FABRICATION AND CHARACTERIZATION OF Q-CARBON FIELD EMISSION DEVICE

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ABSTRACT

High-quality robust field emitting devices with stable emission over a long period of time are desirable for a wide range of applications. In this study, we focused on the design and fabrication processes, characterization, and field emission properties of large area Q-carbon (quenched carbon) composite thin films. The Fowler–Nordheim tunneling model is appropriate to explain the electron field emission (FE) mechanism. The microstructure and the morphology of those films were characterized by the Raman spectroscopy and the high-resolution scanning electron microscopy. The FE measurements on the Q-carbon cathode film show excellent field emission characteristics. The low turn-on field (as low as 2.4 V/μm), a high emission current density at a low applied electric field, and an excellent FE stability are the main features of the Q-carbon field emission devices. This work signifies that Q-carbon composite film can be used as an excellent emitter, which opens new possibilities for this novel material to be used in practical devices.

Keywords: Field emission device, Q-carbon, pulsed laser annealing, and thin film.

1. INTRODUCTION

The intriguing properties of Q-carbon (quenched carbon) arising from its unique structural, optical, magnetic, electronic, and mechanical properties have given rise to numerous fundamental and applied researches [1]. Promising applications ranging from superhard composite coatings, novel micro- and nano-electronic devices, atomic sensors and biomarkers, spintronic devices, nonvolatile memory, superconducting magnetic resonance imaging (MRI) and nuclear magnetic resonance (NMR) devices and field emission (FE) devices are fathomed after the discovery of this new phase of carbon [1–3]. Among these possibilities, the fabrication of practical FE devices is the most foreseeable one in the near future. Although there has been tremendous interest in cold cathode field emission from carbon materials ranging from diamond [4], diamond-like-carbon [5], vertically aligned carbon nanotubes [6], and amorphous carbon structures [7], electron emission from Q-carbon created immense interests in this field due to its outstanding structural, electronic, and mechanical properties coupled with the simple and large-

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scale fabrication capability on rigid to flexible substrates such as tungsten carbide, steel, sapphire, plastic, and glass [8,9].

In the last two decades, the design, realization, and application of a new generation of cold cathodes based on advanced carbon and noncarbon nanomaterials and thin films have been the field of tremendous interest by researchers active in all the areas related to electron emission. The investigation in this field ranges from the space applications to experiments in the field of high energy physics. The aim has been to find materials and systems characterized by the best FE performances in terms of low turn-on field, high density of the emitted current at a low applied field, robustness and long-term stability of the emission, and durability of the cold cathode material. In this regard, many efforts have been directed toward the issues of increasing the stability of the emission current density and other FE related issues. Opportunities to exploit knowledge gained from previous investigations on the emissions from carbon-based systems come primarily from the rapid progress in new application areas and from the need to overcome some technological barriers that other materials cannot solve, as, for example, the thermal degradation of carbon nanotubes and standalone graphene during emission or the effects of adsorbents on the surface of the emitters [10,11]. On this regard, the Q-carbon can play a leading role in resurging attention to carbon-based field emission technologies, due to its exceptional electron emitting properties enabled by good electrical and thermal conductivity, and thermal and chemical stability.

We hereby report the fabrication and investigation of electron field emission from Q-carbon composite films fabricated by pulsed laser deposition and subsequent pulsed laser annealing. This novel kind of field emitters displays good field-emission properties with low turn-on electric field and threshold field, large field-enhancement factor, and good emission stability and uniformity, which are well comparable or even better than those of its existing counterparts.

