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Electrostatic potential variation across the surface of a diamond/β-SiC nanocomposite thin film

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ABSTRACT

Electrostatic potential variation across surface of a diamond/β-SiC nanocomposite thin film is obtained using scanning Kelvin probe microscopy. An average electrostatic potential of ~ −120 mV is measured on the surface (5 × 5 μm^2) of the nanocomposite thin film. Using this value and the nominal work function of the probe, the work function values of the constituents could be estimated. In conjunction with secondary electron surface micrograph, the surface potential map features are relevantly assigned to the respective constituents in the nanocomposite thin film.

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It is well proven that Calvin (or Kelvin) method [1] is used to easily obtain electrostatic potential maps of solid surfaces. In a two-pass scanning Kelvin probe technique, during the 1st pass, surface topography is acquired using the standard semi-contact mode scan while in the 2nd pass, the probe is retraced to a set height (~10 nm) from the surface to map the surface potential V_{sp} as the probe scans the surface [2,3]. The simple relation that defines the measured electrostatic surface potential is given in Equation (1) [4],

$$V_{sp} = \left(\Phi_{probe} - \Phi_{surface}\right)/q = \Delta\Phi/q \quad (1)$$

where Φ_p and Φ_s are work functions of probe and surface, respectively while q is the elementary charge. $\Delta\Phi$ is the work function difference between the probe and surface. If no bias voltage is applied between the probe and surface, V_{sp} is nothing but the contact potential difference between the probe and surface. In this work, using scanning Kelvin probe microscopy electrostatic potential variation across the surface of a diamond/β-SiC nanocomposite thin film is measured and the contiguity of the nanocomposite's constituents could be identified. Moreover, the work functions' ranges of the constituents could also be roughly estimated.

The importance of this work is briefly explained here. In recent past, nanocrystalline diamond thin films have attracted considerable attention as candidates for special field effect transistors [5], bio-electronic systems [6], and other applications [7]. Similarly, nanocrystalline 3C-silicon carbide (SiC) thin film has attracted attention as an electrode material for electro-chemical [8] and biosensing [9] applications. In order to make use of the broad range of properties of diamond and β-SiC, diamond/β-SiC nanocomposite thin films have been developed [10–12]. These nanocomposite thin films have also shown their suitability for biosensing applications [13]. In previous studies, these nanocomposite thin films have been studied using micro Raman scattering, x-ray diffraction, infrared spectroscopy, electron microscopy etc., to obtain various material characteristics [11,12,14]. However, till date, any local electrical/electronic properties of diamond/β-SiC nanocomposite thin films have not been reported. In this work, the electrostatic potential on the surface of a diamond/β-SiC nanocomposite thin film is measured and discussed. Understanding of such local electrical properties will enable suitable integration of such thin films into electrical/electronic applications like the ones mentioned above. Moreover, this work shows that surface potential measurement aids quick characterization of composite materials for the presence of the concerned constituents with a plausible proximity in their work functions.

In this work, Kelvin probe force microscopy (KPFM) was carried out on the surface of a diamond/β-SiC nanocomposite thin film

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constituted by nanosized- diamond and β-SiC grains and graphitic carbon is considered [14]. The thin film was deposited on pre-treated (with nanodiamond particles) (100) Si using microwave plasma enhanced chemical vapor deposition technique [11]. The deposition was carried out using a gas mixture of hydrogen, methane and tetramethylsilane. The reaction was carried out at a constant gas pressure of 25 Torr, with the substrate temperature at 800 °C and using a microwave power of 700 W to ignite the plasma. The flow rates of hydrogen and methane used are 400 and 2.5 sccm, respectively. Tetramethylsilane concentration in the reaction mixture was only 0.0384%. The electrostatic surface potential has been mapped using KPFM module in NTEGRA Aura model (NT-MDT, Russia) scanning probe microscope. The mapping was carried out in semi-contact mode under ambient conditions. Doped silicon cantilever coated with Au (NSG01/Au from NT-MDT) was used as the probe. The probe height and curvature radius are ~14–16 μm and ~35 nm, respectively. The scan rate was 0.5 Hz. Relative humidity and temperature during measurement were in the range of 30–50% and 23–26 °C, respectively. The sample surface was electrically grounded using a copper connector. Prior to the surface potential measurement, the sample was thoroughly cleaned (ultrasonicated in isopropanol for 15 min to remove water layer due to moisture or other contaminants, if any) to avoid the possibility any contaminants' presence on the surface of the samples.

