With the use of novel high power and high frequency electronic devices proper extraction of dissipated heat became a major limiting factor hindering further miniaturization. It becomes necessary to deal with the thermal management of such devices and design suitable heat sinks from materials of superb heat conduction. In this respect, diamond is regarded as the ideal heat-spreading material owing to its exceptional thermal conductivity $k \sim 2200 \text{ Wm}^{-1}\text{K}^{-1}$. However, CVD-deposited diamond films used in most applications have a polycrystalline microstructure which reflects in an anisotropic, thickness-dependent thermal conductivity, whose value increases steadily departing from the small-grained/amorphous nucleation region towards the film’s surface. In order to optimize the thermal properties of the near-to-interface nucleation region it is crucial to understand the correlation between the growth parameters and film microstructure in the earliest stages of film deposition.

Transmission electron microscopy (TEM) offers the unique possibility to study this nucleation layer at the sub-nanometer scale, thus allowing to precisely ascertaining how its composition, structure and thickness are influenced by the choice of the growth parameters.

In this work, nanocrystalline diamond (NCD) thin films were deposited at 760 °C in a hot filament CVD (HFCVD) reactor onto single crystalline silicon substrates nucleated by means of a bias enhanced nucleation (BEN) technique. The growth parameters have been tuned to minimize the nucleation layer thickness and to promote the development of a strong columnar texture in the film. For thinning preparation to the TEM study we applied both FIB technique (with 30 kV Ga+ ions) and standard ion milling (with 10 kV Ar+ ions, followed by low energy milling). The samples were investigated in a JEOL 3010 high resolution TEM (at 300 kV).

The cross sectional TEM images of the interface region shows a transition zone between the diamond film and the Si substrate (Fig. 1). Its thickness is dependent on the deposition parameters but it looks as to contain nanocrystalline grains and amorphous phases as well. With high resolution electron microscopy both diamond and cubic SiC grains of a few nanometer sizes could be identified in this transition region (Fig. 2). In some samples also graphite-related phases (e.g. nano-onions with characteristic inter-shell distance of 0.35 nm) could be observed (Fig. 3). The diamond film itself was found polycrystalline with columnar microstructure. The orientation distribution was more or less random, due to the relatively low deposition temperature.

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Fig. 1: Fig. 1 Cross sectional TEM image of the interface between the substrate and the diamond film. The electron diffraction on the inset shows more or less randomly oriented poly-diamond on the single crystal silicon substrate.

Fig. 2: Fig. 2 The transition zone contains a few nm size grains of both SiC and diamond phases.

Fig. 3: Fig. 3 Spherical non-crystalline nanoparticle (nano-onion) found in the transition zone.