

Irradiation resistance properties studies on helium ions irradiated MAX phase Ti_3AlC_2



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ABSTRACT

The study presents an investigation of irradiation resistance properties of Ti_3AlC_2 under 500 keV He ions irradiation with the doses ranging from 5.0×10^{16} to 1.0×10^{18} ions cm^{-2} at certain temperatures, like room temperature (RT), 300 and 500 °C. X-ray diffraction (XRD) and Transmission electron microscopy (TEM) are used to study the evolution of structural damage and the behavior of deposited He ions respectively. XRD analysis reveals that for the highest dose irradiation (~ 52 dpa at peak), no amorphization occurs. And the structural recovery of Ti_3AlC_2 is more significant accompanied with the gradual disappearance of the irradiation-induced TiC phase as the temperature rises from RT to 300 and to 500 °C with the same dose irradiation. TEM observations show that He bubbles appear in the shapes of sphere, string and platelet but no big bubbles are formed for all irradiations. Moreover, no large cracks form in the sample implanted with the highest helium concentration of $\sim 6.4 \times 10^5$ appm.

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1. Introduction

As one of the typical MAX phases, where M is an early transition metal, A is an element from the IIIA or IVA groups, and X is carbon or nitrogen, Ti_3AlC_2 is characterized by near close-packed Ti layers interleaved with Al layers, with the C atoms filling the octahedral sites between Ti atoms (Ti_6C) [1]. So Ti_3AlC_2 combines the properties of both metal and ceramic, such as high-temperature stability (1460 °C at least), good ductility, excellent thermal shock resistance and intrinsic damage tolerance [1–4]. Therefore, it has been considered as a remarkable fuel cladding or structural material to be used in future fission and fusion reactors, such as the gas-cooled fast reactor (GFR) and international thermonuclear experimental reactor (ITER).

Several works concerning irradiation effect of Ti_3AlC_2 and similar MAX phase Ti_3SiC_2 have been conducted in recent years and most of them are at low damage displacement with high energy ions irradiation. Flem et al. [5] and Whittle et al. [6] confirmed that $\text{Ti}_3(\text{Si}, \text{Al})\text{C}_2$ remained crystalline at 6.67 and ~ 25 dpa respectively except for some loss of its nanolamellar structure. It was also shown by Whittle group that Ti_3AlC_2 showed more tolerance to irradiation damage than Ti_3SiC_2 . Liu et al. [7,8] have demonstrated an evident increase in hardness and the formation of $\beta\text{-Ti}_3\text{Si}_{0.90}\text{C}_{0.10}$

$\text{Al}_{0.10}\text{C}_2$ after irradiation by 74 MeV Kr and 92 MeV Xe ions as well as a complete recovery of properties at high temperature especially at 800 °C. However, the researches regarding the helium effect on Ti_3AlC_2 are rare though in (n, α) transmutation reactions [9] of nuclear materials plenty of helium atoms are produced, which result in deleterious effects such as swelling, blistering and mechanical properties degradation of materials [10]. Wang et al. [11] found the transformation of He bubbles shape from sphere to string in Ti_3AlC_2 with 50 keV He irradiation at room temperature. But there is no work studying the He behaviors in Ti_3AlC_2 at high irradiation temperature. This work is carried out to develop an understanding of the irradiation resistance of Ti_3AlC_2 and the evolution of helium bubbles under different He ions irradiation conditions.

2. Experimental details

The polycrystalline Ti_3AlC_2 was provided by Ningbo Institute of Materials Technology & Engineering, Chinese Academy of Sciences. To obtain the material, a mixture powder of Ti, Al and C with the stoichiometric proportion of 3:1.2:1.9 should be put into a cylindrical graphite mould with 5 min sintering at 1200 °C in the flowing argon gas with an applied pressure of 30 MPa by the spark plasma sintering technique. The specimens were cut into parallel-epipeds of about $10 \times 10 \times 2 \text{ mm}^3$, and one face was polished with diamond spray of particle size down to 1 μm . The parallelepiped

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were cleaned by rinsing in ultrasonic baths of acetone and ethanol and subsequently irradiated on the polished surface.

Irradiation experiments of the Ti_3AlC_2 ceramic were performed at 320 keV multi-discipline research platform for highly charged ions equipped with an ECR (electron cyclotron resonance) ion source in the Institute of Modern Physics, Chinese Academy of Sciences (IMP, CAS), Lanzhou. The samples are irradiated with 500 keV He^{2+} ions and the mean flux was about $5 \mu\text{A}$ (1.1×10^{13} ions $\text{cm}^{-2} \text{s}^{-1}$). The irradiation conditions are summarized in Table 1. The theoretical results of 500 keV He irradiation with a dose of 1.0×10^{18} ions cm^{-2} , i.e. penetration depth, displacement damage (dpa, displacement per atom) and He concentration are calculated by SRIM 2008 [12] full damage cascade simulations (Fig. 1). The threshold displacement energies are determined as 25 eV for Ti, 25 eV for Al and 28 eV for C.

