Dielectric properties of single crystalline diamond wafers with large area at microwave wavelengths

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ABSTRACT

In this paper, we present first measurements, carried out at microwave wavelengths, aiming to characterize the dielectric properties of large size single crystalline diamond (SCD) wafers. While the sizes of the SCD wafers are still not sufficient for practical use, we obtained good optical property results. The sample with both sides polished shows a dielectric loss tangent tanδ as low as or possibly lower than a polycrystalline diamond sample with high quality. Results show the importance of surface treatment, especially on the boundaries of the composed SCD chips even without any graphitic component in the diamond wafers.

1. Introduction

Controlled thermonuclear fusion is considered one of the most prom ising and attractive alternative energy sources due to its reduced CO_2 emission [1]. At present, the international project to build a facility to ver ify its feasibility, the International Thermonuclear Experimental Reactor (ITER), is ongoing [2]. For plasma confinement extended in time, additional heating and current drive systems are required. Injection of high frequency (170 GHz) electromagnetic waves mitigates MHD (Magneto Hydrodynamic) instabilities which occur in the plasma. For injection of millimeter waves with high energy, a microwave transparent window with high thermal conductivity and low microwave losses is mandatory. These windows should also act as a barrier for the tritium present inside the reactor's vacuum vessel, to avoid its dispersion in the facility environment. In principle, material properties of diamond can satisfy these requirements. Diamond has several characteristics which are superior to those of other relevant materials (like, for example, sapphire or fused silica), such as mechanical hardness, optical transmis sivity, and high electrical breakdown threshold [4]. Because of its wide band gap, high thermal conductivity and high carrier mobility, diamond is considered to be one of the promising candidate materials to realize fu ture high performance electronic devices. Part of its characteristics has been confirmed by current devices, such as diodes and transistors [5 7] made of diamond, which show its rapid response and stable performance at high temperatures [8]. For this application, defects underlying the electrodes cause leakage current [9]. On the other hand, the low dielectric loss tangent of diamond allows ultra low loss transmission of high power millimeter waves through the windows [3]. In case of the polycrystalline diamond, there are many grain boundaries with hydrogen carbon bonds and sp^2 components, which are considered to be the cause of power ab sorption of the electro magnetic waves. Single crystal diamonds (SCD) don't have such grain boundary, resulting in improved transmissivity characteristics.

However, commercially available sizes of single crystal diamonds are much smaller than those of polycrystalline ones and other semicon ductor materials. In the case of polycrystalline diamond, wafers with a diameter of up to ~120 cm are available commercially [10,22,23], which is a sufficient size for their use as windows in Electron Cyclotron Resonance Heating and Current Drive (ECRH&CD) fusion reactor systems. Typical size of SCD for studies as semiconductor materials is 2 3 mm in edge length at present [11]. In the cases of Si and SiC, the crystals expand the area of their growing surface during the growth [12,13]. But in the case of SCD, except for sophisticated control of the alpha parameter (ratio between 100 and 111 directions growth rates) [14], the top surface of the seed crystal shrinks during the growth [15]. Surface expansion is only possible under the right conditions, but is counterbalanced by an extremely low growth rate [16]. Even by using three dimensional growth [17], production of the bulk crystal re quires impractical durations, e.g. 1000 h. The first author of this paper proposed a technique to compose relatively small samples of SCD into a larger size area, with sufficient quality to fabricate freestanding wafers from it [18]. The boundaries are expected to induce some degradation of the crystal quality but, for the semiconductor use, this is not considered to be a serious problem if the electrodes don't cross the boundaries. By

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using the "Tiled SCD clones" technique, the production of wafers bigger than 20 × 20 mm^2 is possible [19].

In this manuscript, we report the first measurement of dielectric properties of such tiled clones of SCD. While its size is still smaller than the practical size, it is considered to be meaningful if we could pro vide some fundamental information about it for further development and the fabrication of wafers with larger size. In the next section, the ex perimental procedure and dielectric characteristic are described. The re sults with discussions are given in Section 3. The paper is summarized in Section 4.

