PASSIVATION OF GE CRYSTALS BY B_4C THIN LAYER DEPOSITION

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1. Introduction

Ge crystals are widely used optical elements for guiding, monochromatization, compressing or expanding the X-ray radiation. Synchrotrons of the last generation and intense laboratory X-ray sources such as that with Ga liquid-metal jet anode have the X-ray flux of the order of 10^{12} and 10^{9} photons/mm^2/s, respectively. These fluxes cause unwanted damage on crystals which is especially detrimental e.g. for imaging optics where any distortion of the radiation wave front results in defects of the image. Moreover, air humidity and ozone produced by X-rays cause atmospheric degradation. Repolishing of these crystals is a very expensive and time-consuming procedure. A protective thin layer of B_4C can passivate the Ge surface chemically and also enhance its mechanical resistance. It provides also a better matching of the refractive index with air or vacuum ambient compared to bare Ge surface. The ion-beam sputtering is known to produce very smooth and dense thin layers with the density approaching the bulk values. In this paper, we analyze in detail the Ge crystal damage caused by the exposure to intense X-ray beams from synchrotron and laboratory sources. Relying on this analysis, we designed and successfully tested a protective B_4C layer that was deposited by ion-beam sputtering technique.

2. Experiment

A Ge (220) crystal and Ge (100) wafer were exposed to 10.8 keV synchrotron and 9.2 keV laboratory X-ray source radiation, respectively. The Ge wafer was used as a cheaper alternative easy to handle. The Ge crystal was irradiated at 10^{11} photons/mm^2/s flux at Diamond synchrotron source for approximately 48 hours. (i.e. 2x10^{17} photons/mm^2). The Ge wafer was irradiated at 10^{9} photons/mm^2/s flux at gallium liquid-metal jet X-ray source (Excillum) for 2 weeks (i.e. 10^{16} photons/mm^2). A 5 nm thick B_4C layer was deposited in a custom-designed dual-ion beam sputtering apparatus (Bestec).

The experiment was divided into studies of the damage of Ge (220) crystal, Ge (100) wafer and Ge (100) wafer covered with B_4C protective layer. Imaging ellipsometry (Accurion) was used for mapping the surface changes of optical parameters which reflect changes in the materials density and detect presence of different elements on the surface. Stylus profilometry (Veeco, Bruker) provided surface profile along several mm with nm resolution in the vertical direction. Atomic force microscopy (AFM, Multimode 8 Bruker) was employed to obtain power spectral density function (PSDF) of the scanned area while the confocal Raman spectroscopy (WITec) was used to observe changes in crystallinity and sub-surface damage of the crystal lattice. The diagnostics was complemented by X-ray photoelectron spectroscopy (XPS) to check the elemental composition with regard to resistance of the B_4C protective layer against the etching process.

3. Results and Discussion

The imaging ellipsometry of irradiated Ge (220) performed in the Brewster angle microscopy mode with the ellipsometer parameters set to suppress reflection from the unexposed Ge surface is shown in Fig. 1. Brighter part is damaged and shows the extent of the exposed area that is approximately 5 mm. The true imaging ellipsometry of irradiated Ge (100) wafer tracks changes in the optical parameters of the irradiated area. These changes were evaluated by Cauchy models that provided a 2D map of the sample surface (Fig. 2). The result compares well with stylus profilometry (Fig. 3) and shows that the irradiated area resembles a swelling with maximum thickness around 60 nm above the unexposed area.

