



Performance of field emission cathodes prepared from diamond nanoparticles



C.X. Zhai*, Z.Y. Zhang, L.L. Zhao, X.W. Wang, W. Zhao

School of Information Science and Technology, Northwest University, Xi'an 710127, Shaanxi, China

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ABSTRACT

Nano-diamond field emission cathodes were fabricated using a two-step technique. A mixture of nano-diamond and nano-Ti powders was coated onto a Ti substrate using a spin-coating process, followed by the application of an annealing treatment to form a TiC phase. The effects of the annealing temperature and the number of coating layers on the electron field emission properties of the as-fabricated field emission cathodes were investigated. The samples fabricated under different conditions were analyzed by Raman spectroscopy, X-ray diffraction, and scanning electron microscopy. The differences in terms of the electron field emission properties were explained by a TiC network model. A higher temperature is necessary to form a continuous TiC network when a thicker coating is used on the field emission cathode. In contrast, for the thinner coating, a relatively low temperature is sufficient to form such a TiC network. Only a continuous TiC network coating can facilitate the passage of electrons through the coating and lead to emission.

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1. Introduction

Electron field emission from carbon-related materials, such as diamond-like carbon, diamond, carbon nanotubes, and amorphous carbon (α -C) films, has been widely studied [1–8]. These carbon-based emitters have been reported to exhibit good electron field emission properties under a moderately weak electric field [9–11] because of their unique properties, such as their low work function and outstanding chemical inertness [12].

Diamond is a promising field emitter. Some efforts have been made to improve the emission properties of diamond films, with most work focusing on surface treatments [4,13], micropatterned structures [14, 15] and film doping [16,17]. However, detonated nano-diamond powder converted by carbon at high temperatures and high pressures [18] has been demonstrated to exhibit several special properties [19]. On the basis of these experimental results, we expect explosive detonated nano-diamond powders to exhibit interesting properties, including electron field emission. However, few reports on this topic have been published to date.

In this work, mixtures of nano-diamond powders with nano-Ti powders were coated onto Ti substrates using a spin-coating method, and field emission cathodes were fabricated using an annealing treatment. On the basis of the field emission properties of the samples and analyses

of the composition, structure and morphological characteristics of the samples, a structural model of a TiC network is hypothesized; the influence of the experimental parameters on the electron emission performance of the samples is illustrated by this model.

2. Experiments

Twenty milligrams of nano-diamond powder (unpurified, 3–10 nm, >97% purity, synthesized by the explosive detonation method of HeYuan ZhongLian Nanotechnology Co., Ltd.), 10 mg of nano-Ti powder, 10 ml of adhesive (a 10% solution of polyvinyl acetate in acetone), and 10 ml of active agent prepared by dissolving 6 mg of cetyltrimethylammonium bromide in 10 ml of acetone were added to 30 ml of acetone. The mixture was ultrasonically dispersed for approximately 60 min to form a suspension.

Ti plates (10 mm × 10 mm) were acquired from commercial sources and were mechanically polished, followed by being immersed in a mixture of acetone and carbon tetrachloride (1:1, v/v). The plates were subjected to an ultrasonic treatment to remove organic compounds from the surface and were subsequently rinsed with acetone.

Spin coating was carried out using a SC-1B spin-coater (ChuangShi Micro and Nano Technology Co., Ltd.). The substrate was placed on the spin-coater tray, and the suspension was applied as the substrate was spun at 500 rpm for 10 s; the substrate was then accelerated to 1500 rpm for 10 s. This procedure was repeated a various number of times (3, 9 or 15) to prepare coating layers with different thicknesses. Next, the samples were heated at 10^{-3} Pa for 10 min in a hot-filament chemical vapor deposition system.

* Corresponding author at: School of Information Science and Technology, Northwest University, No. 1 Xuefu Avenue, Chang'an District, Xi'an City 710127, Shaanxi Province, China.

E-mail address: cxzhai@nwu.edu.cn (C.X. Zhai).

Cold-cathode electron emission was measured in a vacuum chamber evacuated to a pressure of 3×10^{-4} Pa. An indium tin oxide plate was used as an anode to collect the emitted electrons. Macroscopic electric fields were applied between the flat anode and cathode in a vacuum diode configuration, which was separated by 200 μm using parallel glass plates.

The structure and morphology of the as-prepared field emission cathode samples were characterized by Raman spectroscopy (Renishaw inVia Raman spectrometer, the United Kingdom, operated at room temperature using the 514.5 nm line of an argon-ion laser), X-ray diffraction (XRD, Rigaku D/MAX-3C diffractometer, Japan, operated with $\text{CuK}\alpha$ radiation source at 35 kV and 35 mA) and scanning electron microscopy (SEM, Hitachi S570, Japan, operated at 15 kV).

3. Results and discussion

3.1. Field emission properties

The samples with the same number of coating layers (i.e., 15) were annealed in groups at 600 $^{\circ}\text{C}$, 700 $^{\circ}\text{C}$ or 800 $^{\circ}\text{C}$. These samples are identified as A, B and C, respectively. Curves representing the field emission current density as a function of the electric field (J–E) for these samples are plotted in Fig. 1. For comparison, a pure nano-diamond sample without any Ti powder was prepared, and the field emission curves from the sample annealed at 600 $^{\circ}\text{C}$ (labeled as A-WTi) are also presented in Fig. 1. The electric field was calculated by taking into account the applied voltage and the anode–cathode distance.

