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

Hideaki Yamada [a,*], Andreas Meier [b], Francesco Mazzocchi [b], Sabine Schreck [b], Theo Scherer [b]

[a] Diamond Research Group, Ubiquitous Energy Research Unit, National Institute of Advanced Industrial Science and Technology (AIST), Japan
[b] Karlsruhe Institute Of Technology (KIT), Institute of Applied Materials (IAM), Germany

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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 promising and attractive alternative energy sources due to its reduced CO_2 emission [1]. At present, the international project to build a facility to verify 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 transmissivity, 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 future 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 absorption 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 semiconductor 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 requires 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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[*] Corresponding author.
E-mail address: yamada-diamond@aist.go.jp (H. Yamada).

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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 provide some fundamental information about it for further development and the fabrication of wafers with larger size. In the next section, the experimental procedure and dielectric characteristic are described. The results 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 structure 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 happens owing to some stresses during the growth caused by, for example, a difference in the impurity concentrations. In such a case, cracking generates regardless of the boundaries. Therefore, we consider that the effect of the boundary on the toughness would be small.

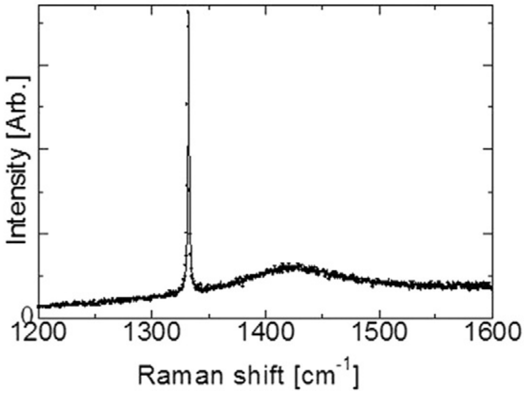

Fig. 2. Raman spectra of a clone substrate.

2.2. Measurements of dielectric constants

The measurements performed included loss tangent characterization in three different Fabry–Perot resonators: hemispherical (variable frequency source, 90–100 GHz, $\Delta(\tan\delta) = 10^{-5}$), double spherical resonator (170 and 145 GHz, $\Delta(\tan\delta) = 10^{-6}$) and hemispherical XY mapping (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

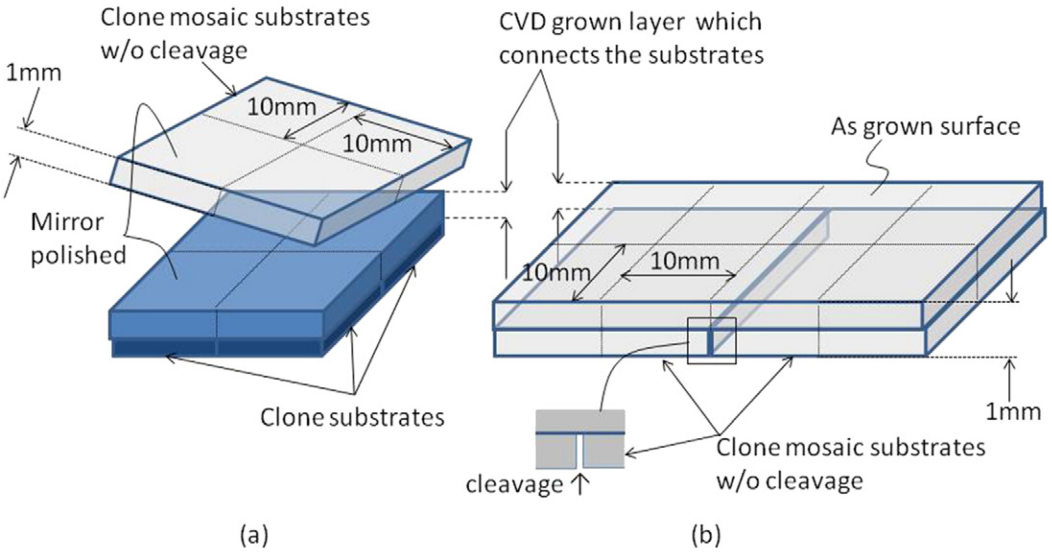

Fig. 1. Schematics of (a) the four- and (b) the eight-fold samples. The substrates colored in gray were adopted for the following measurements.