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FIB and TEM Studies of Diamond Films Prepared by Laser-Assisted Combustion Flame Deposition in Open Atmosphere

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Carbon thin films (diamond and related materials) are readily synthesized by various chemical (CVD) and physical (PVD) vapor deposition methods. These depositions are generally done in an inert atmosphere or in the presence of hydrogen. Recently, researchers have investigated the effects of adding oxygen to the processing atmosphere or depositing the films in open atmosphere [1, 2]. In collaboration with the University of Nebraska-Lincoln, diamond and related materials were produced by an open atmosphere laser-assisted combustion-flame CVD method [3]. Interfacial and defect studies of these thin films are of great importance in understanding the interplay of processing, structure, and physical properties. In this study, a FEI Helios 600 focused ion beam (FIB) dual beam instrument was utilized to prepare cross-sectional TEM specimens instead of conventional standard methods, i.e. slicing, polishing, dimpling and ion-milling. A 300 kV FEI Titan TEM with EDX and EELS was used for high resolution TEM imaging and nano-beam chemical analysis.

Films were deposited at a deposition rate of around 5 micrometers per hour on tungsten carbide (WC) and Si substrates. Fig. 1 (a) is a top-view SEM image of a polycrystalline diamond film on WC. The diamond grains are several microns in size. A typical lift-out cross-section TEM specimen welded on the top of a TEM copper grid is shown in Fig. 1(b). Fig. 1(c) and (d) are the cross-sectional morphologies of the polycrystalline diamond films on a single-crystal Si substrate and on a polycrystalline WC substrate, respectively. Smaller grains form initially at the bottom of diamond films while larger grains appear at the top. Defects and chemistry of diamond films were studied as well. Fig. 2 (a) is a high resolution TEM image of a diamond film on WC substrate showing defects such as twins and stacking faults in the film. The inset is the corresponding twin-related composite electron diffraction pattern (EDP) showing that the deposited films had a diamond structure. Fig. 2 (b) is a nano-beam electron energy loss spectrum (EELS) collected from a diamond grain which only shows an σ^* edge at 291 eV, which corresponds to the σ bonds in diamond. No π^* edge was found at 284 eV, which would correspond to the π bonds in graphite. The above results confirm that a laser assisted, open air combustion flame deposition process can result in polycrystalline diamond grain formation.

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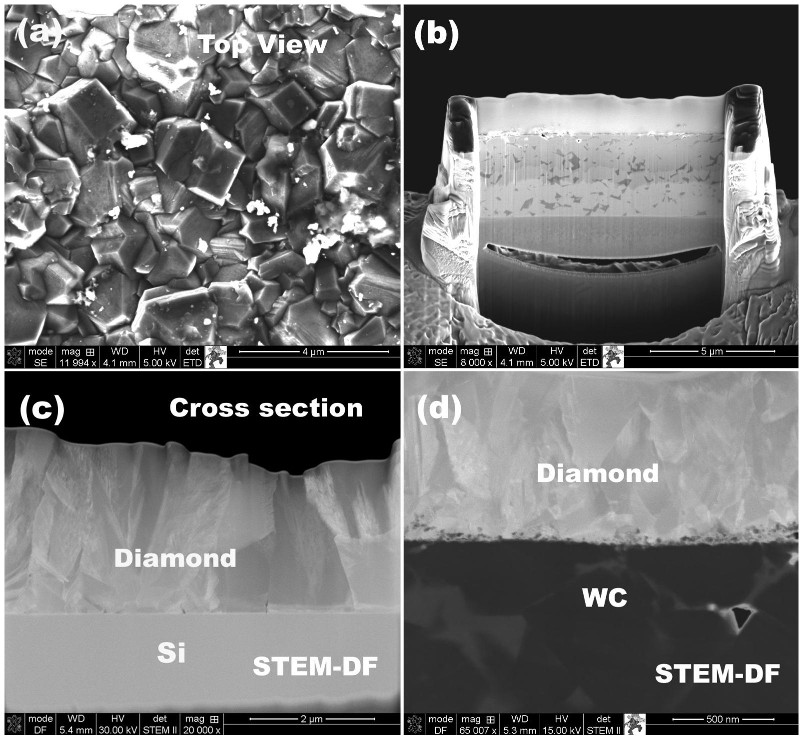

Figure 1 (a) Top-view SEM image of polycrystalline diamond film; (b) A lift-out TEM specimen welded on the top of a copper grid; (c) and (d) dark field STEM images of the diamond films on Si substrate and polycrystalline WC substrates, respectively.

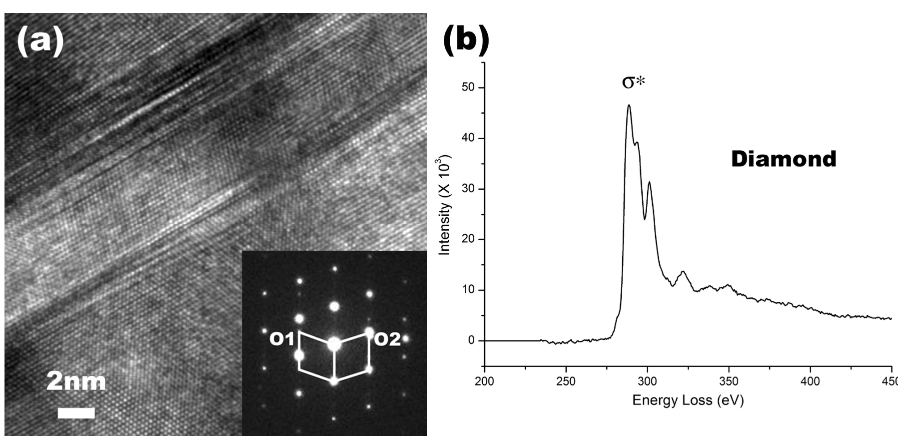

Figure 2 (a) HREM image showing defects in the diamond film. The inset is the corresponding twin-related composite EDP. (b) Nano-beam EELS spectrum only shows an σ* edge at 291 eV.

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