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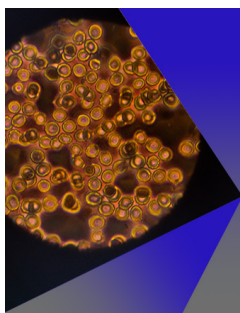
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## Optical transmittance investigation of 1-keV ion-irradiated sapphire crystals as potential VUV to NIR window materials of fusion reactors

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We investigate the optical transmittances of ion-irradiated sapphire crystals as potential vacuum ultraviolet (VUV) to near-infrared (NIR) window materials of fusion reactors. Under potential conditions in fusion reactors, sapphire crystals are irradiated with hydrogen (H), deuterium (D), and helium (He) ions with 1-keV energy and  $\sim 10^{20}\text{-m}^{-2}\text{ s}^{-1}$  flux. Ion irradiation decreases the transmittances from 140 to 260 nm but hardly affects the transmittances from 300 to 1500 nm. H-ion and D-ion irradiation causes optical absorptions near 210 and 260 nm associated with an F-center and an F<sup>+</sup>-center, respectively. These F-type centers are classified as Schottky defects that can be removed through annealing above 1000 K. In contrast, He-ion irradiation does not cause optical absorptions above 200 nm because He-ions cannot be incorporated in the crystal lattice due to the large ionic radius of He-ions. Moreover, the significant decrease in transmittance of the ion-irradiated sapphire crystals from 140 to 180 nm is related to the light scattering on the crystal surface. Similar to diamond polishing, ion irradiation modifies the crystal surface thereby affecting the optical properties especially at shorter wavelengths. Although the transmittances in the VUV wavelengths decrease after ion irradiation, the transmittances can be improved through annealing above 1000 K. With an optical transmittance in the VUV region that can recover through simple annealing and with a high transparency from the ultraviolet (UV) to the NIR region, sapphire crystals can therefore be used as good optical windows inside modern fusion power reactors in terms of light particle loadings of hydrogen isotopes and helium. © 2016 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (<http://creativecommons.org/licenses/by/4.0/>). [<http://dx.doi.org/10.1063/1.4965927>]

### INTRODUCTION

Optical plasma diagnostic techniques play an important role towards nuclear fusion development. They lead not only to a better understanding of the plasma mechanisms involved but also to the proper operation of fusion power reactors. Some examples of these techniques implemented in magnetic confinement fusion (MCF) are the impurity profile diagnostics in the vacuum ultraviolet (VUV)

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region<sup>1</sup> and the laser Thomson scattering measurements in the near infrared (NIR) region.<sup>2</sup> To be able to perform diagnostic measurements inside fusion reactors, the optical windows should be transparent in a broad range of wavelengths. Among potential candidate materials, sapphire ( $\text{Al}_2\text{O}_3$ ) crystals have an ideal characteristics for optical diagnostics in a wide wavelength range.<sup>3</sup> Investigations of ion irradiation effects on the properties of sapphire crystals are important for this particular window application in harsh radiation environments. Although ion irradiation of sapphire has already been studied,<sup>4–8</sup> the irradiation parameters are completely different from the actual conditions inside fusion reactors. Fusion reactors ideally have deuterium and tritium ions with low energies ( $<1$  keV) and high fluxes ( $\geq 10^{20} \text{ m}^{-2} \text{ s}^{-1}$ ).<sup>9,10</sup> In this regard, we investigate the optical transmittances of ion-irradiated sapphire crystals as potential VUV to NIR windows of fusion reactors. Similar to the potential conditions inside fusion reactors, the crystals are irradiated with different ions such as hydrogen (H), deuterium (D), and helium (He). Ion irradiation decreases the transmittances from 140 to 260 nm but hardly affects the transmittances above 300 nm. Results reveal important insights on the use of sapphire towards the development of optical plasma diagnostic measurements inside fusion reactors.