2. EXPERIMENTAL

Utilizing the nanosecond pulsed laser melting and subsequent quenching the Q-carbon is formed from the amorphous carbon thin films. Amorphous carbon films are deposited onto c-sapphire using pulsed laser deposition (PLD) in a stainless steel chamber at 1.0×10^{-6} Torr operating pressure (base pressure). We used a pulsed krypton fluoride (KrF) excimer laser (λ= 248 nm, repetition rate 5 Hz/s, pulse width 25 ns, laser fluence of~2 Jcm^{-2}) during the deposition. The as-deposited films were irradiated by an ArF laser pulse (laser wavelength = 193 nm, pulse duration = 20 ns) with an energy density in-between 0.6 and 0.7 Jcm^{-2} for conversion into Q-carbon. During the laser irradiation process, the as-deposited amorphous carbon film was melted in a super undercooled state and subsequently quenched to complete the whole process within 200–250 ns. The formation of Q-carbon requires higher undercooling than diamond, which has been controlled by the proper selection of the laser, substrate, and film variables. Thermal conductivities of the film and the substrate play a critical role in determining the degree of

undercooling and the quenching rate. Figure 1 depicts a schematic of the detail of the amorphous carbon to Q-carbon conversion by pulse laser annealing (PLA).

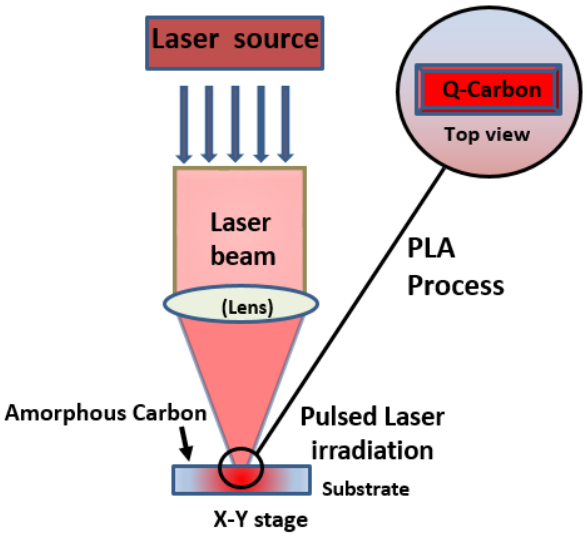

Figure 1: Schematic of the detail of the conversion of amorphous carbon into Q-carbon by PLA using a nanosecond excimer ArF laser.

The high-resolution scanning electron microscopy is carried out using the FEI Verios 460L SEM. A HORIBA Xplora PLUS confocal Raman microscope having 0.5 µm spatial resolution and 532 nm excitation source is used for determining the Raman active vibrational characteristics (at 300 K) of the Q-carbon. Raman spectroscopy provides a distinctive identification of Q-carbon and related materials.

The Q-carbon composite thin films were placed in a high vacuum system under a base pressure of 1 x10^{-7} Torr and the emission current versus anode voltage (*I-V*) characteristics were measured. A tungsten (W) plate was used for the anode electrode. For the FE measurements, the anode–cathode distance (d) was 100 µm and glass spacers were used to maintain the separation between the anode and the cathode. The schematic representation of field emission unit used in the present study is depicted in figure 2.

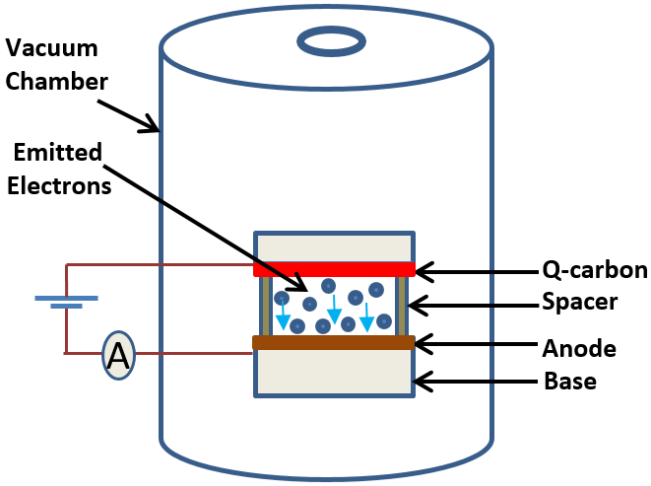

Figure 2: A simplified schematic of the FE measurement system used in this study.

