Manufacturing/Synthesis and Nanostructure of Superhard Thin Films/Materials

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The primary objectives of the research are to: (1) develop manufacturing processes for synthesizing novel superhard materials of controlled nano-structures and properties in the focussed composition region of the ternary (C-B-N) system, (2) develop/apply advanced techniques to characterize the nano-structures of these new superhard materials, and (3) relate the scale of the nano-structure (1-50 nm) and connectivity of the amorphous network to their unusual hardness and other properties. In addition, education and training of graduate and undergraduate students are also an essential objective of this project.

The rationale for these objectives is based on the reasoning that a large number of the technologically useful, unusual, and exciting superhard and superstrong materials such as diamond, carbon nanotubes, C_3N_4 and SiC are derived from carbon by covalent bonding of C to itself, and with other elements such as N, B, and Si. Recent advances in theoretical approaches to design unique materials such as the C_3N_4, and novel ways to synthesize metastable materials such as diamond and cubic-BN under typical laboratory conditions, and theoretically predicted superhard C_3N_4 have provided additional excitement for creating and synthesizing unique materials in the ternary C-B-N system.

To accomplish these goals of the NIRT program, a truly interdisciplinary and complementary team consisting of Profs. Singh, Boolchand, and Jackson from University of Cincinnati (UC) and their students/post docs have been working on this project.

The researches are aimed at developing manufacturing processes for thin films in the focussed region of the C-B-N system by ECR (Electron Cyclotron Resonance)-Microwave Plasma Enhanced CVD (ECR-MPECVD), and characterization of their structure/nanostructure by Raman, SEM, HRTEM, X-ray diffraction, and AFM techniques.

Two areas were emphasized during this year. One was aimed at developing in-situ capabilities for monitoring deposition conditions, and second was related to synthesis and characterization of nanocrystalline diamond thin films at low temperatures of <300°C. Towards the first objective, we installed a QMS (Qudrapole Mass Spectrometer) to monitor chemical species that are produced in the plasma environment during deposition of nanocrystalline diamond. Another in-situ optical diagnostic tool (an optical spectrometer) that can obtain spectral information from the plasma was also installed on the ECR-MPECVD system to identify the chemical species based on their spectral response. The QMS and optical spectrophotometer are interfaced with a PC to continuously obtain and record real time data from plasma environment during deposition. In addition to these facilities, facilities were created to introduce Boron precursors into the plasma environment for synthesizing C-B-N thin films.

Nanocrystalline diamond thin films were successfully deposited on Si and SiC single crystal wafers at a temperature of ~275°C. Diamond coating thickness ranging from 2.6 to 5.7 micrometers was obtained. X-ray dif fraction data in Fig. 1 clearly indicated that well-formed crystalline diamond thin films could be deposited at low temperatures of 275°C. This observation has important implications on use of diamond films for microelectronic devices and packaging because of the high thermal conductivity. Figure 2 shows morphological features of the diamond films on a SiC wafer with grain size in the nanometer range. Raman spectrum in Fig. 3 is also indicative of diamond sp^3 bonding in the films deposited at 275°C on Si wafer from a sharp diamond peak at 1332 cm^{-1}. These results show promise of synthesizing diamond films at very low temperatures of ~275°C. Additional characterization and synthesis of these films are in progress.

In summary, facilities for in-situ monitoring of the plasma during CVD by QMS and optical spectrometer have been done. Microcrystalline and nanocrystalline thin films of diamond have been synthesized at temperatures as low as ~275°C, and their microstructure/structure/bonding determined by SEM, x-ray diffraction, and Raman techniques. Future work is planned to further characterize these films and synthesize superhard materials in the C-B-N system. Properties (thermal conductivity, hardness, electrical, and optical) of the diamond and C-B-N films will also be measured and related to the processing conditions.

A number of graduate students (3 at UC) are working on this project towards their MS, PhD degrees, and a research associate is also contributing to this project. These students have been trained on the manufacturing of thin films by ECR-MPECVD and on a variety of advanced characterization techniques, such as Raman spectroscopy, X-ray diffraction, and SEM.

