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Characterisation of dual ion beam irradiated yttria-stabilised zirconia by specific analytical techniques

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ABSTRACT

The combined effect of dual ion beam irradiated yttria-stabilized zirconia was investigated through Rutherford backscattering spectrometry/channeling (RBS/C), high resolution X-ray diffraction (HRXRD), atomic force microscopy (AFM) and transmission electron microscopy (TEM). Compared with other experimental results of single ion beam irradiation, a multistep damage accumulation model can also explain the irradiation effects of dual ion beam. Irradiation damage created by Ar + He ions are simply additive and no synergy effect has been observed. The variation trends of step height and displacement damage are similar. The synergic effects of displacement damage between heavy recoil atoms and α-particle in nuclear waste matrices will not cause more serious damage than the sum of two kinds of ions. The two experimental damage peaks are consistent with those calculated using stopping and range of ions in matter (SRIM). Phase stability and irradiation resistance is further confirmed by high resolution transmission electron microscopy (HRTEM).

1. Introduction

Yttria-stabilized zirconia (YSZ) has high radiation resistance and high chemical stability because yttria can stabilize the cubic phase. YSZ can be applied for the intermatrix layer of nuclear fuel [1,2] and for the covering and storage of nuclear waste [3]. YSZ is a representative model system for the investigation of irradiation effects due to the representative damage behaviors by ion irradiation. Ion beams provide very efficient tools for the evaluation of nuclear oxide based ceramics. Heavy ions [4–6] and He ions irradiation [7–9] are widely used to simulate the irradiation behavior induced by neutron.

Oxide based ceramics were identified as potential candidates for nuclear waste immobilization and/or transmutation in nuclear reactors [10,11]. As an important nuclear reactors material, YSZ will be exposed to severe irradiation environment of atomic displacement damage (fission fragments, alpha recoils) and accumulation of a large number of helium [12]. Dual ion beam irradiation is a useful and important method to investigate the synergic effects of damage and/or He created in the actual environment of reactors. Helium is released from vacancy defects and is out-diffused under dual ion beam (Zr and He ions) irradiation at room temperature [13]. No synergic effects between nuclear energy loss (Sn) and electronic energy loss (Se) appeared in dual heavy ion beam irradiated YSZ [14]. Both heavy recoil atoms and α-particle irradiation produces atomic displacement damage through nuclear energy loss in nuclear waste matrices. The synergic effects of displacement damage between heavy recoil atoms and α-particle irradiation have not been studied. The aim of the work is to understand the synergic effects of irradiation damage caused by dual ion beam irradiation (Ar and He ions).

2. Experiment

The ZrO_2 ⟨100⟩ single crystal used in this investigation was doped with 6.5 mol% yttria in order to stabilize the cubic phase. 300 keV Ar and 100 keV He ions were irradiated singly or dually on the YSZ specimens. In this low energy range the nuclear energy loss was the main factor. For single ion implantation, half area of the original specimen was masked with a sharp blade as showed in Fig. 1a. For dual ion implantation, Ar ions were irradiated first followed by He ions. And two angled sharp blades were used in dual ion implantation as showed in Fig. 1b. Implantations were carried out at room temperature (RT) in a random direction (by tilting the crystals with an angle of 7° relative to the surface normal) with Ar^{2+} and He^{+} ion fluences in the range of 5×10^{14}–3×10^{16} cm^{-2} and 8×10^{15}–1×10^{17} cm^{-2}, respectively. The beam current density of Ar and He ions was 0.2 μA cm^{-2} to keep the specimen at RT as far as possible. The profiles of injected ions

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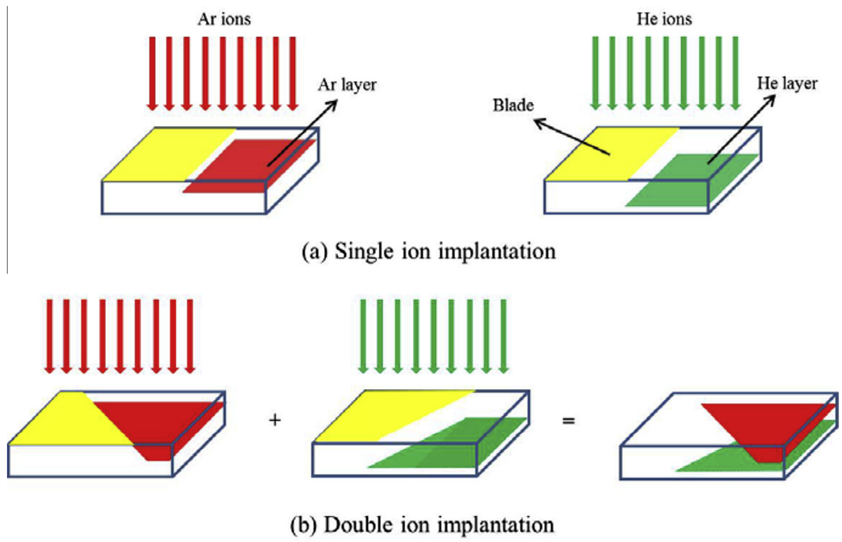

