Dislocation loops in Yttria Stabilized Zirconia induced by electron irradiation: Role of electron energy and irradiation temperatures

KYUSHU UNIVERSITY

AKM Saiful Islam Bhuian[1,2], T. Yamamoto[1], K. Yasuda[1], S. Matsumura[1], H. Yasuda[3] and N. Ishikawa[4]

[1] Applied Quantum Physics & Nuclear Engineering, Kyushu University, Fukuoka, Japan
[2] Atomic Energy Centre, Bangladesh Atomic Energy Commission, Chittagong, Bangladesh
[3] Research Center for Ultra High Voltage Electron Microscopy, Osaka University, Japan
[4] Nuclear Science and Engineering Center, Japan Atomic Energy Agency (JAEA), Japan

Abstract

Yttria stabilized cubic zirconia (YSZ) is considered to be one of the potential matrices to be used in hostile radiation environment. In the present study, we have investigated the evolution of dislocation loops in YSZ irradiated with a wide range of temperatures (from 300 to 773 K) and electron energies (from 1.25 to 3.0 MeV). *In situ* observation revealed that at 300 and 673 K with 1.25 MeV electron irradiation, no dislocation loops were formed up to 3600 sec of electron irradiation with a flux of 2.4×10^{23} $m^{-2}s^{-1}$. At high temperatures beyond 773 K with 3 MeV electron irradiation, perfect dislocation loops started to nucleate from the beginning of irradiation and the size of loops gradually grew up with increasing electron fluence. On the other hand, electron irradiation at medium energy and temperature (2 MeV and 300 K), defects with different type and contrast were formed: tiny perfect type dislocation loops were nucleated covering the whole irradiated beam with a low growth speed. A very large loop with strong strain contrast, which is considered to be consist of solely O ions, were formed in this irradiation condition. The microstructure evolution of dislocation loops dependent on temperature and electron energy is discussed based on the difference in production rate of O and Zr point defects and their migration energies. Role of electron energy and irradiation temperature in accordance with prevalent ion-tracks on YSZ induced by swift heavy Xe^{14+} ions were also discussed.

Introduction

❑Yttria stabilized cubic zirconia (YSZ)

→ Exceptional radiation tolerant quality

→ potential applications for advanced fuel and waste form

❑Recent radiation damage studies with YSZ…

→ Ion irradiation covering energy range of a few MeV – several GeV.

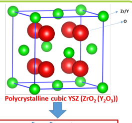
→ Point defect creations, damage build up, damage recovery…

❑Our collaborators investigated…

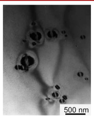
→ Microstructure evolution in YSZ through TEM under irradiation with electron and/or ions.

→ Anomalous formation of large defect clusters consists of O ions.

⇨ Suggesting an important role of selective displacement damage on O-ions, which depends on Irradiation temperature and e⁻-energy



$$Y_2O_3 + 2Zr_{Zr}^{X} + O_O^{X} \rightarrow 2Y'_{Zr} + V_O^{\cdot\cdot} + 2ZrO_2$$



(K. Yasuda et. al., JNM 319, 2003)

Objective: To understand the nucleation-and-growth of radiation-induced defects in YSZ

Experimental

❑Specimens

→ Sintered **Yttria stabilized zirconia** (containing 8 mol% Y_2O_3)

→ TEM specimen by Ar-ion milling

❑ TEM Irradiation Conditions

High Voltage (MeV)	1.25	1.5	2.0	2.5	3.0
Electron Flux (e^-/m^2s) (Dose Rate (×10^{-3}dpa/s))	1.3×10^{24} (O: 1.7, Zr: 0.7)		-		2.0×10^{24} (O: 1.5, Zr: 1.3)
Temperature (K)	300		-		773
Irradiation Time (sec)	~ 1200				

JEM-1300NEF TEM (JEOL Ltd.) Ultra-microscopy Research Center, Kyushu University

Ultra-High Voltage Electron Microscope (Hitachi H-3000) Osaka University

***In situ* TEM Observation**

❑Ion Irradiation of YSZ

200 MeV Xe^{14+} irradiation

Ion Accelerator Facility, JAEA

Dose Level: 5×10^{11} cm^{-2}

Irradiation Temperature: 300 K

***In Situ* Observation**

TEM Facility : Hitachi H-3000

High Voltage: 3.0 MeV

e^- Fluence : 1.8×10^{26} m^{-2}

Temp.: 300, 573 and 773 K

Tandem Accelerator Section
Department of Research Reactor and Tandem Accelerator
Japan Atomic Energy Agency (JAEA)