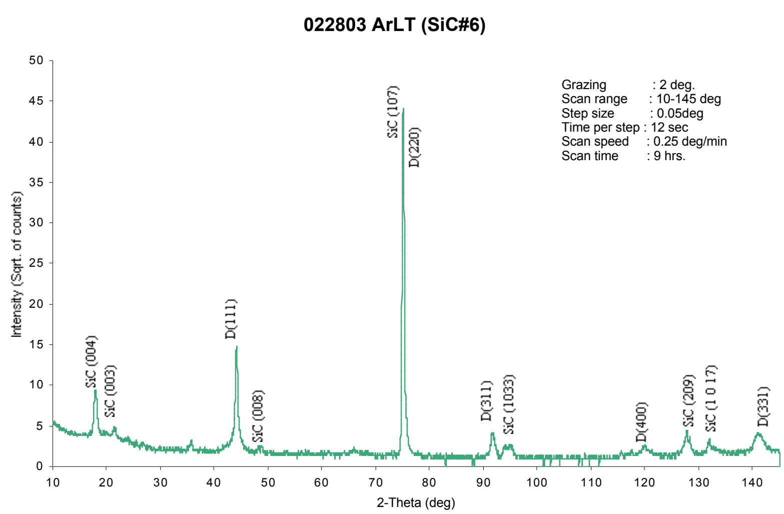

Figure 1. X-ray diffraction from diamond film deposited on Si at low temperature showing essentially a cubic diamond structure.

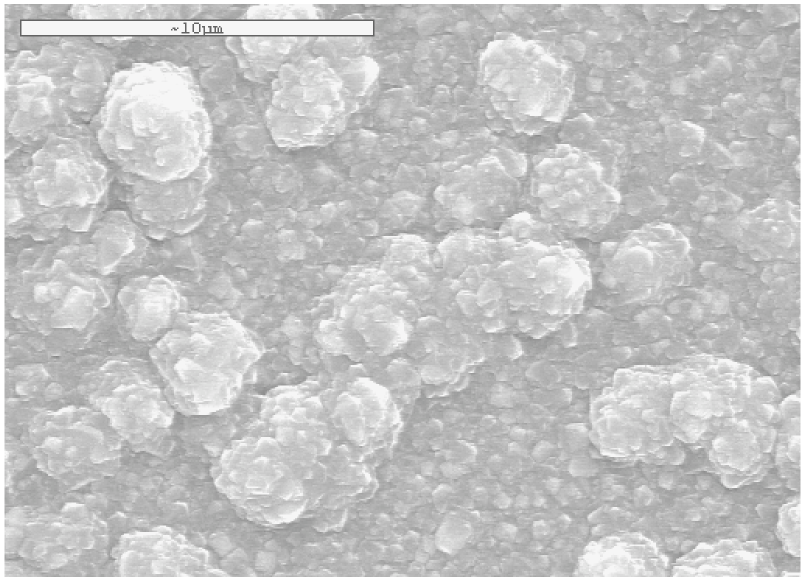

Figure 2. SEM micrograph of the diamond film deposited on SiC substrate at low temperature.

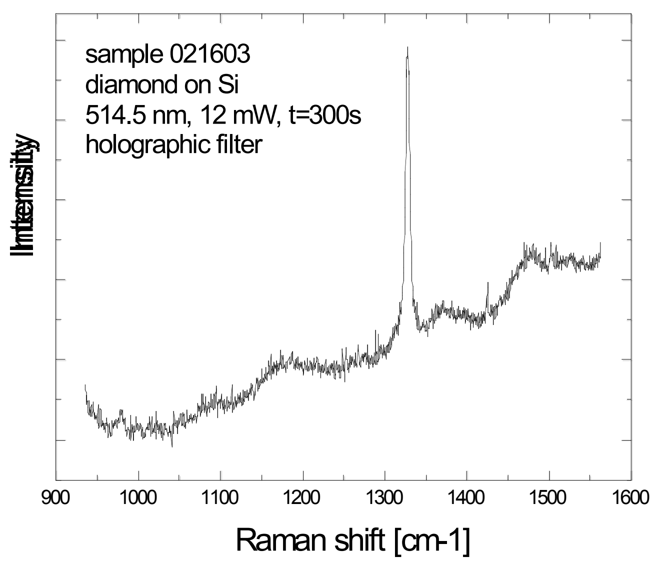

Figure 3. Raman spectrum from the diamond film deposited on Si substrate at low temperature.

References

[1] For further information about this project contact PI at Raj.Singh@uc.edu