2. Experimental setup

2.1. Preparation of diamond wafers

As reported in the preceding works [15,19,20], a single crystal diamond was grown by using microwave plasma chemical vapor deposition (MPCVD). 5 10 kW of microwave power was introduced into the vacuum chamber which was filled with the source gas mixture at 10 20 kPa of pressure. The source gas mixture was 90% of hydrogen and 5% of methane, with approximately 100 ppm of nitrogen. The grown diamond layers are divided from the seed wafer by using a lift off process with high speed ion beam injection [17]. As described in the previous reports [18,19], we applied such lift off process several times on identical seed crystals to have freestanding wafers all with similar characteristics. We shall call such wafers as "clone wafers". Then, we grew SCD layers on these clone wafers. This overgrown SCD layer connects clone wafers with each other, as the so called "tiled clones". Finally, we had the free standing wafers of the tiled clones. The thickness of the tiled clones, which were used for the measurements were adjusted around 1 mm. All samples consist of 4 and 8 clones with an area of 1 cm^2 each. In the following we shall call them four and eight fold samples, respectively. Fig. 1(a) and (b) shows the four and eight fold samples, where the substrates colored in gray were adopted for the dielectric measurements. In the case of the four fold sample, the substrate was polished and the boundary is almost invisible. On the other hand, in the case of the eight fold sample, the boundary is visible because the surface opposite to the as grown side was not polished and not fully filled by the CVD layers as shown in Fig. 1(b). The main reason of this is the high effort necessary to polish the substrates even at this size, i.e. 40 mm edge length and this was left for future work. Fig. 2 shows the Raman spectrum, which was measured at a boundary of a mosaic substrate by using a HORIBA Jobin Yvon T64000 system with a Nd:YAG laser source operating in second harmonic at a wavelength of 532 nm. The full width half maximum (FWHM) of the peak at 1332 cm^{-1} originated from the diamond struc ture is approximately 2 cm^{-1} even on the boundary, which is similar to that of commercially available CVD diamond samples. The broad peak around 1420 cm^{-1} is associated to the fluorescence from a nitrogen vacancy center. As it can be seen in the figure the sp^2 graphite like peak at 1583 cm^{-1} is missing. Breaking of the mosaics sometimes hap pens owing to some stresses during the growth caused by, for example, a difference in the impurity concentrations. In such a case, cracking gen erates regardless of the boundaries. Therefore, we consider that the ef fect of the boundary on the toughness would be small.

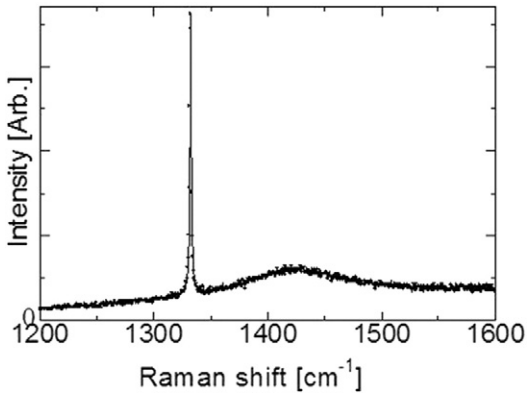

Fig. 2. Raman spectra of a clone substrate.

2.2. Measurements of dielectric constants

The measurements performed included loss tangent characteriza tion in three different Fabry Perot resonators: hemispherical (variable frequency source, 90 100 GHz, $\Delta(\tan\delta) = 10^{-5}$), double spherical res onator (170 and 145 GHz, $\Delta(\tan\delta) = 10^{-6}$) and hemispherical XY map ping (145 GHz, $\Delta(\tan\delta) = 10^{-5}$). The beam profile used in the tests is the basic Gaussian TEM$_{00}$. The measurement technique used in the