Fig. 1. Schematic of single and dual ion beam (Ar, He) irradiation process.

and irradiation damage were calculated using SRIM 2013 [15]. The ion induced damage in the YSZ crystal was determined by RBS/C measurements with a collimate 2.022 MeV He^+ beam. The energy resolution was around 15 keV. The lattice strain was measured by HRXRD. θ–2θ scan was recorded in the vicinity of (400) Bragg reflection. This high resolution diffractometer uses a Ge (220) double-crystal monochromator providing a parallel and monochromatic ($Cu_{K\alpha 1}$ radiation, k = 0.15406 nm) incident X-ray beam. The swelling of the ion implanted specimens were measured using step height method by Agilent 5500 atomic force microscopy (AFM) in the tapping mode. A second-order flattening routine was used in all the obtained images. TEM specimen was prepared by conventional ion-thinning instrument (Precision Ion Polishing System Gatan 691). Cross-sectional TEM micrographs were taken to observe the crystal morphology using a H-9000NAR transmission electron microscope, with an accelerating voltage of 300 kV. HRTEM investigation was used to obtain information about the structure (organization in the atomic scale of the layers).

3. Results

3.1. SRIM calculation

Ion range and damage distribution calculated by SRIM is shown in Fig. 2. The distribution of Ar and He ions take arbitrary units. SRIM calculations indicate that the Ar^{2+} and He^+ implantation results in a maximum damage in the depth of about 150 and 350 nm, respectively, considering displacement energy of 40 eV for both Zr and O atoms [16]. According to SRIM, damage density was 5×10^{14}, 5×10^{15} and 2×10^{16} cm^{-2} for Ar ions implantation, 8×10^{15}, 5×10^{16} and 1×10^{17} cm^{-2} for implanted He ions. Later damage (dpa) is used in order to express conveniently.

3.2. RBS data

Fig. 3 displays selected RBS/C spectrum of pristine and irradiated samples recorded with 2.022 MeV He ions. The random and aligned RBS/C spectrum of the pristine sample is for a comparison. Two plateaus (below energy 1600 and 800) corresponding to the backscattering of He ions from Zr and O atoms of the target for cubic ZrO_2, respectively, are exhibited on the spectrum recorded in a random direction. The O signal increases with the energy

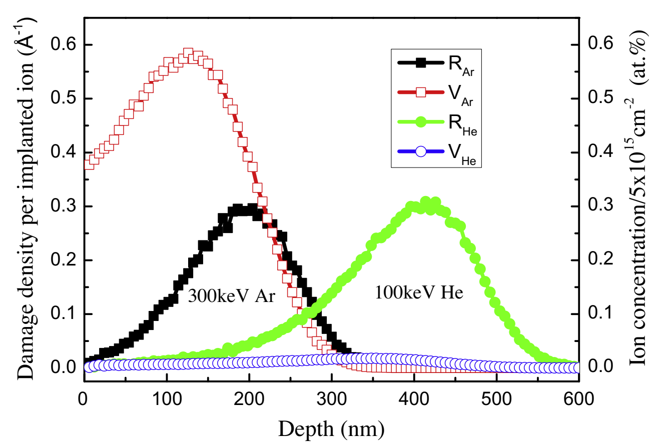

Fig. 2. Damage and range distribution of YSZ irradiated by 300 keV Ar and 100 keV He ions calculated by the SRIM 2013 code. The left and right Y-axes represent the vacancy caused by per injected ions at per unit depth (Angstrom) and the distribution of implanted ions, respectively.

increasing of injected helium. In our RBS/C measurements the injected helium energy is 2.022 MeV, which corresponds to a very weak O signal. The dechanneling yield χ_{min} = 7% is measured at the surface peak for the pristine crystal, indicating good crystallinity. Creation of damage by He or Ar ions irradiation in the Zr sublattice is attested by the increase of the backscattering yield (bumps) observed in the aligned spectra around energy 1450 and 1600, respectively. Several remarkable features should be noted: (i) the amplitude and width of the damage peaks increase with the ion fluence in both single He and single Ar ion beam irradiation. (ii) For dual ion beam irradiation, there is a superposition of two damage zone. Assuming that the observed RBS/C yield is proportional to the number of displaced atoms, the depth distribution of the accumulated damage f_D in both virgin and implanted YSZ (Zr sublattice) crystals is extracted from a computer program DICADA [17] (dechanneling in crystals and defect analysis). Variation of accumulated damage (f_D) as showed in Fig. 4 exhibits the following features: (i) Accumulated damage (f_D) grows symmetrically for single He ion irradiated condition and more pronounced for single Ar ion irradiation; (ii) two plateau presents in dual ion beam irradiation; (iii) the accumulated damage (f_D^{max}) value < 1 in all irradiated