3. RESULTS AND DISCUSSION

Figure 3 (a) shows the formation of Q-carbon composite structure on r-sapphire after a single laser pulse of ArF laser. The glowing regions in the SEM image are the Q-carbon and the rest of the dark regions are rich with amorphous carbonaceous entities. Figure 3 (b) illustrates the Raman spectrum from the Q-carbon structure and its inset shows the Raman spectrum of the amorphous carbon region. In the Raman spectrum of Q-carbon the characteristic D peak at 1345 cm^{-1} is a characteristic peak of carbonaceous structures and the G peak at 1570 cm^{-1} is also present. The fitting analysis of this peak gives an sp^3 percentage of around 84% and the rest of the carbon is sp^2 bonded. On the other hand the amorphous carbon region (the dark region in the SEM image) an sp^3 percentage of around 35% meaning that the rest of the sp^2 bonded carbon (65%) can provide enough free charge carrier to form a highly conductive zone. The Raman spectrum of the carbon film before the laser annealing gives a completely different sp^3-sp^2 bonded states. Therefore, Raman studies provide clear and consistent evidence for the conversion of amorphous carbon film into Q-carbon composite structure (due to laser-assisted melting and resolidification) after PLA treatment.

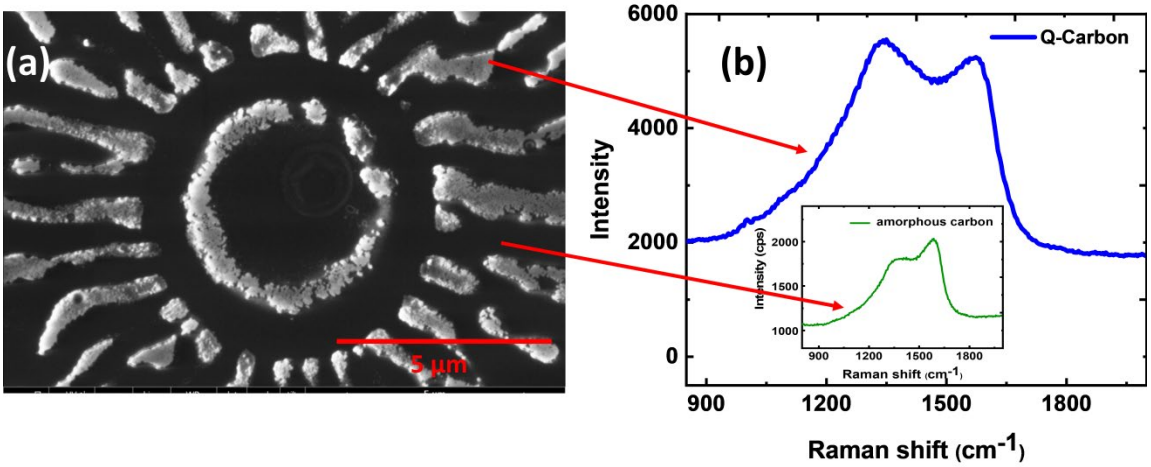

Figure 3: (a) SEM micrograph of Q-carbon composite film after PLA at 0.6 Jcm^{-2}, (b) Raman spectrum of Q-carbon. Inset of (b) shows the Raman spectrum of the amorphous carbon region.

The field emission (FE) current *vs.* applied field behavior of the Q-carbon composite is shown in figure 4. A low threshold field of 2.4 V/μm was observed for the field emission. The field emission was stable and reproducible upon cycling. The plateau in emission current was observed at an applied field of 2.67 V/μm which produces a current density of around 32 $\mu A/cm^2$. The Fowler-Nordheim (F–N) theory is the most widely used model for understanding the electron emission from a cathode surface under a strong applied field, and it has been also widely used to investigate the electron-emission behavior of various carbon nanostructures [12–

14]. To elucidate the electron-emission mechanism and extract the related parameters, we plotted ln (J/E^2) *vs.* 1/E, which yields a line in good agreement with the F-N equation. This plot is shown in the inset of figure 4. The straight lines indicate the quantum mechanical tunneling characteristic of field electron emission. The F-N theory for semiconductors can be described as following [15,16].