Plane view secondary electron (SE) micrograph of the diamond/β-SiC nanocomposite thin film's surface is shown in Fig. 1. Bright and dark regions represent diamond and β-SiC, respectively. Careful observation shows that the diamond features are smaller than the β-SiC features. Generally, the sharp contrast observed in the SE image can be explained based on the secondary electron yield from the sample surface and its dependence on the atomic number of the materials constituting the sample. It is well known that in scanning electron microscope (SEM), the SE yield increases with increasing atomic number of the specimen under inspection i.e., the brightness of the image taken at a particular electron voltage increases with increase in the atomic number of the specimen. Thus one would expect diamond regions to appear darker than the β-SiC regions. However, the diamond regions appear much brighter. Diamond is a highly insulating material and surface charging effects play an important role while imaging it using SEM. Brightness means that the amounts of emitted SEs in the region are greater than the other region. Topographic contrast may also have contributed to the increased SE yield. When the dimensions of nano-crystallites become comparable to, or smaller than, the escape depths of the SEs, generated inside each crystallite, with energies higher than the surface barrier, they may escape from the crystallite. In the case of diamond/β-SiC nanocomposite, both the phases are nanocrystalline in nature and therefore, both should equally contribute to the topographic contrast. However, the smallest between them should show up being brighter. In the present case diamond crystallites being smaller than the β-SiC crystallites, it is plausible that they appear brighter. Because of this geometric effect in addition to the insulating nature of diamond, even though diamond (carbon) is a low atomic number material, it gives a bright image contrast in the presented high-resolution SE images. Here it is important to mention that energy dispersive x-ray analysis and back scattered electron mapping of the sample surfaces have separately confirmed the assignment of brighter and darker regions to diamond and β-SiC, respectively [15]. While imaging using SEM, if the electron beam enters the sample perpendicular to the surface, then the activated region is uniform about the axis of the beam and a certain number of secondary electrons escape from within the sample and reach the detector. As the angle of incidence increases, the escape distance of one side of the beam will decrease, and more secondary electrons will be emitted. Thus, steep surfaces and edges tend to be brighter than flat surfaces when viewed under SEM. To avoid this effect, the SEM image shown in Fig. 1 is recorded with the electron beam entering perpendicularly the sample surface. Here it should also be mentioned that the above discussed brightness corresponding to diamond regions in SE image (Fig. 1) may plausibly also because of negative electron affinity (due to possible H_2 termination of diamond crystallites during synthesis which was carried out using a very high concentration of H_2 in the gas phase) of the diamond regions. Raman studies indicated the presence of very low graphitic carbon content in the thin film [14]. For convenience the diamond/β-SiC nanocomposite thin film will be henceforth addressed as 'composite film'.

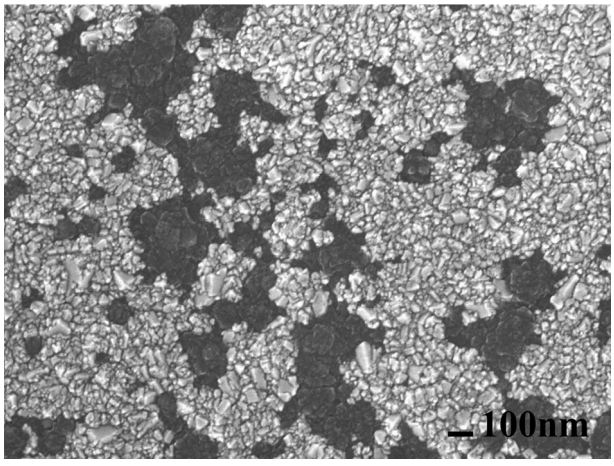

Fig. 1. High magnification plane-view secondary electron micrograph of diamond/β-SiC nanocomposite thin film surface. Bright and dark regions represent diamond and β-SiC, respectively.

The above discussion indicates that the composite film under a measurement method like Kelvin method should also give contrast images depicting the contiguity of the constituents. In the present case, it is thus trivial that diamond (being a better SE emitter than β-SiC) should indicate a higher surface potential (i.e. lower work function) than β-SiC. In other words, in a surface potential map of the composite film, diamond regions should appear bright while the β-SiC regions should appear dark.

Fig. 2(a and b) shows the surface topography and surface potential images, respectively of the same area of the composite film shown in Fig. 1. The local surface potential map clearly shows the variation between the two phases through the depiction of dark and bright regions (their variation in intensity is represented in mV). The average surface potential is found to be ~ −120 mV (with a range of values from ~ −135 to ~ −110 mV across the considered area). Based on Equation (1), the measured average surface potential (~−120 mV) and range of surface potential values across the surface of the considered composite film are correct only when the work function of the probe is less than that of the work function of the composite thin film surface. Moreover, surface potential values across the surface of the considered composite film indicate that the differences in work function ($\Delta\Phi$) fall in the range ~ 110–135 mV. For discussion purposes, the nominal work function of Au, 5.1 eV is considered. Here it should be mentioned that it has been customary to use this value for Au in such studies [16–19] even though it is known that the value can be lower (by 0.4 eV) [20] than 5.1 eV under ambient conditions. With this consideration and the measured surface potential values across the surface of the considered composite film, work function values of the composite surface fall in the range 5.21–5.235 eV. The arithmetic mean of 5.21 and 5.235 eV is ~5.222 eV which corresponds to the work function

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