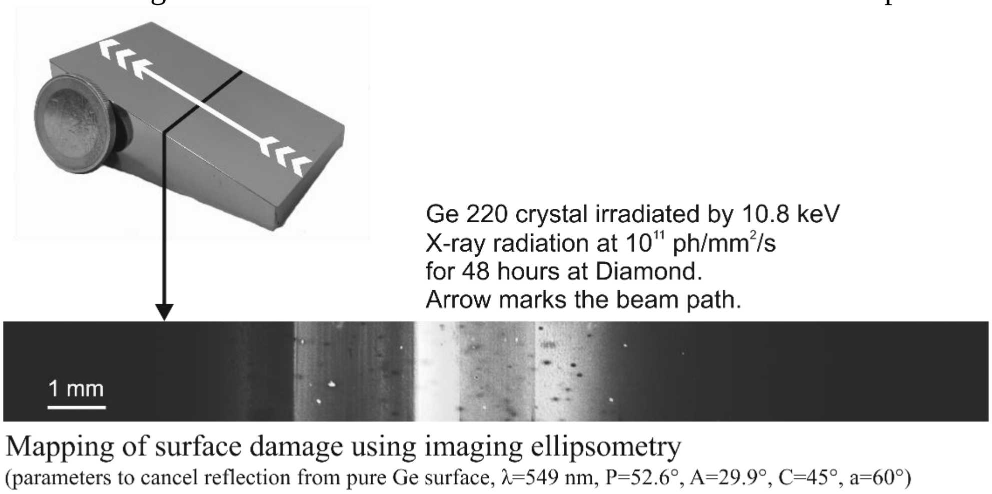

Fig.1: *Imaging ellipsometry performed like Brewster angle microscopy across the Ge crystal reveals damage approximately 5 mm wide.*

AFM measurements revealed no systematic changes of the surface roughness for unexposed and irradiated areas (Fig. 4). The confocal Raman spectroscopy was performed at the wavelength of 532 nm because of a small probing depth for Ge (about 35 nm). No difference in crystallinity was found. However, enhanced fluorescence due to contamination of unknown elements was observed (Fig. 5).

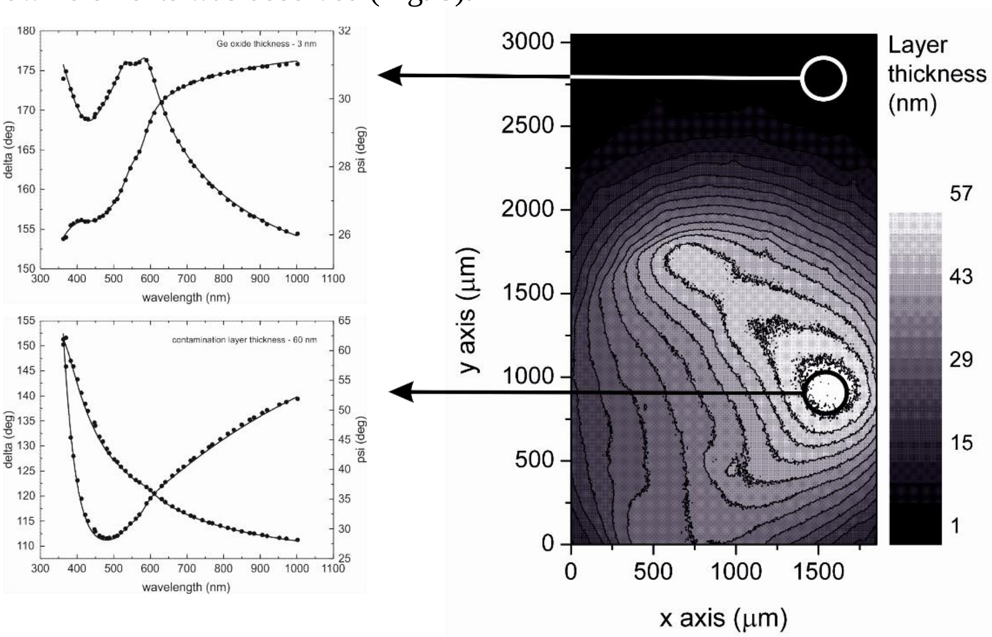

Fig. 2: *Imaging ellipsometry map evaluated from Cauchy models developed on the basis of ellipsometric measurements.*

