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Characterization of the irradiation-induced phase transition in the monoclinic polymorph of zirconia

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ABSTRACT

Two groups of pure zirconia (ZrO_2) specimens with the monoclinic polymorph and distinct nanopowder size ranges of approximately 20–30 nm and 30–60 nm were irradiated separately at room temperature with 100 keV H^+ and 3 MeV Fe^{2+} ions to various fluences. Following TEM observations and XRD analyses, the irradiation with 100 keV protons to a fluence of over 3×10^{15} ions/cm^2 was demonstrated to induce transformation from monoclinic (m) to tetragonal (t) phases in the specimen with the size range of approximately 20–30 nm due to the energy crossover that facilitates the $m \rightarrow t$ transformation. However, the transformation did not occur in the irradiated specimen with the other size range even though the fluence of protons was up to 1×10^{17} ions/cm^2. Furthermore, although irradiation with 3 MeV Fe^{2+} ions brought about low electronic energy loss, the $m \rightarrow t$ transformation can be induced under the low fluence of 1×10^{14} ions/cm^2 because a high level of damage defects (oxygen vacancies) may promote the $m \rightarrow t$ transformation. Consequently, the largest crystallite size of t-ZrO_2 induced by the proton irradiation is approximately 29 nm, as a result of the metastable critical size effect of t-phase zirconia at room temperature.

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1. Introduction

Zirconia (nominally with stoichiometry ZrO_2) has become one of the most important ceramic materials over the past several decades because of its superior physical and electrical properties, high ionic conductivity, excellent chemical durability, and low thermal conductivity in a wide range of industrial applications (e.g., as a catalyst, gas sensor, and electrolyte in solid oxide fuel cells, and as a gate dielectric in microelectronics) [1–5]. In addition, the discovery of transformation toughening published in the journal *Nature* by Garvie et al. [6] proclaimed new high-performance applications of zirconia, ranging from bearing and wear applications to thermal barrier coatings on metal components [7,8]. Moreover, zirconia polymorphs demonstrate excellent radiation tolerance for nuclear applications [9], such that they have been suggested as a promising parent material for use as a fuel matrix in nuclear reactors [10]. However, zirconia exhibits three crystallographic polymorphs as a function of temperature at normal atmospheric pressure [11]: the monoclinic baddeleyite (m-ZrO_2) is thermodynamically stable from room temperature to 1170 °C, the tetragonal phase (t-ZrO_2) is thermodynamically stable from 1170 to 2370 °C, and the fluorite-type cubic phase (c-ZrO_2) is thermodynamically stable from 2370 to 2680 °C (melting point). These polymorphs significantly affect the physical and mechanical properties of zirconia used as fuel or a parent material for nuclear applications.

Zirconia phase transition under irradiation has been widely studied under different temperature ranges and the mass and energy of bombarding ions, and these studies have shown that polymorphism plays an important role in the radiation damage evolution of zirconia. In a series of experiments, Benyagoub et al. [12,13] found that irradiation with 135 MeV ^{58}Ni ions, 300 MeV ^{76}Ge ions, and 250 MeV ^{127}I ions generates a transformation from the monoclinic to the tetragonal phase (hereafter denoted as an $m \rightarrow t$ transformation), where primarily electronic stopping exceeding 13 keV/nm dominates over nuclear stopping. Based on transmission electron microscopy (TEM) and Raman spectroscopy examinations, Sickafus et al. [14,15] demonstrated that m-ZrO_2

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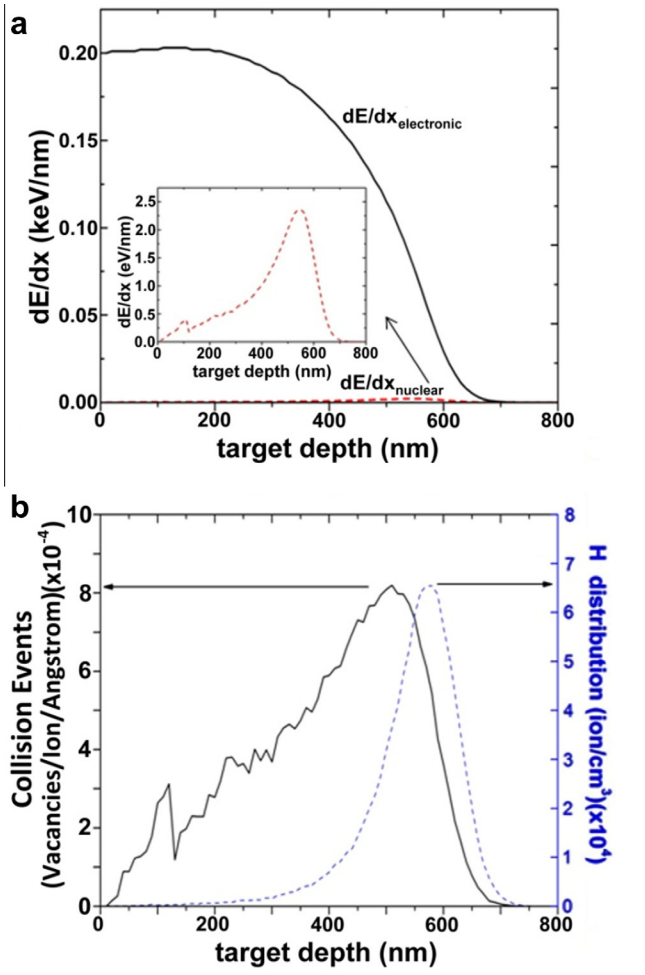

Fig. 1. SRIM simulation results for 100 keV H^+ ion irradiation of ZrO_2. (a) Energy loss as a function of depth for the nuclear and electronic stopping components of the stopping power and (b) collision events (left-hand ordinate) and implanted H^+ ion concentration (right-hand ordinate) as a function of depth for the irradiation.

