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Etch-pit and grown-in dislocations in CVD homoepitaxial diamond

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Abstract

To evaluate grown-in extended defects included in microwave plasma chemical vapor deposition homoepitaxial (001)-oriented diamond films, formation of etch pits is attempted on the surfaces of these films, using an oxygen and hydrogen (O/H) plasma. Correspondence of the formed pits to the extended defects using Nomarski images of the film surface and the transmission birefringence images are reported.

1. Introduction

Diamond has received considerable attention for their applications in high-power, high-temperature, and high frequency diamond devices, because of its extreme physical properties such as a wide bandgap, high breakdown electric field, high thermal conductivity, negative electron affinity. However, to realize high performance diamond electronic devices, high-quality single crystal (SC) diamond wafers are preferrable. Diamond homoepitaxial growth by chemical vapor deposition CVD is a promising method to obtain high-quality SC diamond needed for diamond device fabrication, as there are principally no differences between lattice constants of the grown film and the substrate unlike heteroepitaxy. Though, to date, much effort has be devoted to improving quality of CVD SC diamond, a further decrease in extended defects should be required for diamond device applications.

For the purpose to reduce such defects, it is important to know details of lattice imperfections, particularly grown-in dislocations involved in CVD SC diamond in order to produce diamond electric devices. Among several evaluation methods including TEM, X-ray topography, etch pit analysis by the use of plasma treatment as a dry process is an effective way to observe imperfections of diamond crystal, mainly because of its high chemical inertness.

In this study, we formed etch pits on the surfaces of CVD diamond films by the oxygen and hydrogen (O/H) plasma treatment and investigated the correlation of strain patterns around the dislocations observed in the birefringence image with such etch pits.

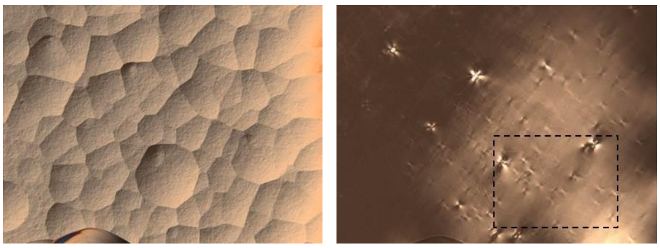

Fig. 1. (left) Nomarski image of an as-grown surface and (right) a transmission birefringence image in the same region.

2. Experiments

Etch pits on the film surface was formed in a 5 kW, 2.45 GHz microwave CVD system by an exposure of O/H plasmas from an O_2 and H_2 gas mixture. During the plasma treatment, the gas pressure was adjusted to 120 Torr, and the substrate temperature was set in the range ~850 °C, where the temperature was monitored using a two-color infrared emission thermometer through a quartz-window viewing port. The freestanding SC diamond films used in the etching experiment were produced in our laboratory, by the CVD epitaxial film growth on a high-pressure high-temperature (HPHT) synthetic type-Ib (001)-oriented diamond plate. After the film growth, freestanding films are prepared by the elimination of the Ib substrate using lift-off process. For the lift-off process, the substrate was pre-ion-implanted, in advance, before the film growth. Characterizations of the films before and after the above plasma treatment were performed using transmission birefringence micrographs taken with polarizers and Nomarski micrographs.

3. Results and discussion

Fig. 1 (left) shows a Nomarski image of an as-grown surface of our freestanding CVD homoepitaxial film. Although the overall surface is covered with rounded hillocks, the crystal defects, so-called "unepitaxial crystallites" are not observed at all. Fig. 1 (right) shows a transmission birefringence image in the same region as that in Fig. 1 (left). Since diamond itself does not have birefringence, its image should be dark on an entire of the sample. However, in

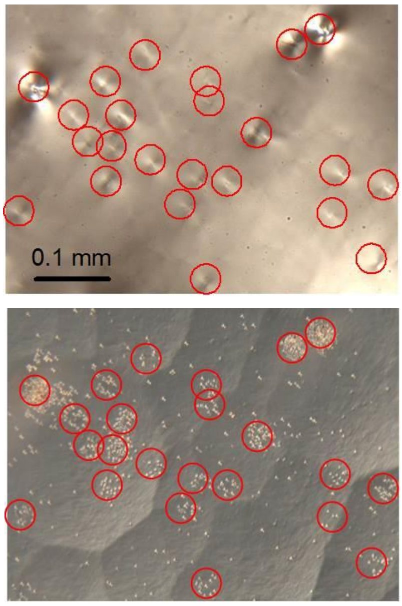

Fig. 2 (up) Transmission birefringence image of the as-grown film and (down) a Nomarski image in the same region after the etch pit formation. Both images correspond to the area indicated by the dashed square in Fig. 1 (right).

fact, there are white, patchy regions and many butterfly-shaped white dots as shown in Fig. 1 (rignt). These patterns originate from birefringence contrasts induced by local strain. For the white butterfly-shaped dots, it is suggested that the local strain induced by threading dislocations along the growth direction <001> near the center of each dot generates such birefringence patterns.

Fig. 2 (up) shows a magnified transmission birefringence image of the selected area (indicated as a dashed squire) in Fig. 1 (right). Red circles in Fig. 2 (up) show relatively clear and large, butterfly-shaped patterns, induced by large strain possibly due to high-density threading dislocations (bundle). Fig. 2 (down) shows a Nomarski image after etch pit formation in the same area as Fig. 2 (up), where many small dots correspond to the formed etch pits. The shape of an individual etch pit is inversed-pyramidal and the direction of the edge is the <110> crystallographic direction, as depicted in Fig. 3. Red circles in Fig. 2 (up) and Fig. 2 (down) are placed in the same areas on the surface each other. Intriguingly, agglomerates of etch pits inside the red circles in Fig. 2 (down) seem to correspond to the butterfly-shaped pattern in the red circles in Fig. 2 (up). These observations suggest that etch pits formed after the plasma treatment is identical with threading dislocations. On the other hand, there are isolated etch pits in the regions except for those denoted by the red circles. Some of such isolated etch pits do not seem to have clear corresponding

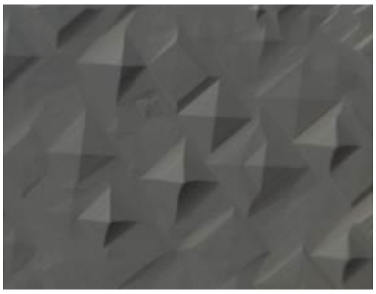

Fig. 3 Magnified image of etch pits taken by a Nomarski microscope.

dot contrasts induced by strain in the birefringence image. Only one dislocation is unlikely to induce a sufficiently large strain for us to observe a clear dot pattern in the birefringence image.

4. Conclusions

Etch pits were formed using the O/H plasma treatment on the surface of a freestanding CVD-grown (001)-oriented single crystal diamond film. Both Nomarski images of the film surface after such plasma treatment and transmission birefringence images of this film were compared. The comparison of both images suggests that one etch pit individually corresponds to one threading dislocation line in the film.

Acknowledgments

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