$$J = A(\frac{\beta^2 E^2}{\emptyset})\exp(-\frac{B\emptyset^{3/2}}{\beta E})\exp(-\frac{\Delta W^S - \Delta W^P}{2KT})$$

Where J is the emission current density, E is the applied electric field, b is the field enhancement factor, $\boldsymbol{\phi}$ is the work-function, A= 1.54 x 10^{-6} $AV^{-2}eV$ and B=6.83 X 10^9 $eV^{-3/2}$ Vm^{-1} [17]; β is the so-called field enhancement factor, which reflects the ability of an FE surface to enhance the local electric field at the emission spots as compared to the average macroscopic value, k is Boltzmann constant, T is the absolute temperature, ΔW^S is the increase of the surface potential barrier for sp^3-bonded nanostructures due to surface states, and ΔW^P is the decrease of the surface potential barrier owing to field penetration. Assuming that the work function of Q-carbon is 3.5 eV, the field-enhancement factor (β) of the Q-carbon composite cathode film is calculated to be 2900 from the constant F–N slope in the low-current region. This value is significantly higher than that of most of the carbon-based field emitters [18–20]. This large enhancement factor allows for sufficient tunneling of electrons from Q-carbon through barriers, which results in the low turn-on and threshold voltages.

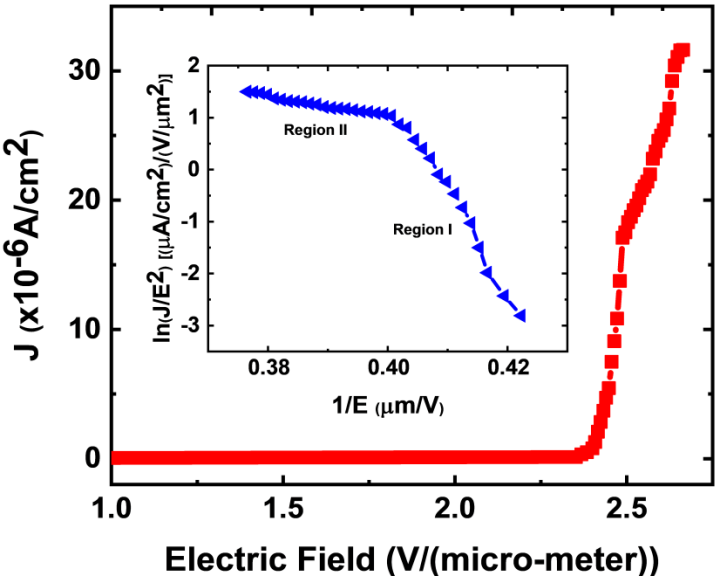

Figure 4: The electron-emission current density (J) as a function of the applied electric field (E) for the Q-carbon cathode. The inset shows corresponding F–N plot. The F-N plot represents a nonlinear behavior with two distinct slopes at high and low applied electric fields.

The FE features of the Q-carbon composite structures are attributable to strong field-enhancement factors, including the morphology of the sample, optimum sp^2 bonded carbon content in the sp^3 matrix, and the presence of diamond tetrahedra in the Q-carbon film. The FE characteristics shown by the Q-carbon has created the demand for a deeper understanding of the capabilities offered by nanostructured carbon-based films as an effective player for the fabrication of the state-of-the-art cold cathodes. Devices such as flat panel displays, lamps, gas discharge tubes, and x-ray and microwave generators, using Q-carbon as electron sources, can be demonstrated. The enhanced FE properties due to structural modification of the amorphous carbon film after PLA can be explicitly explained as follow. First, the Q-carbon is an excellent electron emitter with a work function Φ around 3.5 eV. The F-N equation illustrates that a low value of Φ helps attaining good emission current density. Second, the Q-carbon field emitter exhibit both a strong hardness combined with high flexibility and durability, which is of great interest for the stability of the emitter. In addition, a Q-carbon film without defects is expected to exhibit no chemically reactive sites, which infers an improved chemical stability and thus longer lifetimes compared to conventional field emission tips. The results on Q-carbon field emitters show that excellent current stability for hours can be obtained. Third, surrounding graphitic regions that make up the composite structure are excellent electrical conductors which can act as a free highway for the electrons during the FE process. Finally, the deposition of the Q-carbon films using the ultrafast PLA technique is simple yet scientifically profound. From the cost and scalability viewpoint the Q-carbon is very favorable for the industrial level production with the present set-up.