## EXPERIMENTAL

Commercial sapphire single crystals grown by the Kyropoulos method<sup>11,12</sup> were obtained from Optostar, Ltd. The samples have surfaces perpendicular to the *c*-axis and uniform thicknesses of 0.4 mm. The crystals were then set inside a high-flux ion beam test (HiFIT) device<sup>13,14</sup> using a molybdenum (Mo) mask. The HiFIT device can irradiate samples with low-energy and high-flux ion beams simulating the conditions inside an ideal fusion reactor.<sup>15–17</sup> Table I summarizes the different conditions of the sapphire crystals irradiated with H-ions, D-ions, and He-ions. For all experiments, the ion energy was set to 1 keV, and the ion flux was around  $10^{20} \text{ m}^{-2} \text{ s}^{-1}$ . The sample temperature was also maintained at 310 K during irradiation. After irradiation, the optical transmittances of the sapphire crystals were taken using a tabletop VUV spectrometer (Bunkoukeiki KV-202) for wavelengths from 120 to 300 nm and a UV-visible-NIR spectrometer (Hitachi U-4100) for wavelengths from 300 to 1500 nm. All transmission spectra were acquired at room temperature with background correction. The measurement errors were only less than 0.5 and 1.0 % for the VUV to UV and the UV to NIR ranges, respectively.

## RESULTS AND DISCUSSION

Figure 1 shows the VUV to UV optical transmittance spectra of the sapphire crystals irradiated with H-ions, D-ions, and He-ions. Regardless of the ion species, the ion-irradiated crystals exhibit lower transmittances from 140 to 260 nm and improved transmittances from 260 to 300 nm compared with the non-irradiated sample. The H-ion and D-ion irradiated sapphire crystals are also observed to have absorption bands above 200 nm. The differential spectra of the VUV to UV optical transmittances with 10-nm smoothing is shown in Fig. 2. Similar to electron spectroscopy, the differential spectra aid in distinguishing very small absorption peaks induced by ion irradiation. Both H-ion and D-ion irradiated sapphire crystals exhibit absorption peaks near 210 and 260 nm. On the other hand, the He-ion irradiated crystal has no observable absorption peak above 200 nm. For the UV to NIR optical transmittance spectra, no difference among the non-irradiated and irradiated sapphire crystals are observed. All samples exhibit high transparency of more than 80 %

TABLE I. Different conditions of sapphire crystals irradiated with hydrogen (H), deuterium (D), and helium (He) ions.

Ion species	Ion beam components (%)			Ion flux ( $\text{m}^{-2} \text{ s}^{-1}$ )	Irradiation time (h)	Ion fluence ( $\text{m}^{-2}$ )
Hydrogen (H)	H <sup>+</sup> (27)	H <sup>2+</sup> (27)	H <sup>3+</sup> (46)	$1.18 \times 10^{20}$	3.0	$1.27 \times 10^{24}$
Deuterium (D)	D <sup>+</sup> (45)	D <sup>2+</sup> (20)	D <sup>3+</sup> (35)	$6.69 \times 10^{19}$	3.0	$7.23 \times 10^{23}$
Helium (He)	He <sup>+</sup> (100)			$3.21 \times 10^{19}$	3.0	$3.47 \times 10^{23}$



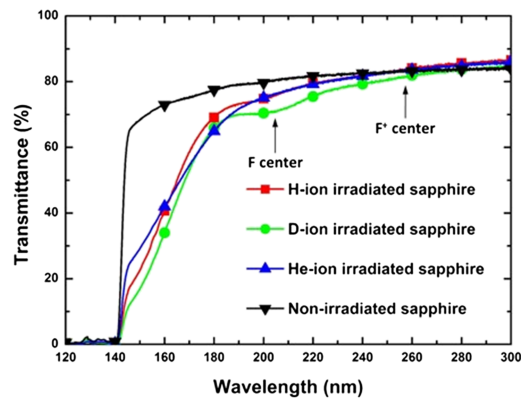


FIG. 1. VUV to UV optical transmittance spectra of non-irradiated and ion-irradiated sapphire crystals. The ion-irradiated crystals exhibit lower transmittances compared with the non-irradiated sample.

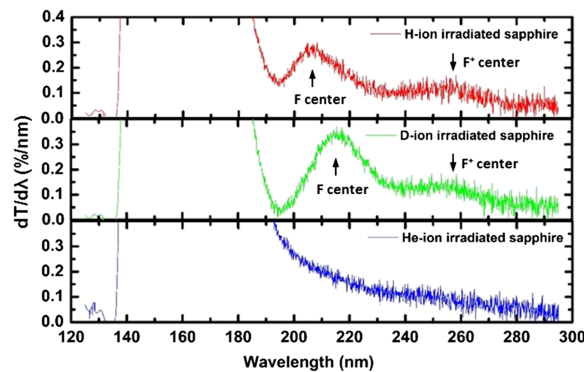


FIG. 2. Differential VUV to UV optical transmittance spectra of sapphire crystals after H-ion, D-ion and He-ion irradiation. The H-ion and D-ion irradiated crystals exhibit absorption bands near 210 and 260 nm.

from UV to NIR. Ion irradiation hardly affects the optical transmittance of sapphire in wavelengths above 300 nm.

