



# Irradiation effects of displacement damage and gas atoms in Yttria-stabilized zirconia irradiated by Au and helium ions



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## ABSTRACT

Single and sequential ion beam irradiated Yttria-stabilized zirconia (YSZ) was carried out to study the irradiation effects of vacancies and helium gas atoms. The results show that the displacement damage value of sequential ion beam irradiation is less than that of single He ion irradiation and larger than that of single Au ion irradiation. The irradiation effects of displacement damage (mainly vacancies) and gas atoms may lead to a strong reduction of the interstitial helium atoms. Sequential ion beam irradiation generates more vacancies-helium bubbles than single helium ion irradiation. The results are important for fundamental understanding of interaction between vacancy and helium bubbles, and it also plays a guiding role in the practical industrial applications in the nuclear reactor.

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## 1. Introduction

YSZ is a representative model system for the investigation of irradiation effects due to the representative damage behaviors by ion irradiation. Heavy ions [1–3] and Helium ions irradiation [4–6] are commonly used to simulate the irradiation behavior. YSZ was also identified as potential candidate for nuclear waste immobilization and transmutation in nuclear reactors [7–9], and can be applied for the intermatrix layer of nuclear fuel [10,11].

As an important nuclear reactors material, YSZ will be exposed to severe irradiation environment of atomic displacement damage and accumulation of a large number of helium gas atoms [12]. It is necessary to examine the irradiation effects of displacement damage and helium bubble, which constitutes the aim of the letter. Irradiation effects were characterized by comparing sequential ion beam irradiation results with single beam irradiation.

## 2. Experiment

In order to stabilize the cubic phase, the ZrO<sub>2</sub> (1 0 0) single crystals used in this investigation were doped with 6.5 mol% Yttria. YSZ specimens were irradiated singly or sequentially by 4 MeV Au ions with a fluence of  $1.0 \times 10^{16}$  ions/cm<sup>2</sup> and 100 keV He ions with a fluence of  $2.0 \times 10^{17}$  ions/cm<sup>2</sup> at room temperature. The beam cur-

rent density of Au and He ions were  $0.02 \mu\text{A cm}^{-2}$  and  $0.1 \mu\text{A cm}^{-2}$ , respectively. The range peak (about 400 nm) of the Helium ions in YSZ might coincide with the displacement damage peak of Au ions calculated by the SRIM 2013 [13], considering displacement energy of 40 eV for both Zr and O atoms [14] as shown in Fig. 1(a). The left Y-axes represents the variations of the nuclear (Sn) energy losses as a function of depth in YSZ. The right Y-axes represents ions distribution. Four kinds of irradiation were used: single He, single Au, Au + He(sequencely), He + Au(sequencely). Fig. 1(b) shows the Sn and Se value of 4MeVAu and 100KeV He ions.