The Q-carbon exhibit stronger delocalization of carriers, indicative of smaller energy separation between the intraband states in the band gap. The emission current density from the measured FE characteristic is encouraging as the electron affinity shifts upward up to a few eV with increasing the film conductivity due to the presence of sp^2 carbon in the sp^3 bonded diamond tetrahedra in Q-carbon, thereby decreasing the effective potential barrier height for the emission. This is attributed to higher probabilities of electron injection into upper intraband levels during the transport process, originating from internal band bending and increasing band continuity [21]. The sp^2 clusters present within the sp^3 matrix help to raise the field enhancement in carbon-based films [22][23]. The mixture of sp^2 and sp^3 bonding in Q-carbon structure creates dielectric inhomogeneity [24]. Therefore, the field enhancement in the film is higher in Q-carbon composite structures. Additionally, the sp^2 carbon present within the sp^3 hybridized carbon matrix helps in a way that the energy levels of the former one located at or close to the Fermi level. These defect-induced bands near the Fermi level raise the level towards the conduction band and hence the work function decreases. Thus, combination of these two effects, (i) decrease in the work function and (ii) increase in the field enhancement factor due to the coexistence of sp^3-bonded carbon with the sp^2-bonded carbon at a definite ratio, result in improved FE characteristics from the Q-carbon composite film.

During the field emission property investigation of tetrahedral amorphous carbon, the sp^2/sp^3 ratio in the film was found to be one of the fundamental parameters to obtain a better understanding of its emission properties[25]. Although the sp^2 bonded carbon atoms help to raise the Fermi level towards the conduction band resulting in a low work function, however, it was found that the excessive amount of sp^2-bonded carbon atoms deteriorate the FE performance [25]. The optimum percentage of sp^2 bonded carbon atoms for enhanced field emission depends on the atomic features (cluster size and arrangement of the atoms) in the film, and these features depend upon the growth conditions and deposition routes. In the Q-carbon, which is formed by the liquid phase during non-equilibrium laser annealing treatment, the homogeneous mixture of the small sp^2 carbon with tetrahedrally bonded carbon has proven to be effective in attaining outstanding field-emission properties. Therefore, the turn-on field of the Q-carbon film is lower than most of the conventional carbon-based field emitters.

In the sp^2 rich carbon-based field emitter surfaces, such as CNTs and graphene, the carbon nano-structures sputter away due to heat accumulation arising from resistive heating, leading to degradation of the surface and inferior current stability [26,27]. The composite structure of Q-carbon and amorphous carbon hereby form a continuous structure where the Q-carbon clusters apparently act as heat sinks thus avoiding the heat accumulation on the cathode. They seem to rapidly eliminate a large amount of heat produced locally by the amorphous carbon during the emission process. In this way, the fluctuation in the emission current density from the Q-carbon cathode is minimized resulting in enhanced long term current stability and reproducibility. There are theoretical models in the literature that give support to the above explanation by taking into account the thermoelectric effect of phonon drag in a nanocarbon structure consisting of sp^2 and sp^3 carbon-rich regions [28,29]. In these models, the emission is considered to be due to processes occurring in the region where the electron affinity is negative and these regions are called as emission centers that are heated by the Ohmic current passing through the sp^2 rich channels (amorphous carbon region in the composite structure). The highly thermally conductive sp^3 rich Q-carbon region acts as a coolant to remove the heat from the sp^2 rich channels and make the FE operation more efficient. The present composite can be considered to be a working example of the above model. Although sp^2-bonded carbon provides tremendous advantages for the improved FE characteristics in carbon-based structures, however, a large amount of sp^2-carbon in the emitting structure is undesirable owing to the fact that the negative electron affinity is involved with the sp^3-content and an excessive amount of sp^2-carbon in the emission region is responsible for the increase in the electron affinity [30–32]. This is due to a graphitization of the emitting region at a high fraction of sp^2-carbon content.