The non-irradiated and ion-irradiated sapphire crystals exhibit different optical transmittance spectra from 140 to 300 nm. The improved transmittances of the ion-irradiated sapphire from 260 to 300 nm possibly indicates a crystal surface cleaned by ion irradiation. On the other hand, their lower transmittances from 140 to 260 nm can be associated with different factors. The optical absorption bands of the H-ion and D-ion irradiated sapphire crystals near 210 and 260 nm can be attributed to F-type centers.<sup>18</sup> F-type center is well known as a defect in the research of sapphire crystals. Energetic particle irradiation induces oxygen vacancies and charges trapped at the defects.<sup>19–21</sup> Previous studies report that an F-center and an F<sup>+</sup>-center cause absorption bands at 205 and 258 nm, respectively.<sup>22,23</sup> In order to confirm this finding, the H-ion irradiated crystal was annealed isochronally in air. After annealing, F-type centers are expected to decrease through lattice reordering or oxygen (O) atom recapture.<sup>24–26</sup> Figure 3 shows the optical absorption coefficients of the H-ion irradiated sapphire crystal at 210 nm after isochronal annealing. A non-irradiated sample is also similarly investigated for comparison. The sapphire crystals were annealed for 10 min in ambient air at temperatures from 623 to 1223 K with 50 K intervals. The optical absorption of the non-irradiated sample remains almost the same at different annealing temperatures. On the contrary, the optical absorption of the H-ion irradiated crystal starts to decrease at 1023 K. A similar trend was also observed for the optical absorption of the non-irradiated and H-ion irradiated crystals at 260 nm. The decrease in absorption indicates an induced Schottky defect, a state where O atoms scattered by ion irradiation escape from the sapphire crystal. According to H. Peisl *et al.*,<sup>27</sup> F-type centers can be classified into Schottky and Frenkel defects, and the absorptions of additive-colored and of radiation-exposed samples are

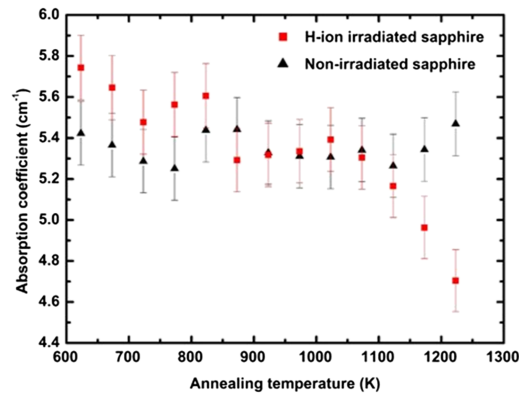


FIG. 3. Optical absorption coefficients at 210 nm of non-irradiated and H-ion irradiated sapphire crystals after isochronal annealing in air at different temperatures. The optical absorption of the non-irradiated sample remains almost the same, while that of the H-ion irradiated crystal starts to decrease at 1023 K.

caused by Schottky and by Frenkel defects, respectively. K. H. Lee *et al.* also reported that the defects of the additive-colored sapphire are thermally stable and start to disappear through annealing at a temperature ( $\sim 1400$  K) higher than that of the radiation exposure ( $\sim 500$  K).<sup>24</sup> For this case, the fact that the absorption decreased at relatively high temperatures ( $>1000$  K) shows the possibility of an existence of Schottky defects. With a low ion irradiation energy of 1 keV, O vacancies are induced only near the sample surface, and O atoms easily escape from the crystal. From the Stopping and Range of Ions in Matter (SRIM),<sup>28</sup> the estimated penetration depths of the ion irradiation conditions range only from 5 to 10 nm. Any radiation-induced damage is therefore limited within 10 nm from the surface of the sapphire crystals. This explains why the annealing temperature where the absorption coefficient starts to decrease ( $\sim 1000$  K) is lower than that of what have been previously observed ( $\sim 1400$  K).<sup>24</sup>

In addition, the D-ion irradiated sapphire crystal exhibits a more intense 210-nm absorption peak compared to the H-ion irradiated sample. The ion fluence during D-ion irradiation is smaller than the ion fluence during H-ion irradiation. The intense absorption peak can then be explained by the amount of O vacancies formed during irradiation. From simple elastic collision theory, the energy deposited to the O atoms from the irradiated ions in one collision is higher for D-ions than for H-ions. D-ion irradiation creates O vacancies more frequently than H-ion irradiation. As the optical absorption becomes intense, it eventually broadens. Hence, the intense peak of the differential spectrum of the D-ion irradiated crystal is shifted to a longer wavelength as shown in Fig. 2.

