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Control of abnormal grain inclusions in the nanocrystalline diamond film deposited by hot filament CVD

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ABSTRACT

Formation of abnormal grain inclusions in nanocrystalline diamond films deposited by hot filament CVD (HFCVD) was investigated. The phenomenon was attributed to two different origins: an intrinsic and an extrinsic one. The inclusions due to the intrinsic origin could be either avoided or weakened by controlling chamber pressure, CH_4/N_2 concentrations in H_2, and by positive substrate bias. The extrinsic origin for the abnormal grains was found to be the contamination from the alumina insulation tubes for the thermocouple placed near the substrate, which were degraded by the extended exposure to the high temperature and strongly reducing atmosphere.

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1. Introduction

The deposition of nanocrystalline diamond films has received a great deal of attention in recent years. Although there are many applications for microcrystalline diamond films as thick as 1 mm, nanocrystalline diamond films are most frequently used at a thickness below 1 µm. Among the various properties of thin film materials, the microstructural uniformity is one of the typical requirements for industrial applications. Since the nanometer-sized grain structure is the key feature of nanocrystalline diamond films competing against the conventional microcrystalline diamond films, the uniformity of the grain size is of prime importance. On the other hand, the inclusion of abnormally large grains among the otherwise-uniform microstructure is a well-known problem in the fabrication of polycrystalline materials of various types. A typical example is the cemented carbide (WC–Co) alloy, where the abnormally large grain inclusion is known to cause a serious deterioration in fracture strength [1]. Likewise, the abnormally large grains embedded in the otherwise-uniform microstructure of nanocrystalline diamond film must give a serious damage to various performances of the film. For example, it can cause crack initiation for various mechanical applications where the nanocrystalline diamond film is subjected to mechanical stress. It can also damage the integrity of the sophisticated structure of MEMS devices or other electronic devices based on nanocrystalline diamond film.

Recently, the existence of the microcrystalline grain inclusions in nanocrystalline diamond films and the technique to prevent them were reported for the microwave plasma CVD process using Ar-rich chemistry [2,3]. The control technique was reported to be based on controlling the carbon-to-hydrogen ratio in the source gas composition. In the present study, we show that the abnormally large grain inclusions indeed occur in the HFCVD nanocrystalline diamond films in some regions of the parameter space. We also show that they can be avoided or weakened by a proper control of the deposition conditions.

2. Experimental

Depositions were performed on 4-inch boron-doped Si wafers with a thickness of 0.5 mm in a large-area HFCVD reactor with and without positive bias applied to the substrate with respect to the filaments. A Si wafer was placed on a 5-inch tungsten disk with a thickness of 20 mm which, in turn, was placed on the copper cooling block.

The W filaments with a diameter of 0.3 mm were carburized before the deposition process. The distance between the filament and the substrate surface was about 10 mm. Prior to deposition, the substrates were treated ultrasonically in a methanol slurry containing nanodiamond powder with an average particle size of 5 nm. They were subsequently cleaned ultrasonically in ethanol and blown dry with nitrogen gun. A gas mixture of H_2, CH_4 and N_2 was used as the precursor gases. The temperature of the filaments was around 2100 °C, as measured by an optical pyrometer through a quartz window. Unless otherwise stated the deposition temperature was about 670 °C, as measured by a thermocouple placed on the top surface of the tungsten disk, close to the Si wafer during the deposition. In other cases, for the reason stated later in the next section, the deposition was carried out without temperature monitoring by the thermocouple. In these cases the deposition temperature was kept at about 670 °C by maintaining the

