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HRTEM Studies of Morphological and Interfacial Changes of Nanodiamond in Field Emission Experiments

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In this paper we report on the morphological, structural and interfacial changes of small amounts of nanodiamond on the surfaces of ultra-sharp molybdenum needles as a result of the large electric fields and current densities experienced during field emission. The samples were prepared by electrochemically etching 125μm Mo wire in KOH to yield needles with radii less than 50nm at the tip. Diamond was then deposited on the tips by electrophoresis from a suspension nanodiamond in ethanol; the nanodiamond is shock-synthesized powder produced by explosive detonation, with an average crystallite size of ~5nm. The advantage of such samples is that they can serve as field emission sources and are *TEM-ready* (i.e. no additional sample preparation is required).

The as-deposited diamond is initially examined under high-resolution TEM as seen in Fig. 1. After initial examination in HRTEM, the sample is then used as a field emission source, where the current-voltage characteristics are measured in vacuum at 10^{-9} torr at a tip to anode distance of 1cm. Subsequent examination in HRTEM then permits direct observation of the morphological changes in the nanodiamond as well as any differences in the Moly/Diamond interface. Typical electric fields and current densities experienced under field emission can be in excess of 10^7 V/cm and 10^7 A/cm^2, respectively. The effects of such large electric fields and current densities can be seen in Fig. 1 and Fig. 2, where the morphology of the diamond before emission is compared to that after emission. It was observed that before emission the diamond particles were in clusters of several individual nanodiamond crystallites, which were loosely bound to the surface (actually moving under the influence of the beam). After emission, however, it can be seen that the clusters no longer remain, but instead more tightly bound individual crystals are present (one is seen in Fig. 2). As can be seen in Fig. 2, the interface properties between the nanodiamond particle and tip have changed: instead of loosely bound particles, a particle partially embedded into the tip body can be seen. Possible mechanisms of morphological and interfacial evolution of nanodiamond coatings under high current densities and high electric fields will be discussed.

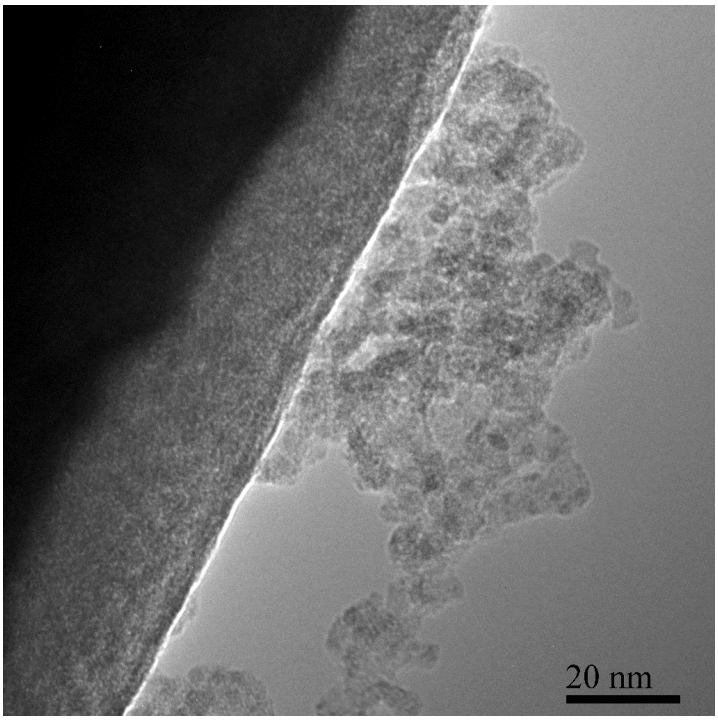

Figure 1 Loosely bound diamond particles before undergoing field emission.

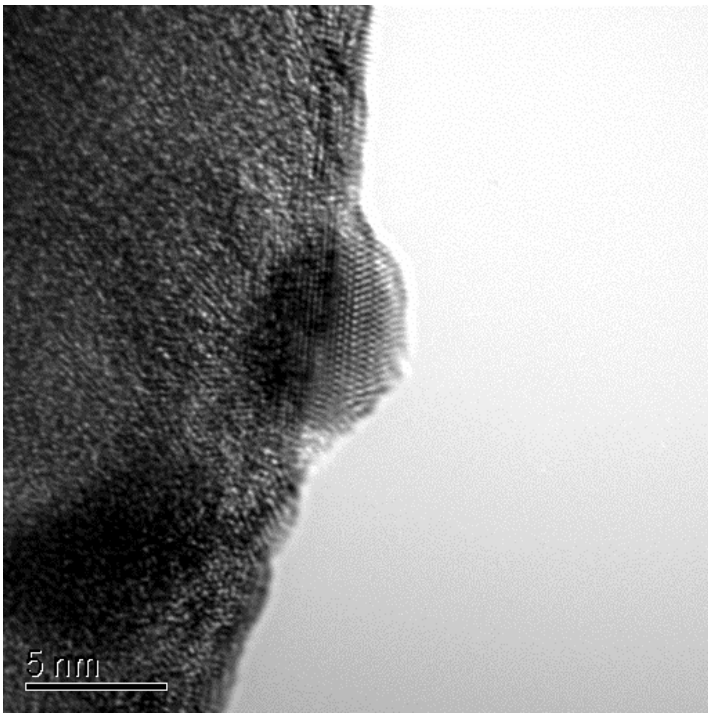

Figure 2 Tightly bound single nanoparticle after undergoing field emission

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