Compared to the H-ion and D-ion irradiated sapphire crystals, the He-ion irradiated sample does not exhibit any absorption bands near 210 and 260 nm. Similar phenomenon is observed and reported by D. M. Gruen *et al.*<sup>5</sup> This can be explained by the differences of the radii of the ion irradiation species. F- type centers are probably formed by ion species which do not have electrons, such as H<sup>+</sup>-ions and D<sup>+</sup>-ions. H<sup>+</sup>-ions and D<sup>+</sup>-ions have radii (1 fm) much smaller than that of He<sup>+</sup>-ion (31 pm)<sup>29</sup> and of other ion species. With small radii, H<sup>+</sup>-ions and D<sup>+</sup>-ions can easily be incorporated in the crystal lattice and can create F-type centers than He<sup>+</sup>-ions. As the ion radius becomes large, the ions instead ablate or etch the surface of the sapphire crystal during irradiation. The surfaces of the ion irradiated sapphire crystals are investigated using an atomic force microscope (AFM). He-ion irradiation causes a depth of 500 nm from the crystal surface, while D-ion irradiation only causes a depth of 300 nm.

From 140 to 180 nm, all irradiated sapphire crystals exhibit decreased optical transmittance regardless of the ion species. This transmittance decrease can be associated to ion plasma etching. In contrast, proton-irradiated sapphire crystals do not exhibit decreased transmittances in the same wavelength range.<sup>6</sup> The proton-irradiation conditions have higher energy (200 keV) and lower ion fluence (by three orders of magnitude) than our experiments. High-energy ions hardly deposit energy near the sample surface, thus the irradiation effects are possibly too small to be observed.

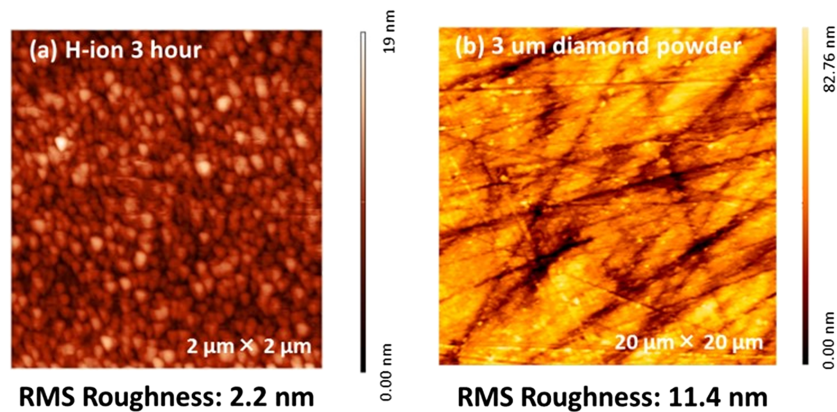


FIG. 4. AFM images (surface topography) of (a) H-ion irradiated and (b) diamond-polished sapphire crystals. The H-ion and diamond-polished crystals have RMS roughness values of 2.2 and 11.4 nm for a  $2\text{-}\mu\text{m} \times 2\text{-}\mu\text{m}$  and a  $20\text{-}\mu\text{m} \times 20\text{-}\mu\text{m}$  scanning area, respectively.