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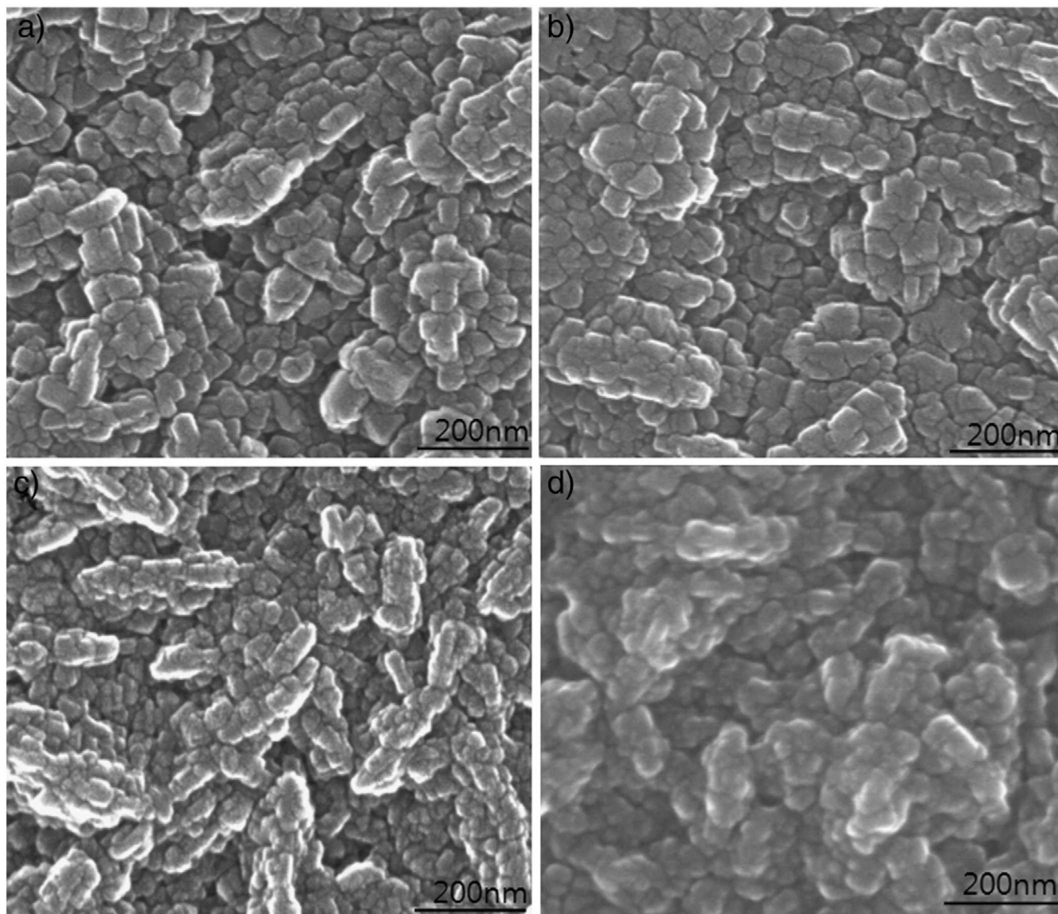

Fig. 1. SEM pictures for depositions at 30 Torr and 50 Torr: a) 30 Torr, bias, 3% $CH_4 + 0.3\% N_2$, b) 30 Torr, no bias, 3% $CH_4 + 0.3\% N_2$, c) 50 Torr, bias, 3% $CH_4 + 0.3\% N_2$, and d) 50 Torr, no bias, 3% $CH_4 + 0.3\% N_2$.

filament temperature, the filament to substrate distance and the substrate holder configuration. The two strands of the R-type thermocouple were guided through a series of short alumina insulation tubes, each of which was about 7 mm long, a few mm in diameter and had two thin holes for the thermocouple wire guidance. The welded tip of the thermocouple, exposed at the end of the series of the insulation tubes, was in contact with the top surface of the tungsten disk on which the Si wafer resided. In this configuration, the actual surface temperature of the silicon wafer would have been somewhat higher than the temperature measured by the thermocouple because the Si wafer surface was closer to the filament by the amount of the wafer thickness and because the thermal conductivity of Si is lower than that of tungsten. The bias current and voltage were 5–10A and 30–110 V, respectively, depending on the deposition conditions. The reactor pressure was varied from 10 Torr to 50 Torr. The deposition was carried out for 2 h and 10 min. After the deposition, the deposited films were characterized by X-ray diffraction (XRD, MAC Science), scanning electron microscopy (SEM, FEI Nova 200 NanoSEM), energy dispersive X-ray spectroscopy (EDX, FEI), high resolution transmission electron

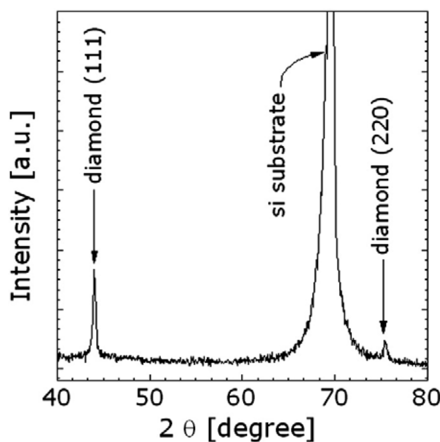

Fig. 2. XRD pattern for the film shown in Fig. 1a.

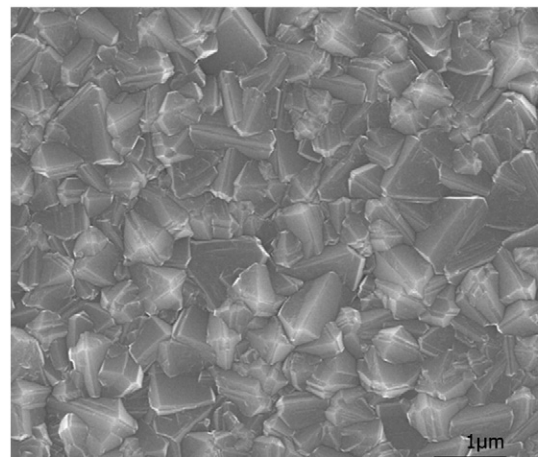

Fig. 3. SEM picture for the film deposited with a gas composition of 3% CH_4 in H_2 and with bias.

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