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Composite diamond films for short-mm wave and THz traveling wave tube windows

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ABSTRACT

With an increase in frequency, the diamond thickness of the microwave windows for short-mm wave and THz traveling wave tubes (TWTs) approaches 100 μm or even tens of μm. This poses problems of mechanical strength and air tightness to the polycrystalline diamond (PCD) window. To overcome these problems, we have studied a composite diamond film that consists of PCD and ultra-nanocrystalline diamond (UNCD). First, SEM was used to examine the early growing process of UNCD on PCD. The 5 μm thick UNCD grown on 40 μm PCD exhibited a hillock structure with densely packed ≤20 nm granules, in contrast to the PCD layer showing randomly packed, micrometer sized grains. Then, the effect of UNCD thickness on fracture strength and thermal conductivity was studied using the test samples with thin layers of UNCD having thicknesses of 1, 2.5, 5, and 10 μm on 100 μm thick PCD films, respectively. The fracture strengths of all the films are 2–3 times higher than that of the PCD films, which is 350 ± 150 MPa. As expected, the thermal conductivity of the samples measured at ~20 °C decreases with an increase in UNCD thickness, particularly in the range of 0 to 2.5 μm. At a thickness of 10 μm, the thermal conductivity was found to be ~10 W/cm * K. Finally, a 100 μm sandwich-like structure with a total UNCD thickness of 10 μm was fabricated and two 180 GHz TWT windows were assembled. RF tests show that for the operating frequency range of 175 to 185 GHz, the transmission loss (S_{21}) was found to be ≤1.22 and ≤1.71 dB, respectively, indicating an excellent RF performance. Mechanical strength and air tightness of the windows were also found improved and able to meet the requirement of the device. This work provides a novel approach for fabricating relatively thin diamond films for RF applications, such as TWT windows.

1. Introduction

There is an increasing interest in short-mm wave and THz vacuum electron devices, such as Traveling Wave Tubes (TWTs), due to their potential applications in several areas [1–3]. TWTs are known as a type of vacuum microwave devices for power amplification, which normally have two windows for transmission of input and output microwave signals. As a key component of TWTs, apart from having a low microwave loss, the window is required to be vacuum-sealed from the atmosphere by brazing window disks to metallic frames connected to waveguides. [4,5]. With an increase in frequency, the thickness of the window disks, made from conventional polycrystalline diamond (PCD), approaches ~0.1 mm or even tens of micrometers [6]. This poses some problems to mechanical properties of PCD films. First, it is too thin to be lapped and polished without cracking and breaking up. Second, such thin PCD window disks are difficult to meet the requirements of a vacuum device, such as air tightness, and mechanical strength against bake-out (an environment: up to 450 °C at a pressure of ~2 Pa) and cooling cycles. Although PCD films have low microwave loss and high thermal conductivity, the window made of relatively thin (≤0.15 ± 0.005 mm) PCD films is subject to cracking and slow air leaking due to their tens of micrometer sized grains. In contrary, UNCD films, developed by Gruen [7,8] at Argonne National Laboratory, possess a lower surface roughness and higher fracture strength [9–12], resulting from densely packed ultra-fine nanocrystalline grains. In comparison with PCD, however, UNCD has a relatively poor thermal conductivity due to a large amount of boundaries, where there exists a small amount of sp^2 carbon atoms [9]. Furthermore, relatively thick UNCD films may result in high microwave loss to some extent [13,14]. It is, therefore, desirable to engineer a composite film of PCD–UNCD for mm-wave and THz TWT windows, as to combine the advantages of PCD and UNCD, while minimizing their disadvantages. Though composite diamond films of PCD with UNCD for characterization of dielectric properties at lower frequency [14] and applications of cutting tools [15] have been investigated previously, studies of such freestanding composite films for short mm wave and THz TWT windows have rarely been reported. This study is aimed at developing relatively thin diamond films for short mm wave and THz TWT windows. In doing so, PCD–UNCD composite films with thin layers of UNCD were fabricated and characterized to get insight into effect of UNCD thickness on mechanical and thermal properties. Then, the composite film for the

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window was designed partly based on the findings from the characterization data. Finally, the TWT windows made from the composite films were tested for the transmission and mechanical properties.

2. Experimental procedure

2.1. Growth processes of PCD and UNCD

Diamond films were produced on a N-type Si(100) wafer using a 6 kW, 2.45 GHz MPCVD reactor (DiamoTek 700) [16]. Prior to the growth process, the silicon substrate was mechanically polished with diamond powder 0.25 μm in size, attaining a nucleation density of $\geq 10^{10}$ cm^{-2}. PCD was prepared by a conventional H_2/CH_4 reaction gas mixture. The condition for PCD diamond films was as follows: absorbed microwave power of 1.6–1.8 KW, total pressure of 13.3 kPa, gas flow rate of $CH_4/H_2 = 3/300$ (sccm) and substrate temperature at 800–900 °C. In contrast, UNCD was deposited under a distinct condition: absorbed microwave power ~1.4 kW, total pressure ~11.0 kPa, a gas flow rate ratio of $CH_4/H_2/Ar = 1:2:100$(sccm) and at a substrate temperature of 650–750 °C. Since the reactor has no heater for the substrate, the microwave power and total pressure used in the UNCD growth are relatively high as compared to those reported by Gruen [8]. The growth rate for both PCD and UNCD was ~1 μm/hr. Freestanding diamond films were obtained by etching away Si substrate in a chemical solution.