Figure 4(a) shows the AFM image of the H-ion irradiated sapphire crystal. The surface of the H-ion irradiated crystal exhibit nanometer-sized globules and has a root-mean-square (RMS) roughness of 2.2 nm for a  $2\text{-}\mu\text{m} \times 2\text{-}\mu\text{m}$  scanning area. Since surface roughness can greatly affect the optical transmittance at shorter wavelengths,<sup>30,31</sup> it is suggested that a decreased transmittance is due to light scattering on the sample surface. The VUV optical transmittance spectra of a sapphire polished with 2 to 3- $\mu\text{m}$  diameter diamond powders have also been previously reported.<sup>32</sup> For comparison, we studied sapphire crystals polished with 3- $\mu\text{m}$  diameter diamond powders. Figure 4(b) shows the AFM image of the diamond-polished sapphire crystal. The surface of the polished sample exhibit scratches and has a RMS roughness of 11.4 nm for a  $20\text{-}\mu\text{m} \times 20\text{-}\mu\text{m}$  scanning area. The non-irradiated and diamond-polished samples also underwent similar annealing procedure at 973 K for 10 min to remove possible contributions of intrinsic defects and F-type centers. Figure 5 shows the VUV to UV optical transmittance spectra of the non-irradiated, diamond-polished, and H-ion irradiated sapphire crystals before and after annealing. The optical transmittance of the non-irradiated crystal does not change after annealing. On the other hand, the optical transmittances of the diamond-polished and H-ion irradiated crystals improve after annealing. Although with different transmittance values, the diamond-polished and H-ion irradiated samples have similar spectral shapes before annealing and exhibit a similar increase in transmittance after annealing. The similar increase of optical transmittance after annealing cannot be associated with lattice reordering nor with the samples' intrinsic defects. The transmittance increase is a direct result of the change in the surface topography of the

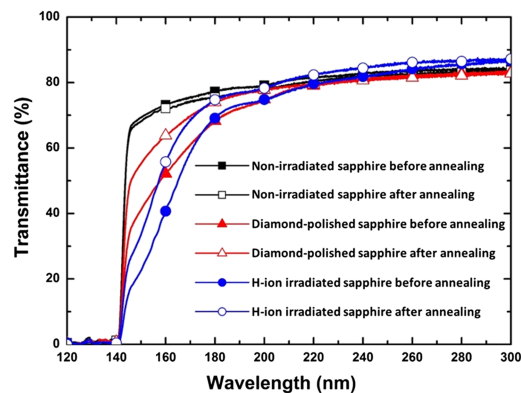


FIG. 5. VUV to UV optical transmittance spectra of non-irradiated, diamond-polished and H-ion irradiated sapphire crystals before and after annealing at 973 K for 10 min. The optical transmittances of the diamond-polished and H-ion irradiated samples improve after annealing.

sapphire crystals. Crystal surfaces of sapphire transform at around 473 K.<sup>33</sup> This is probably due to the surface diffusion.<sup>34</sup> With an annealing temperature of 973 K, the surfaces of the diamond-polished and H-ion irradiated crystals are flattened by the surface diffusion and transform thereby affecting light scattering and improving the optical transmittances.

## CONCLUSION

In conclusion, the optical transmittances of the ion-irradiated sapphire crystals were investigated for potential VUV to NIR window applications. Sapphire crystals were irradiated with H-ions, D-ions, and He-ions with 1-keV energy and  $10^{20}\text{-m}^{-2}\text{ s}^{-1}$  flux simulating the potential conditions of fusion reactors. The ion-irradiated crystals exhibited lower transmittances from 140 to 260 nm compared with a non-irradiated sample. H-ion and D-ion irradiation caused optical absorptions near 210 and 260 nm which are attributed to an F-center and an  $\text{F}^+$ -center, respectively. These ion-induced F-type centers were classified as Schottky defects that can be removed through annealing above 1000 K. D-ion irradiation also results to a stronger absorption which can be related to the energy deposited from the irradiated ions to the O atoms in one collision. On the other hand, He-ion irradiation did not cause any optical absorptions near 210 and 260 nm due to He-ions' ionic radius. With a radius larger than those of  $\text{H}^+$ -ions and  $\text{D}^+$ -ions, He-ions were more difficult to be incorporated in the crystal lattice in order to create F-type centers. Moreover, the decrease in optical transmittance of the ion-irradiated sapphire crystals from 140 to 180 nm was related to the light scattering on the crystal surface similar to a diamond-polished sample. During irradiation, the ion beam ablates or etches the surface of the crystal thereby affecting the optical properties especially at shorter wavelengths. Although this is the case, the optical transmittance of the ion-irradiated samples in the VUV region improved after annealing above 1023 K as the surface of sapphire starts to be flat by the surface diffusion at around 473 K. Since the decrease of the VUV to UV optical transmittances recovers through annealing and the UV to NIR optical transmittance is hardly affected by ion irradiation, sapphire crystals can therefore be used as good optical window materials with transparency in a wide range of wavelengths. These results yield important insights towards the development of optical plasma diagnostic measurements inside future fusion reactors.

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