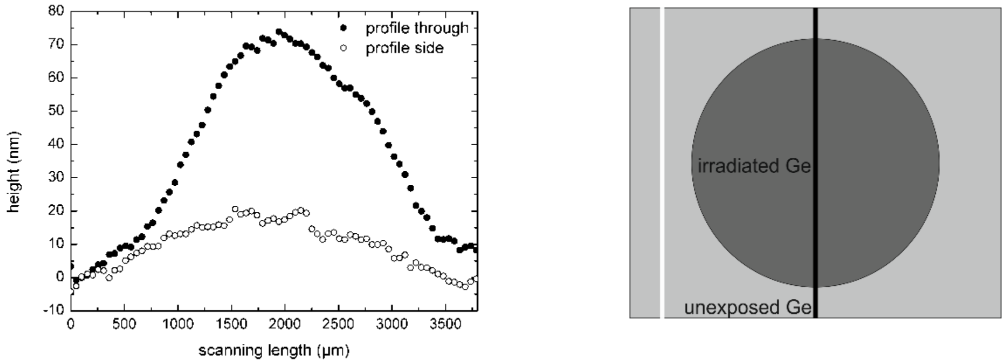

Fig. 3: *Stylus profilometry across the irradiated area and outside (left) as shown schematically (right).*

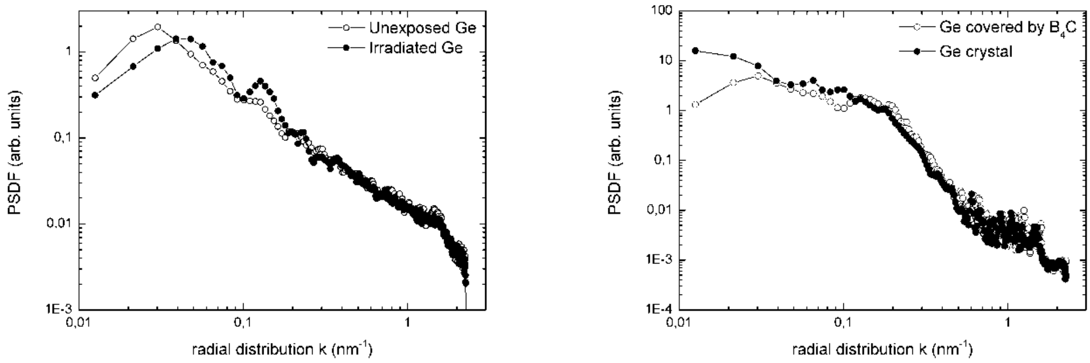

Fig. 4: *PSDF of the unexposed and irradiated Ge wafer (left); bare and capped Ge wafer (right).*

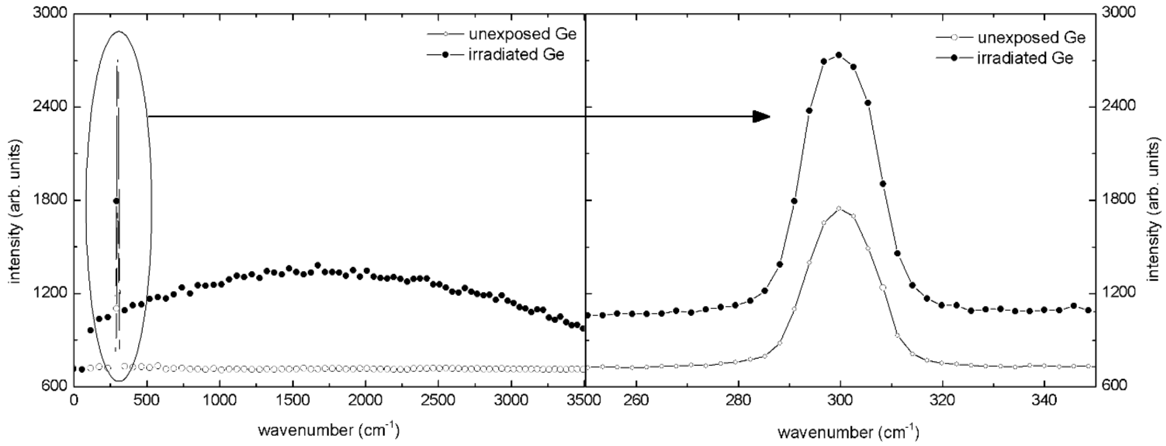

Fig. 5: *Raman spectra of the unexposed and irradiated Ge wafer with no Raman shift. Irradiated Ge shows enhanced fluorescence due to some contamination.*

