated temperature, materials that have high enthalpy (or heat) of vaporization must have longer life time, as compared to materials that have low enthalpy (or heat) of vaporization, because the evaporation rate is exponentially slower. This can be seen through the relation $R \sim \exp(-\Delta H/kT)$. The same $R - \Delta H$ relation suggests—when having the same enthalpy of vaporization, lower temperature (1000–2000 K for field emitting CNT) must lead to longer lifetime as compared to field emitting nanodiamond that has temperature of 2000–4000 K. Therefore, graphite like emitters heating to 2000–4000 K are predicted to have a short lifetime and CNT emitters heating to 1000–2000 K are predicted to have longer lifetime while allowing for larger output current.

A very recent publication from the KEK, a high energy physics institute in Japan, [40] points out that the observations reported here may have broader implications. The work by KEK discusses intense light generation on the copper wall surfaces directly inside an operating large size 500 MHz megawatt RF cavity observed in situ, in real time. Most breakdowns/arcs were connected with instabilities or appearance and then disappearance of the light emitting centers. Optical spectroscopy revealed that the emission is black body radiation, that temperatures in those locations are between 1500 and 2000 K and that the temperature increased with the electric field. This is a confirmation of the Nottingham effect. This additionally confirms the hot cathodic scenario of the vacuum arc formation. The results of this study obtained using copper RF structures are in agreement with our conclusions obtained using a DC nanodiamond vacuum diode. This means that detailed studies of the physics behind vacuum breakdown/arc taking place in large-scale high-power systems can be accomplished using alternative platforms that enable different diagnostics and therefore additional insights into the problem.

![Figure 5. Raman spectra for nanographitic and UNCD sites on the damaged (a) and pre-damaged (b) cathodes due to electron field emission.](image)
4. Conclusion

We have conducted field emission microscopy of two $sp^2$ rich ultrananocrystalline diamond films. We found that a glow plasma discharge can be triggered as the $sp^2$ content exceeds 10 at.%. Put in short, electron emission self-induced heating through the Nottingham and Joule heating channels led to emitter material (carbon atoms) evaporation. The evaporated plume was further ionized by co-existing flux of electrons and formed microplasma discharges that were stable for a certain amount of time, enough to be observed and recorded. Such process could be stabilized by material evaporation that induces cooling. Our results are in agreement with previous black body light generation on material evaporation that induces cooling. Our results are observed and recorded. Such process could be stabilized by thermally driven cathodic plasma mechanism of the vacuum breakdown/arc.

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