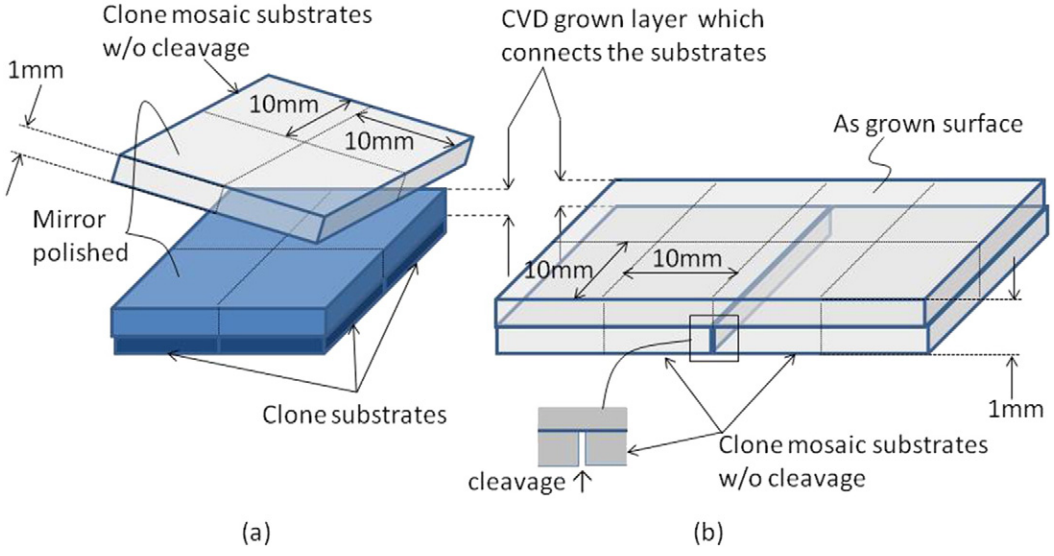

Fig. 1. Schematics of (a) the four- and (b) the eight-fold samples. The substrates colored in gray were adopted for the following measurements.

resonator consists in a first set of measurements with empty resonator to record the Q factors of the various resonator modes followed by the measurements with the samples inserted. A comparison between the resonance frequency and cavity quality factor of the empty resonator and of the resonator with the sample is then carried on. The final values of the loss tangent are obtained from the calculation of the inverse Q factors. The relative permittivity ε_r is extracted by solving the resona tor equation iteratively [21]. The double spherical resonator is also equipped with a micro positioning system on which the sample is mounted. In this way, it is possible to move the sample to alternate between wave maxima at the surface and inside the sample itself. This allows us to distinguish between bulk and surface losses. The surface contribution to the global tanδ varies from a minimum when the node of the wave corresponds to the face of the sample (resonant condition) to a maximum when this correspondence involves the antinode (antiresonant condition).

3. Results and discussion

The single crystalline samples attained good results in the loss tan gent measurements, even if they lacked proper surface polishing in one (4 fold) or both (8 fold) sides. The XY mapping resonator worked at its resolution limit for the tanδ of 10^{-5} (Fig. 3a) and improvement over surface losses are expected once the samples receive surface treat ment. As a reference, the tanδ measurements performed on the poly crystalline, surface polished, sample in the hemi spherical resonator showed a loss tangent in the order of 10^{-5} (Fig. 3b), which is compara ble to the current state of the art [24 26]. Theoretically the