Before the FE measurements, we heated the Q-carbon cathode to 300 C for 1 minute to remove the unwanted volatile/organic species from the surface. The room temperature FE current density was examined with time after this preliminary heat treatment. We monitored the current over a period of 500 minutes with an initial current of $30\mu A/cm^2$. Excellent emission stability was observed for the entire period of time (measured fluctuation less than ±4%). As an FE cathode

material, the Q-carbon composite sample exhibits better emission stability than single-walled-CNT (SWCNT) and multi-walled-CNT (MWCNT) films [33]. Additionally, no obvious degradation was observed in the sample after the FE measurements in the electron microscope. Thus the room temperature *I-V* characteristics of Q-carbon composite films were almost the same before and after field emission test. This also implies that no field induced damage occurred in the Q-carbon sample. The stable and persistent FE current from Q-carbon over a long period also supports the robustness nature of Q-carbon under a high applied electric field. Table 1 shows a comparison of the field emission characteristics, turn on field, field enhancement factor and the current stability of the present Q-carbon composite films with those of other carbon nanostructures including CNTs, nanocrystalline graphite, nanocrystalline diamond-carbon nanowall composite. It shows that the Q-carbon field emitter has excellent FE properties compared to other carbon-based materials.

Table 1 – Field enhancement factor, turn on field and current stability of various carbon nanostructures.

Carbon nanostructure	Field enhancement factor	Turn on field (V/μm)	Current stability (min)	Reference
CNTs	1.0×10^3–2.0×10^3	1.0–3.0	1000–1100	[34]
Nano crystalline graphite	3.0×10^2	9.0–12.0	260	[35]
Diamond-like carbon nanorods/TiO2/Ti	2.4×10^3	3.0	480	[36]
Nano crystalline diamond-Carbon	4.0×10^3	1.0	N/A	[37]
Diamond	1000	4.27	-	[38]
Q-carbon	2930	2.4	>500	Current study

4. CONCLUSION

A simple, low cost, and scalable method to deposit Q-carbon composite films for field emission have been demonstrated. This process of conversion of earth abundant inexpensive carbon into Q-carbon field emitter can be scaled by laser scanning, where 200 Hz laser can generate 200 cm^{-2} area per second. Field emission characteristics of Q-carbon cathode with NEA has been extensively discussed in this article. From the field emission measurements, the external electric field of about 2.4 V/μm was necessary to overcome the internal barrier for electron emission.

The electron microscopy and Raman spectroscopy reveal that the prime factor enhancing the FE properties of these films is the presence of the graphitic amorphous carbon channels along the thin film cathode and the ideal sp^3-sp^2 ratio in the Q-carbon structure which facilitates the transport of electrons through the films and reduce the barrier height for electrons to escape. This allows electron emission to occur at low threshold voltage, making Q-carbon an excellent candidate for field emission applications. The ability to deposit field emitting Q-carbon composite thin films by PLA process could allow large area deposition on inexpensive and flexible substrates which may open up exciting applications. This process can be scaled up to cover a large area, over 100 cm^2 per second, using 100 to 200 Hz and utilizing an automatic motor controlled synchronized system with the excimer laser operating system. The Q-carbon can be doped selectively with n- and p-type dopants with concentrations far higher than thermodynamic solubility limits, by incorporating dopants before melting, which could potentially generate more field emission current density at a low applied electric field by reducing the barrier height for electron emission. Overall, this discovery of the synthesis of Q-carbon at room temperature and atmospheric pressure and its outstanding FE characteristics will open a new frontier for the fabrication of carbon-based FE devices for a variety of applications ranging from FE electron sources and displays to energy efficient frictionless motors.

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