On the characterization of MPA CVD diamond for fracture toughness measurements

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***Abstract*—Failure due to crack propagation is the crucial failure mode of the optical quality polycrystalline diamond disks, which are used as safety and vacuum barriers in the electron cyclotron systems of nuclear fusion devices. This paper reports on the characterization of preliminary polycrystalline diamond samples for experimental measurements of the diamond fracture toughness by the double-torsion method.**

Keywords—EC systems, CVD diamond, diamond windows, fracture toughness, microfeatures

I. Introduction

Optical grade polycrystalline chemical vapour deposition (CVD) diamond for use in nuclear fusion in the form of disks is grown in microwave plasma assisted (MPA) reactors. Such diamond disks allow high-power (MW-range) and long-pulse gyrotron operations in the electron cyclotron (EC) systems required by the fusion devices while confining tritium and other hazardous materials such as radioactive dust [1]. During the CVD process, microfeatures appear in the disks both as single microcracks and discrete clusters of microcracks. Although the mechnical loads that are exerted to the disks are minimized by an appropriate design of the metallic housing where they are integrated, beyond normal operation, off-normal events may occur leading e.g. to an overpressure acting on the disk. Failure to fracture, i.e. failure due to crack propagation, is the crucial failure mode of the diamond disks. Therefore, measurements of the fracture toughness, which describes the resistance of a material against crack extension, of such a diamond are required. Diamond has in fact a fundamental safety function in fusion, but data in literature about the fracture toughness are quite limited [2, 3]. This paper reports on the characterization activities carried out on preliminary MPA CVD diamond samples whilst awaiting real-size samples for fracture toughness measurements.

II. Diamond samples

The double-torsion method was selected for the experimental measurements of the diamond fracture toughness [4]. A dedicated setup was designed and drawings of the parts to manufacture are being finalized. Diamond, particularly the optical grade, is quite expensive and attention must be paid to the size of the samples. The measurements are planned for samples with the dimensions 1.11 mm x 15 mm x 30 mm. The thickness of 1.11 mm is dictated by the resonance condition of the ITER torus EC disks. An optical disk of 80 mm diameter and 1.11 mm thickness was manufactured by Diamond Materials [5]. It shall be emphasized that this disk was manufactured in the same way as the 60 disks produced for the ITER EC torus window units. It is planned to extract 6 samples by laser cut. In addition, a 150 mm disk of a thermal-grade diamond was also manufactured by Diamond Materials to extract 9 samples for the measurements from the mid-region of such a disk. Thermal grade diamond has a higher density of microfeatures than the optical one, thus a different failure mode might occur in case of fracture. In parallel to the growth of the two-above mentioned diamond disks, 6 preliminary optical and thermal grade diamond samples of 1.11 mm x 12 mm x 22 mm were kindly provided by Diamond Materials for initial characterization. Microscopic, X-rays diffraction (XRD), electron backscatter diffraction (EBSD), and Raman measurements were carried out on the two grades of diamond considering both samples' surfaces, i.e. the nucleation (NS) and growth (GS) sides.

III. Characterization of preliminary samples

The samples were first examined by a high-resolution Olympus BX53M digital light microscope with about 50 scans in the vertical direction, combined then in an extended focal imaging (EFI) image of the samples, as shown in Fig. 1.

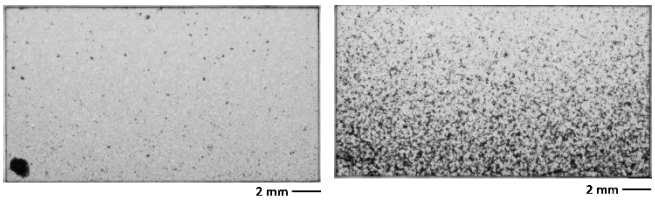

Fig. 1. Images of the diamond samples obtained by optical microscopy with growth side on the top: optical quality on the left and thermal quality on the right. The mark on the left side of the samples is used for their orientation.