Samples A and A-WTi were annealed at the same temperature, and the properties of A were observed to be better than those of A-WTi. We deduced that the use of nano-Ti powder in the coating promotes the field emission properties of the sample. Although the difference between A and B is not readily apparent, in general, the field emission current density increases from A to B to C, whereas the turn-on electric field decreases.

The field emission characteristics of samples treated at 600 $^{\circ}\text{C}$ with a coating of 3, 9 or 15 layers (denoted as D, E and A, respectively) are shown in Fig. 2. As evident in the figure, the field emission current density decreases with increasing coating-layer thickness, whereas the turn-on electric field increases.

In Figs. 1 and 2, the Fowler–Nordheim (F–N) curves noticeably deviate from a straight line. The micro-arc discharge caused by the electron field emission leads to the exfoliation of some powder from the coating. As a result, the number of emission sites and the value of the emission current change; as a consequence, the shape of the F–N curves is affected.

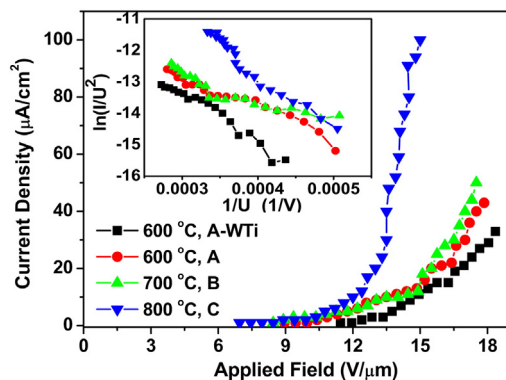


Fig. 1. The field emission J–E characteristics for samples with the same number of coating layers, annealed at different temperatures. The inset shows the corresponding F–N plots.

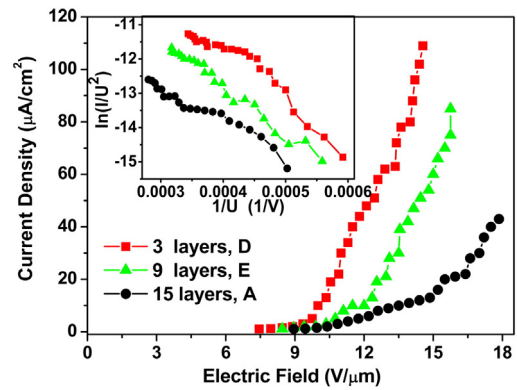


Fig. 2. The field emission J–E characteristics for samples with different numbers of coating layers, treated at 600 $^{\circ}\text{C}$.

3.2. Analysis of the samples

Raman spectrum of one sample was collected before being annealed, and the result is shown in Fig. 3(a). A Raman line is observed at 1327 cm^{-1} , which is shifted to lower frequency by approximately 5 cm^{-1} relative to the line in the Raman spectrum of the crystal. The Raman band at 1630 cm^{-1} suggests that the sample contained a small fraction of C=O bonds, likely because of the organic substance used in the coating. The Raman bands are asymmetric, tailing toward lower Raman shifts, with broad and weakly defined shapes. The simultaneous downshift and asymmetric broadening of a Raman line can be explained by a phonon-confinement model [20,21].

Fig. 3(b) shows the Raman spectra for the samples annealed at different temperatures. The first peak, which is located at approximately 1315 cm^{-1} , is assigned as the sp^3 structure. The peak at approximately

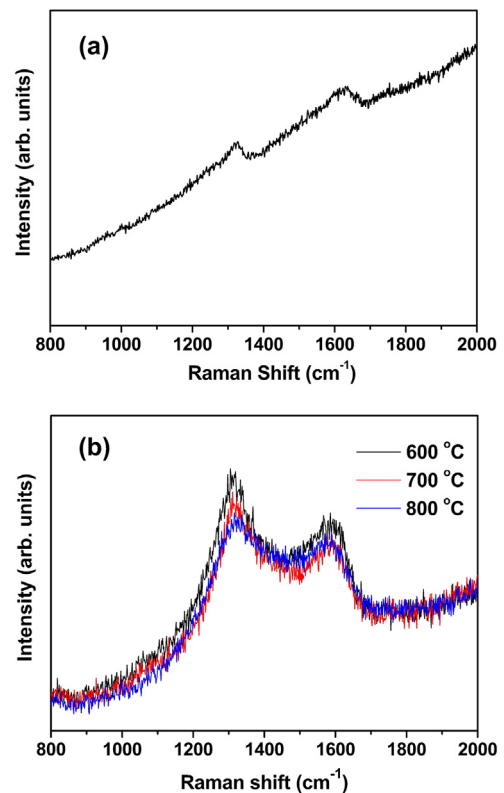


Fig. 3. Raman spectra of the samples (a) before being annealed and (b) after being annealed at different temperatures.

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