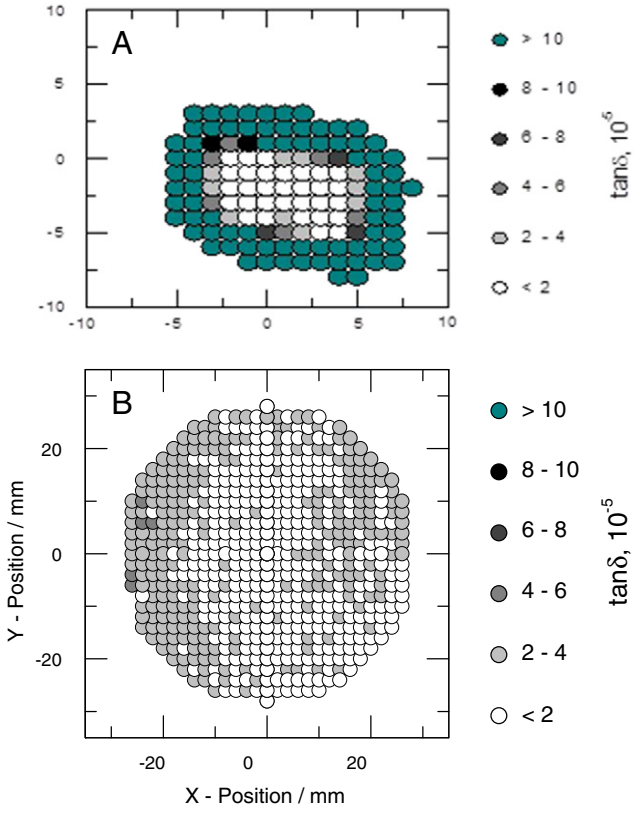

Fig. 3. (A) XY tanδ mapping of one of polished four-fold square SCD samples with 20 mm edge length; (B) XY tanδ mapping of a polished circular polycrystalline sample with a diameter of 80 mm. The cross shaped low tanδ region is due to the processing reactor electrode geometry.

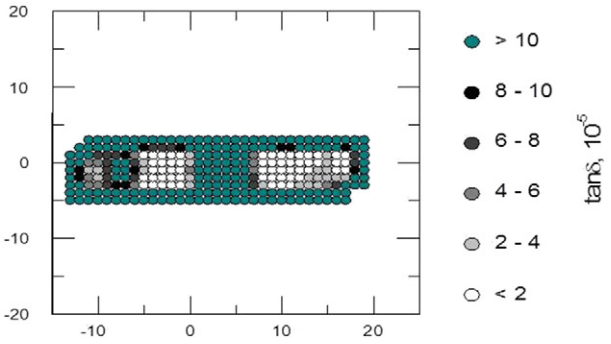

Fig. 4. XY tan δ mapping of one of polished eight-fold square SCD samples with 20×40 mm^2 area.

polycrystalline samples suffer from the bulk losses given by sp^2 bonds between the single crystals [27,28].

The measurements may indicate that a good surface quality and thickness homogeneity improves the transmission performances. Further tests on a polished single crystalline sample are required to confirm this assumption. For the four fold and eight fold samples, we found that the tanδ is less than or equal to 2×10^{-5} in minimum, where its relative permittivity is 5.7. In the central region of the sample, losses are smaller than in the periphery. At present, we consider that the diffraction in the edge region of the sample (diamond to air interface) causes degradation of the measured Q value in the resonator and results in an artificial measured increase of tanδ. Nevertheless, assuming such edge effect caused by the diameter of the microwave beam waist, the loss tangent of the whole sample can be estimated to be lower then 2×10^{-5} excluding the interface structure of the connected diamond tiles.

On the other hand, in the case of the eight fold sample, in the central region, the magnitude of tanδ is relatively high, peaking over 10^{-4} (Fig. 4) As mentioned above, in the case of the four fold sample, the sample was lifted off from the polished surface and the grown surface was also polished as shown in Fig. 1(a). Therefore, the boundary of the four fold sample is almost invisible. In the eight fold sample, the bound ary is visible because the surface opposite to the as grown side was not filled as shown in the Fig. 1(b). The cleavage of the eight fold sample may enhance the edge effect. In addition a slight increase of the tanδ could be also observed in the vertical directions around $x = 15$ mm. Given that the boundaries between tiles extend in both x and y direc tions, this effect is more likely given by the growing angle offset. The crystals don't grow along the normal to the surface, but they have an an gular offset of about 3°. As shown in the preceding report, this affects the quality of the samples: better results are obtained when offset vector is perpendicular to the boundary. The present results suggest that such effect also contributes to the dielectric properties.

4. Summary

The measurement of dielectric properties of large size SCD tiled clone wafers was conducted for the first time. Even though it is still technically hard to produce 4 inch size wafers of such SCD tiled clones, we obtained results that are promising. Results indicate that the sample where both sides are polished has dielectric tanδ as low as that of the polycrystalline diamond with high quality. On the other hand, results show importance of surface treatment, especially on the boundaries between the single tiles even if there are no sp^2 graphite like components in the wafers. To obtain practical size and quality of the SCD wafers for the use in windows, it is required to polish such large size wafers without cracking within and to achieve this within a practical processing time. This is left for future work.

Prime novelty statement

Dielectric properties of single crystalline diamond wafers with large area at microwave wavelengths were studied for the first time. A dielectric loss tangent tanδ as low as or possibly lower than a polycrystalline diamond sample with high quality was found.

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