It can be immediately observed that the density of dark microfeatures generated during the CVD process is much greater in the thermal than in optical grade. This is due to the higher deposition rate applied for the thermal grade. In general, it can be stated that these microfeatures appear as single microcracks or discrete clusters of microcracks where light is trapped and some of them might have faces decorated with sp^2 phase, increasing microwave absorption. With faster growth rate, the microcrack clusters in a thermal grade diamond connect to each other's into a continuous network (Fig. 1, right). Extensive Raman investigations were thus carried out in the dark microfeatures and the "clean" parts of the preliminary samples by lasers at 532 nm and 785 nm. The measurements did not show the presence of sp^2 phase in the selected areas although several positions in the samples were measured.

Subsequently, since residual stresses might influence diamond failure due to crack propagation, XRD residual stress analysis was performed on both NS and GS of the samples. The XRD pattern and macro-texture were also measured. The $\sin^2\psi$ method was applied to determine the surface macroscopic residual stresses [6]. Plane stress conditions were assumed. The lattice spacing d of a specific plane is measured at different sample inclination angles (tilt angles ψ), along a certain ϕ direction, being ϕ the rotation angle around surface normal. A plot d - $\sin^2\psi$ is generated and residual stress is obtained from the straight-line slope (least squares regression) and the material elastic constants. As shown in Fig. 2, a central sampling area of 3 mm x 3 mm was considered with three directions of measurements and 16 sample tilt angles varying from 0 to 60°. The (331) lattice plane was selected from the XRD pattern and it corresponds to $2\theta = 140.6°$. Cu K-Alpha radiation was used and the peak position was determined by modified Lorentzian shape function. The average macro-residual stress appears to vary from few to circa 200 MPa, both in tensile and compression state. These are preliminary results which require further investigations. A large value of standard deviation appeared, which might be caused by texture and/or columnar growth of diamond, as observed in [7] for thick diamond samples. It shall be also noted that stresses relax when the samples are extracted from the diamond disk. Since the diamond disks were manufactured and delivered, next XRD residual stress analysis is planned for the entire disks, aiming to check any macroscopic residual stress difference between central and outer regions.

Finally, microstructure and micro-texture of the samples were examined by EBSD measurements. As diamond is an electrical insulator, different tests in a stepwise approach were required to find the appropriate method for a good measurement to perform. This method shall be later applied to the samples to extract from the manufactured disks.

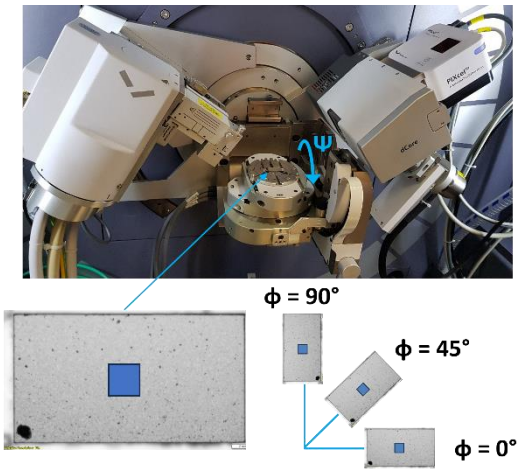

Fig. 2. Arrangement of the samples in the diffractometer for XRD residual stress analysis.

Outstandingly, it was possible to carry out EBSD on the entire surface of the samples' GS while on the NS, where grains are much smaller, a central area of 3 mm x 3 mm was investigated together with two additional 1 mm x 1 mm areas along the samples' diagonal. The results rely on a quite good statistics: about 46k grains in the GS and 280k in the NS central area. The main outcomes were that no distinctive feature was observed between optical and thermal quality diamond, texture is very weak with no crystallographic direction perfectly aligned with the growth vertical direction. The average grain size is about 10 µm on the NS while in the range 30-40 µm on the GS. Grain boundaries are mainly coincident site lattice (CSL) boundaries of type Σ3.

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