All samples were characterized by low-incidence X-ray diffraction (LI-XRD) using a Philips X'pert diffractometer with Cu $\text{K}\alpha$ radiation. The X-ray diffraction data were collected between 20° and 85° in 2θ scale under an incidence of 5° with the corresponding maximum depth of about $1.5 \mu\text{m}$. The data from the irradiated zone was covered in XRD data according to the SRIM calculation results. The irradiated specimens are prepared by the cross-sectional specimen technique and then thinned by 3.0 keV Ar^+ ion beam milling to form a wedge for sufficient electronic transparency, so that damage level as a function of depth could be obtained directly. The TEM observation is performed with an FEI TECNAI G² F30 and all micrographs are taken at 300 keV.

3. Results

3.1. XRD patterns

XRD diffraction patterns of the virgin sample and all irradiated samples are shown in Fig. 2. With increasing doses of samples irradi-

Table 1
The parameters of irradiation experimental condition.

Temperature	Room temperature	300 °C	500 °C
Irradiation dose (ions cm^{-2})	5.0×10^{16} 1.0×10^{17} 3.0×10^{17} 1.0×10^{18}	3.0×10^{17}	3.0×10^{17}

ated at RT, an evident drop of peak intensity and rise of peak width are found, which reveals some loss of crystallinity. Both (102) and (112) peaks begin to shift remarkably to higher 2θ and (105) peak to lower 2θ with the similar trend when the irradiation dose reaches 5.0×10^{16} ions cm^{-2} . This phenomenon is principally caused by the presence of new TiC phase whose corresponding peaks are (111), (200) and (220). It has been previously confirmed that the TiC phase is produced by nuclear shock in irradiated Ti_3SiC_2 or Ti_3AlC_2 [11,13–15]. In addition, the relative intensities of (102) and (103) to (101) peaks become larger as the irradiation dose increases. It might be induced by the phase transformer from α - Ti_3AlC_2 to β - Ti_3AlC_2 and the similar transformation has been found in Ti_3SiC_2 after irradiation [8,14]. Furthermore, Farber et al. have verified these polymorphic phase transformations in $\text{Ti}_{n+1}\text{AX}_n$ ($n \geq 2$), such as Ti_3SiC_2 , Ti_3AlC_2 and Ti_4AlN_3 through HRTEM [16].

For the samples irradiated with the dose of 3.0×10^{17} ions cm^{-2} at various temperatures, the peak intensity, peak position and peak width recovered progressively as the irradiation temperature increased. That phenomenon serves as proof of the beneficial effect of temperature in reducing irradiation damage. Moreover, the new phases formed with irradiation at RT cannot be detected when the irradiation temperature increases to 500°C . Considering the noteworthy thermal stability of the new phase TiC up to 820°C [14], we conclude that the possibility of TiC formation is eliminated by the recombination of irradiation effects during collision cascade formation.

3.2. TEM results

The TEM analysis confirmed the presence of polycrystalline Ti_3AlC_2 with a grain size of $\sim 1.5 \mu\text{m}$ (not shown). And an overview of the nanolamellar structure and damage area is shown in Fig. 3. In the virgin sample, the periodic multilayer structures are evident (Fig. 3a). With the irradiation dose of 5.0×10^{16} ions cm^{-2} (~ 2.62 dpa), it is detected obviously that the depth of the damaged area ranges from 1.134 to $1.354 \mu\text{m}$ in accordance with the SRIM calculation results. And some isolated nanometer-scale spherical helium bubbles are spread at the damaged region with an average radius of ~ 0.6 nm around the maximum concentration (3.2×10^4 appm) depth of implanted He atoms (Fig. 3b). The bubble radius presents a Gaussian-like distribution and the mean radius is determined by measuring the radius of a total of 80 bubbles [11]. In addition, the damaged area contains clusters of

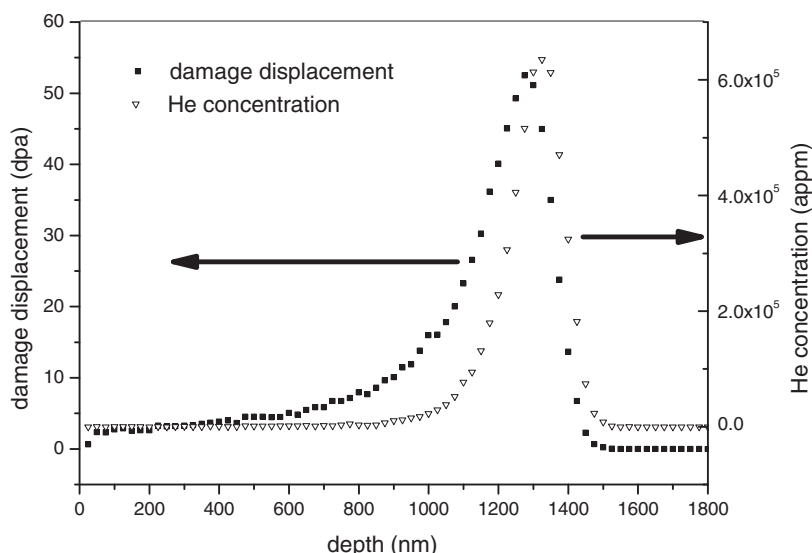


Fig. 1. He concentration and number of displacements per atom induced by 500 keV He irradiation at the dose of 1.0×10^{18} ions cm^{-2} .

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