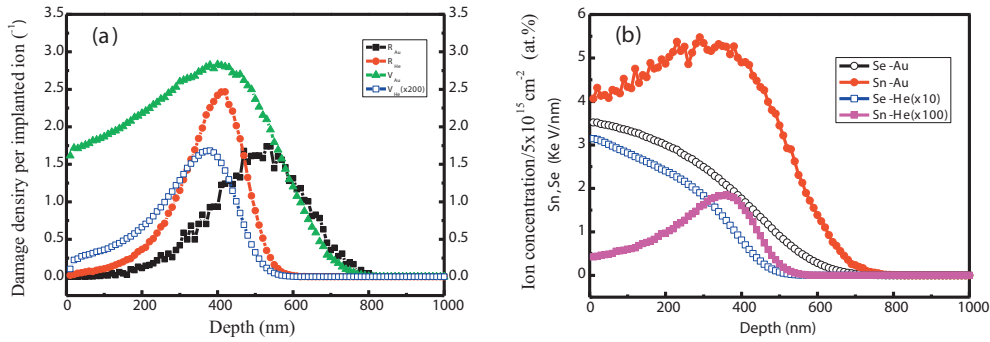
## 3. Results

### 3.1. RBS data

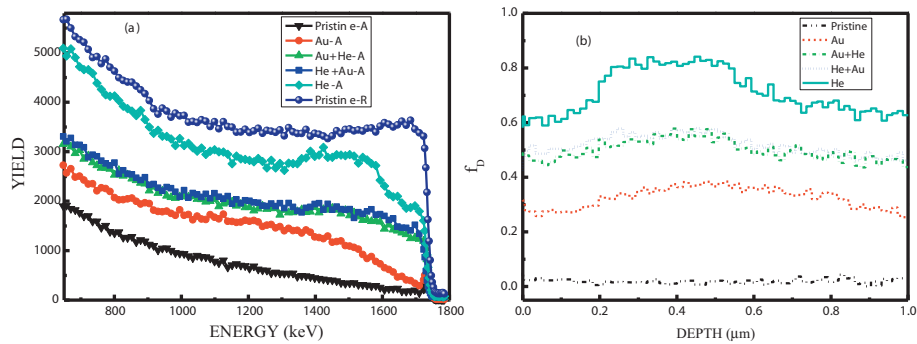
The ion induced damage was determined by Rutherford backscattering spectrometry/ channeling (RBS/C) measurements with a collimate 2.022 MeV He ion beam. The energy resolution was around 15 keV. Fig. 2(a) shows RBS/C spectrum of pristine and irradiated samples. Two plateaus (below energy 1750 and 930) are exhibited on the spectrum recorded in a random direction, corresponding to the backscattering of He ions from Zr and O atoms in the cubic ZrO<sub>2</sub> target. The dechanneling yield  $\chi_{\text{min}} = 5\%$  is measured at the surface peak for the pristine crystal, indicating good crystallinity. Creation of damage by He or Au ions irradiation in the Zr sublattice is attested by the increase of the backscattering yield (bumps) observed in the aligned spectra around energy 1500.

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**Fig. 1.** (a) is damage and range distribution of YSZ irradiated by 4 MeV Au 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. (b) is Sn and Se values of 4 MeV Au and 100 keV He ions calculated by the SRIM 2013 code.



**Fig. 2.** (a) RBS spectra for the pristine, Au, He, Au + He and He + Au irradiated specimens. (b) The corresponding damage depth profiles in the Zr sublattice of YSZ extracted from the analysis of RBS spectra of Fig. 2(a).

Several remarkable features should be noted: (i) the amplitude and width value of the damage peaks has a similar trend for Au + He and He + Au irradiation. (ii) The yield value of sequential ion beam irradiation is in the middle of two single ion beam irradiation. (iii) Single helium irradiation has the largest yield value. 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 dechanneling in crystals and defect analysis (DICADA) [15]. Variation of accumulated damage ( $f_D$ ) as showed in Fig. 2(b) exhibits the following features: (i) The maximum accumulated damage ( $f_D^{\max}$ ) were 0.84, 0.38, 0.55, 0.56 for single He, single Au, Au + He and He + Au ions irradiation. (ii) The  $f_D$  value of sequential ion beam irradiation is less than that of single He ion irradiation and larger than that of single Au ion irradiation. (iii)  $f_D$  value of Au + He is very similar to that of He + Au in all the irradiation depth.

The structural damage buildup in YSZ was analyzed by a multi-step damage accumulation (MSDA) model [16,17].  $2.0 \times 10^{17}$  ions/cm<sup>2</sup> helium irradiation belongs to the third step. The damage consists of small interstitial-type defects and helium-vacancy clusters, which will increase the damage level [18]. The level of damage measured by RBS/C depends on both the dpa value and the interaction of implanted helium atoms and vacancies. After thermal annealing, dpa is decreased due to complex of interstitials and vacancies, but the damage measured by RBS/C is more serious because of the formation of helium bubbles and elongated fractures [19]. More helium interstitials show little tendency to cluster but can also induce swelling [20]. Lattice distortions caused by interstitial Helium atoms can also increase the values measured by RBS/C method. For sequential ion beam irradiation helium is released from vacancy defects and is out-diffused at room

temperature [21], so the damage of Au + He and He + Au measured by RBS/C is smaller than single He ion irradiation.

### 3.2. SEM and AFM data

The area of surface helium bubble was measured by scanning electron microscope (SEM). Fig. 3(a–c) shows surface helium bubble distribution of YSZ irradiated by single He, He + Au and Au + He ions. Spherical Helium bubbles are formed at the fluence of  $2 \times 10^{17}$  cm<sup>2</sup>. The volume and density of helium bubbles generated by He + Au ions irradiation is larger than that of single He ions irradiation. Larger helium bubbles and severe exfoliation appeared at the surface of Au + He irradiated YSZ. Quantitative statistics of the number of Helium bubbles with different size are shown in Fig. 3(d). A maximum value of the average bubble radius is 2.8 μm for single helium ions irradiation. The trend of He + Au is similar to that of single He ions irradiation, but the number of bubbles was significantly increased. Au + He ions irradiation has a minimum number of helium bubbles but a maximum average radius of 3.8 μm. The percentage of helium bubble area is 11.6%, 14.8% and 9.3% for single He, He + Au and Au + He ions irradiation. Atomic force microscopy (AFM) was employed in order to investigate surface morphology changes caused by ion irradiation. Fig. 4(a–c) shows the morphologies image of AFM. The image was taken over an area of  $100 \times 100$  square microns. Fig. 4(d–f) is step height along the horizontal lines in Fig. 4(a–c). The maximum height of bubbles is about 100 nm and 500 nm for single He and He + Au ions irradiation, respectively. Maximum bubble height is about 1000 nm and a depth of peeling off is about 400 nm for Au + He ions irradiation. The height of a bubble is surface Helium bubble longitudinal length which is perpendicular to the ZrO<sub>2</sub> irradiation plane. The diameter of a bubble is surface Helium bubble lateral

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