

He⁺ Irradiation Induced Cracking and Exfoliating on the Surface of Ti₃AlC₂

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M_{n+1}AX_n (MAX) phases, where M is an early transition metal, A is an A-group element (mostly IIIA and IVA), and X is either C and/or N, have been attracting much attention [1, 2]. A MAX phase usually has a hexagonal structure with the P₆₃/mmc symmetry. Its structure is comprised of two-dimensional closed packed A layers linked by Ti₆C octahedra. The MAX phases show a combined properties of both metal and ceramic, such as good electrical and thermal conductivity as well as good irradiation damage tolerance [3], which leads them to be candidate materials for future fission and fusion reactors with much higher burn-up requiring the materials to survive up to 200 dpa [4]. In the present study, we report our systematical study on the surface microstructure evolution of Ti₃AlC₂ bulk samples irradiated with a 400 keV He⁺ at both of RT and 500 °C.

The Ti₃AlC₂ samples were synthesized by a hot pressing method. All samples for helium irradiation were mechanically grinded and then polished to obtain smooth surface. He irradiation experiments were performed at energy of 400 keV at RT and 500 °C, respectively, with He fluence up to 2.0×10^{17} He⁺/cm² at a helium flux of 0.78×10^{14} He⁺/(cm²·s). SRIM calculation shows that the damages of 2.1 dpa, 4.2 dpa and 8.4 dpa at the depth of 1330 nm are correspondence with the fluences of 0.5×10^{17} He⁺/cm², 1.0×10^{17} He⁺/cm² and 2.0×10^{17} He⁺/cm², respectively. The crystal structures of the samples were examined by grazing incidence X-ray diffraction (GIXRD). The surface microstructures of the original and irradiated samples were studied using a scanning electron microscope (SEM). The TEM specimens, prepared by focused ion beam, were examined by bright-field image (BF) in a TEM.

Figure 1 shows a series of SEM images for the samples irradiated at RT (first row) and 500 °C (second row), respectively. On the surface of the sample irradiated to 2.1 dpa at RT, a great many of intragranular cracks were seen to form after irradiation. Very interestingly, the cracks grow parallel to the *c* direction of the Ti₃AlC₂ crystal. With the increase of He fluence, surface exfoliating starts to occur after irradiation to 4.2 dpa. It was evident that the number of orientated cracks and area density of surface exfoliating are remarkably increased with irradiation fluence increasing up to 8.4 dpa. However, no cracks were seen in the sample irradiated at 500 °C. Figure 2 shows the BF images of specimens irradiated to 0.5×10^{17} He⁺/cm² at RT and 500 °C. As shown in Fig. 2(a), it is clear that the cracks pass through the whole irradiated layer and extend to the non-irradiated bulk. It is determined that the crack grows along the (100) crystal plane of the Ti₃AlC₂ phase.

At RT, reduction of *a*-LP and expansion of *c*-LP, leading to the volume expansion, were observed in the sample irradiated to 2.1 dpa, as shown in Fig. 3(a). However, with the fluence increasing from 2.1 dpa to 8.4 dpa, the reduction of *a*-LP changes from 0.65% to 0.16% and the expansion of *c*-LP drops from 3.61% to 1.09%. At 500 °C, the continuous reduction of *a*-LP and expansion of *c*-LP were found with

the increasing He fluence, which is contrary to the result from the RT irradiation. The irradiation could produce a lot of point defects, which leads to the shrinkage of TiC cell volume, and then causes the decrease of *a*-LP of Ti₃AlC₂ phase. It was proposed that the formation of orientated cracks is strongly related to the *a*-LP reduction.