The XPS showed that the irradiated area contained mainly differently bonded carbon atoms accompanied by oxygen and a small amount of nitrogen on the top. Etching process resulted in a lower amount of oxygen (Fig. 6). We also irradiated Ge wafer in UV/ozone reactor and compared the results with the X-ray irradiation. Element composition on the top of the surface was different. Differently to carbon in the composites produced by X-ray sources, UV/ozone reactor produced mainly oxides. The 5 nm thick B_4C layer deposited by ion beam sputtering proved to be fully sufficient to provide satisfactory results as the XPS shows (Fig. 7).

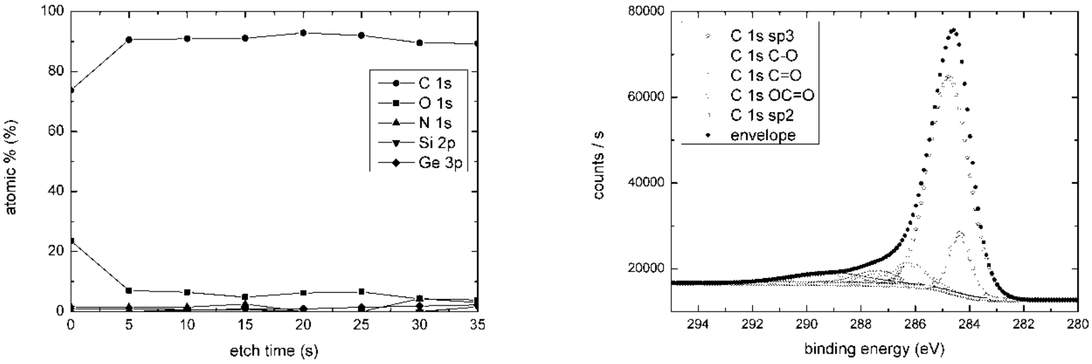

Fig. 6: *XPS measurement of the irradiated Ge wafer during etching (left) and composition of the damaged area before etching (right).*

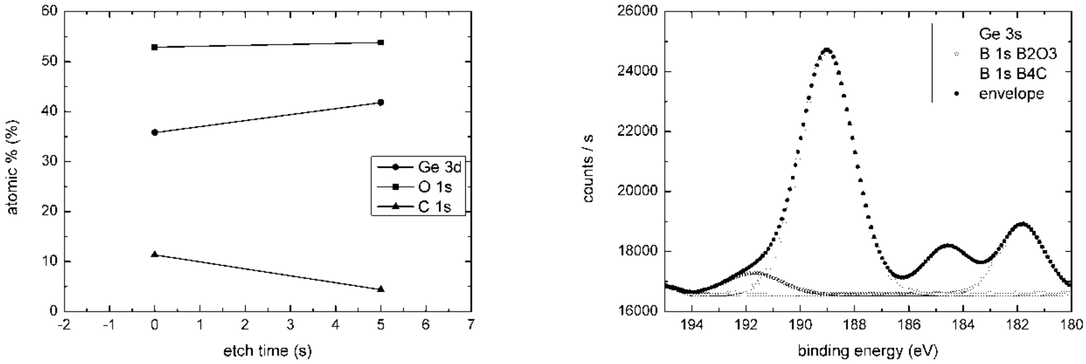

Fig. 7: *XPS measurement of the protected Ge wafer during etching (left) and composition of the damaged area before etching (right).*

4. Conclusion

We measured radiation damage of a Ge (220) crystal and Ge (100) wafer. A 5 nm thick protective B_4C layer deposited by ion beam sputtering showed to be fully sufficient to passivate chemically Ge surface, providing mechanical resistance and optical constant matching at the same time.

Acknowledgement

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