2.2. X-ray diffraction and Raman analysis of the UNCD film

Fig. 1 shows an X-ray diffraction pattern of the top UNCD film. Characteristic (220), (111) and (311) diamond reflections are observed with no graphite phases. Using Scherrer formula [17]:

$$D = \frac{K\lambda}{B \cdot \cos\theta}$$

where, D is the average grain size, B, the width of the reflection at half maximum, K, Scherer factor (taking $K = 0.89$) and λ, wave length of X-ray, the average grain size of the UNCD is estimated to be ~10 nm. This value is slightly larger than that reported by Gruen, possibly due to the higher growth pressure.

Fig. 2 gives a Raman spectrum of the UNCD, taken by using the 514.5 nm line of a Ar + laser at a spot size of 1 μm. The spectrum has a characteristic diamond peak at 1332 cm^{-1} and broad features at 1140, 1340, 1470 and 1565 cm^{-1}. Peaks around 1340 and 1565 cm^{-1} known as D and G bands normally occur in UNCD and are attributed to non-diamond carbon phases, while these around 1140 and 1450 cm^{-1} are assigned to C═C stretching and wagging of trans-polyacetylene [18,19] in a nanocrystalline diamond or UNCD.

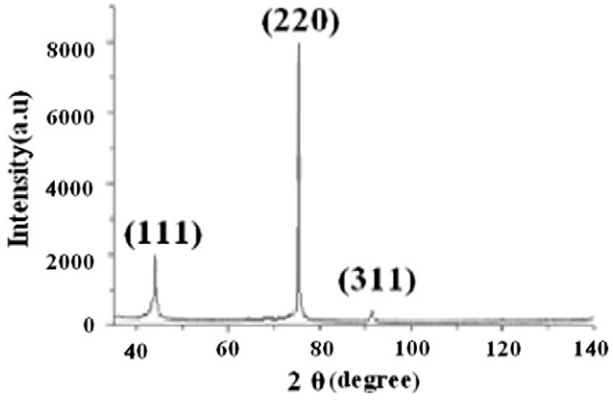

Fig. 1. An X-ray diffraction pattern of the 5 μm thick UNCD grown on 40 μm PCD film.

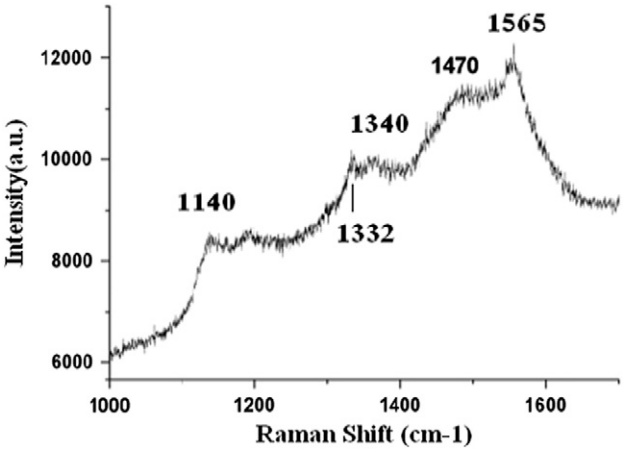

Fig. 2. A Raman spectrum of the 5 μm thick UNCD grown on 40 μm PCD film.

3. Results and discussions

3.1. Characterization of composite films

3.1.1. Growing process of UNCD on PCD

To get an insight into the growing process of UNCD on PCD, SEM was used to monitor variations of surface morphology during the fabrication process. A 40 μm thick PCD film was first deposited on a silicon wafer, followed by a consecutive growth of UNCD for 0.5, 2.5, and 5 h, respectively. Fig. 3 shows SEM images for: (a) PCD, (b) UNCD (0.5 h), (c) UNCD (2.5 h), (d) UNCD (5 h, high magnification) and (e) a cross-section view of a composite film with 5 μm UNCD. From Fig. 3(a), it can be seen that the rough surface exhibits sharp peaks and valleys, resulting from randomly packed diamond grains up to ~10 μm size. After a 0.5 h deposition of UNCD, the PCD surface is fully covered with nanometer granules with sharp corners and straight edges becoming smeared (Fig. 3(b)). Following the 2.5 hour deposition of UNCD, the surface becomes smoother, as sharp corners and straight edges disappear and the PCD grains are transformed into hillock appearance, as shown in Fig. 3(c). With the 5 hour deposition of UNCD, the hillocks further develop while valleys and gaps continue to be filled with UNCD granules and the PCD grains has finally lost their identity. Fig. 3(d) gives a high magnification SEM image after the 5 hour deposition, showing a UNCD feature with a granule size of ≤20 nm.

It is interesting to note the transformation from a rough PCD into a relatively smoother UNCD with a hillock structure. In terms of the major feature of UNCD growth, one would naturally relate it with the high secondary nucleation rate. From the evolution of SEM images, it seems that the high secondary nucleation rate or the UNCD deposition rate does not favor the sharp corners and edges facing upward rather than valleys, which may result from ion sputtering or H^+ etching effect [20,21]. Jiang et al. [20] reported direction-dependent H^+ ion etching of diamond crystals leading to smooth [001] film growth during a biased MPCVD process. Though there was no bias applied to the substrate in our UNCD growth process, Ar + ions from the UNCD plasma possess a much higher kinetic energy than H^+ ions. The cross section view in Fig. 3(e) clearly shows that at an interface area near the growth surface, the top UNCD layer contains dense nanometer sized granules, whereas the bottom PCD layer features a columnar structure with large grains. There is no interlayer between UNCD and PCD, indicating the UNCD film starts to grow on PCD immediately after the process transformation from PCD to UNCD. This is, in fact, confirmed by the SEM image of Fig. 1(b), where UNCD grains grow on PCD surfaces at an early stage.