References:

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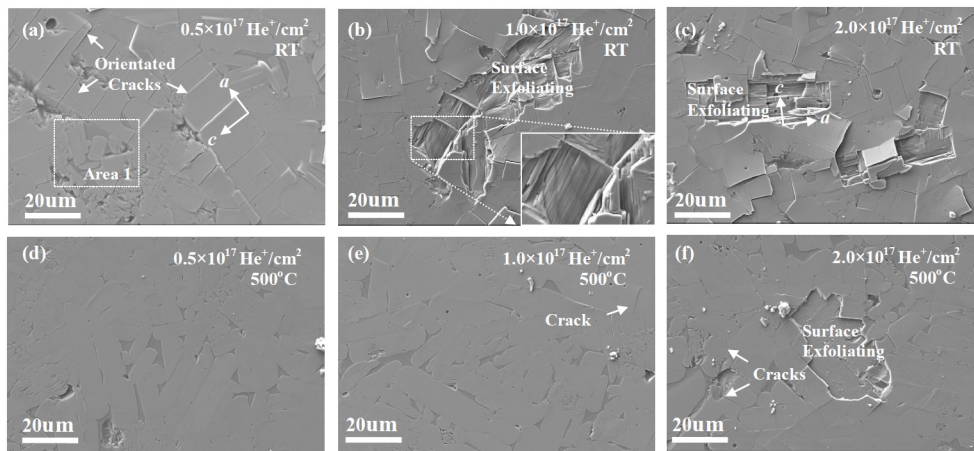


Figure 1. SEM images of Ti₃AlC₂ sample after He irradiation up to $2.0 \times 10^{17} \text{ He}^+/\text{cm}^2$ at RT ((a)-(c)) or 500 °C ((d)-(f)).

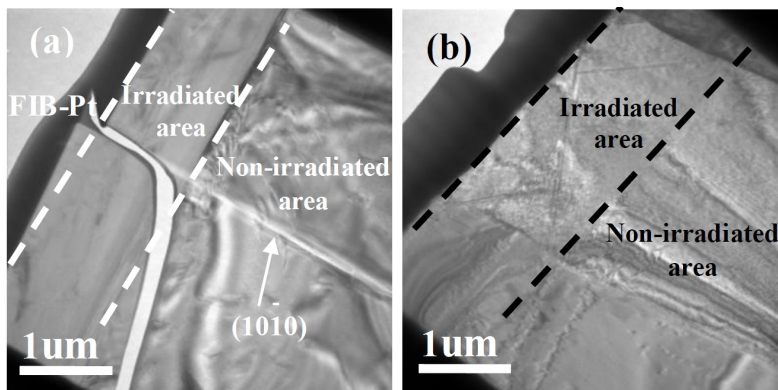


Figure 2. BF images of specimens irradiated to $0.5 \times 10^{17} \text{ He}^+/\text{cm}^2$ at RT (a) and 500 °C (b). Helium irradiation induced an irradiated layer with a thickness around 1330 nm, which is followed by the non-irradiated bulk.

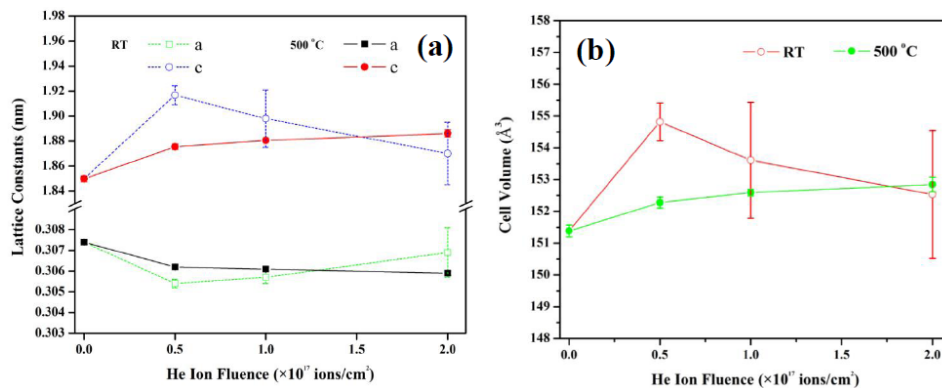


Figure 3. The *a*-LP, *c*-LP (a) and cell volume (b) of Ti₃AlC₂ change as a function of irradiation temperature and fluence.