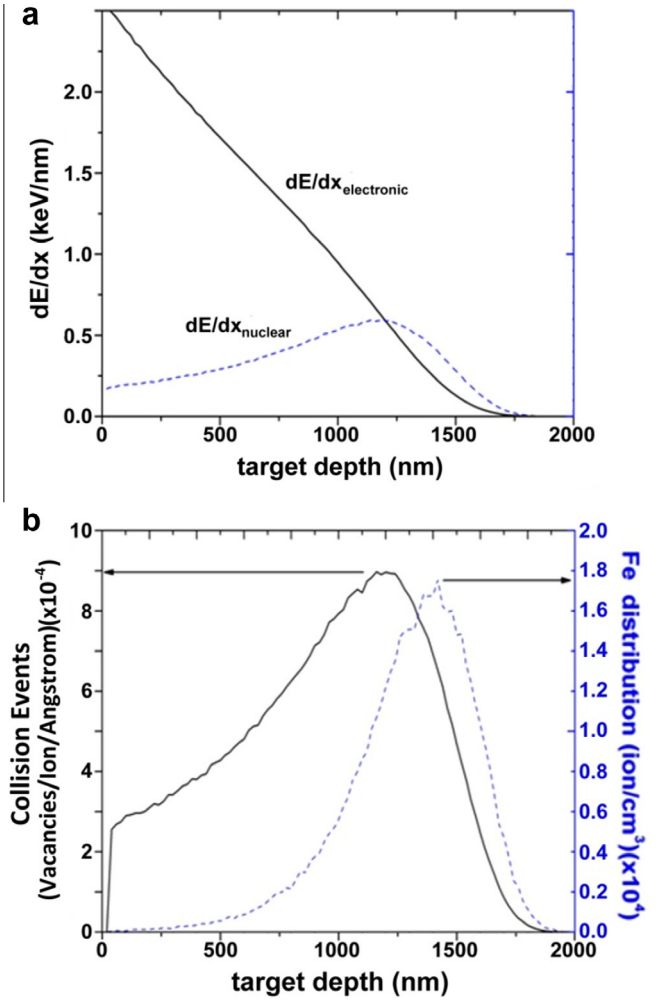

Fig. 2. SRIM simulation results for 3 MeV Fe^{2+} ions irradiation of ZrO_2. (a) Energy loss as a function of depth for the nuclear and electronic stopping components of the stopping power and (b) collision events (left-hand ordinate) and implanted Fe^{2+} ions concentration (right-hand ordinate) as a function of depth for the irradiation.

exposed to 340 keV Xe^{2+} ions at 120 K and to 300 keV Kr^{2+} ions at approximately 80 K (in the nuclear stopping energy regime), respectively, transformed to t-ZrO_2. Valdez et al. [15] showed that 150 keV Ne^+ ions irradiation of m-ZrO_2 also produces $m \rightarrow t$ transformation (in both electronic and nuclear stopping regime). Simeone et al. [16] observed a similar $m \rightarrow t$ transformation using 800 keV Bi ions and suggested, based on grazing X-ray diffraction analysis, that the tetragonal phase is localized in the damaged area.

Nanocrystalline zirconia is known to have unique mechanical, thermal, and electrical properties compared to its bulk counterparts, and the phase stability of nanosized zirconia can be affected significantly by its grain size [17]. In the considerable body of published literature on the effects of irradiation on bulk zirconia, few systematic studies of the behavior of nanocrystalline zirconia under similar conditions exist. We are aware of one report, in which an $m \rightarrow t$ transformation was observed in the monoclinic-dominant ZrO_2 with an average grain size of 40–50 nm upon the ion irradiation of 350 keV O^+ and 1 MeV Kr^{2+} at room temperature [18]. Therefore, in this study, we focus on the evolution of pure m-ZrO_2 nano particles under 100 keV protons and 3 MeV Fe^{2+} ions irradiation at room temperature and document an unusual behavior of ZrO_2 nanoparticles under size effects in the irradiation-induced $m \rightarrow t$ transformation under 100 keV protons irradiation.

2. Material and methods

Two distinct particle-size ranges of m-ZrO_2 powders, approximately 20–30 nm and 30–60 nm (verified by TEM observation), purchased from CERAC Corp. (99.995% pure) were used to prepare the two sample groups to which ion bombardment is executed. First, a small amount of powder with the same size range was added to DI-water, followed by ultrasonication to disperse the particles; then a small drop of suspension was spread repeatedly on a clean silicon chip. The evenly spread specimen was finally dried using an electric hot plate at 60 °C. The thickness of the deposited powder was controlled over 15 μm. To depict the specimen with two distinct particle size ranges of m-ZrO_2 powders, the prepared specimens with the smaller particle sizes of approximately 20–30 nm and with the larger particle sizes of approximately 30–60 nm were designated as S30 and L30 specimens, respectively. These zirconia specimens were sequentially irradiated with 100 keV H^+ ions in the Nuclear Science & Technology Development Center at National Tsing Hua University using a 500 kV High Voltage Engineering Europa ion implanter and with 3 MeV Fe^{2+} in the Institute of Physics at the Academia Sinica using National Electrostatics Corporation 9SDH-II 3MV Tandem Accelerator. The implantation energy of 100 keV protons, based on the capability of the instrument, was chosen to achieve a relatively large electron

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