Tandem Accelerator

Results and Discussion

1. Mislaying of defects at 1.25 MeV e⁻ irradiation:

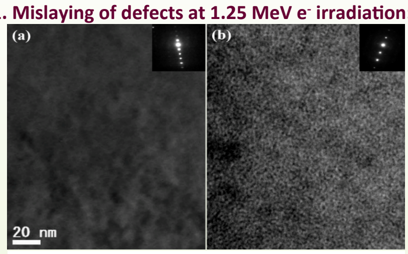


Fig. 1: BF-TEM images showing no defect clusters up to 4.5×10^{26} m^{-2} of electron fluence at 300 K (a) and 673 K (b) under 1.25 MeV e⁻ irradiation. Inset in (a) and (b) show the selected area diffraction pattern of the irradiation condition.

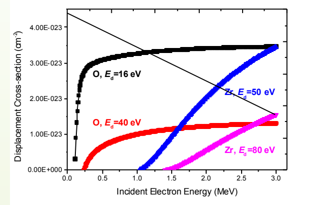


Fig. 2: Displacement cross-section vs e⁻ energy with threshold displacement energies of 16-40 eV for O sublattice and 50-80 eV for Zr sublattice.

– Reported values of $\boldsymbol{E}_d$ of O and Zr in cubic zirconia are limited (shown with a certain range in Fig.2).

– O-interstitials are diminished by the recombination with empty interstitial sites in O-sublattice, which were induced to compensate the charge neutrality by doping of Y^{3+} ions.

2. Perfect loops followed by O-interstitial type at 2.0 MeV e⁻ irradiation:

– Combination of dot contrasts with a size of few nm and tiny perfect loops were seen covering the whole irradiation area.

– Large O-interstitial type loop with strong strain contrast, as observed in 1.5 MeV irradiation, was seen at the periphery of the beam.

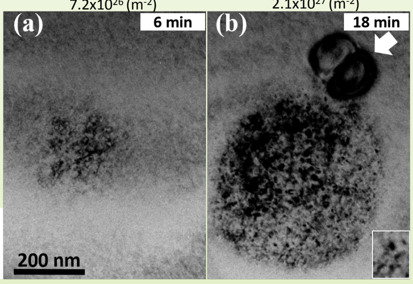


Fig. 3: BF-TEM sequential images showing defect nucleation and growth with 2.0 MeV e⁻ irradiation at 300 K. The observations were performed from [012] direction with **g** = 200 reflection. Inserted micrograph in (b) is a magnified image of the defects formed in (b). The white arrow in (b) points to an oxygen displacement type dislocation loop.

3. Defects in YSZ as a function of e⁻ energy and irradiation temperature:

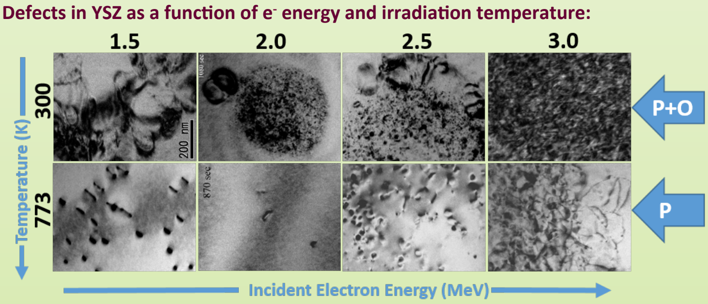
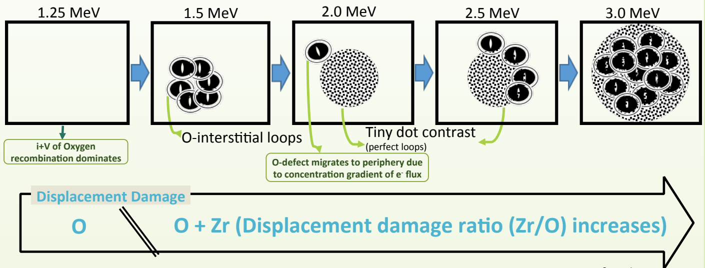


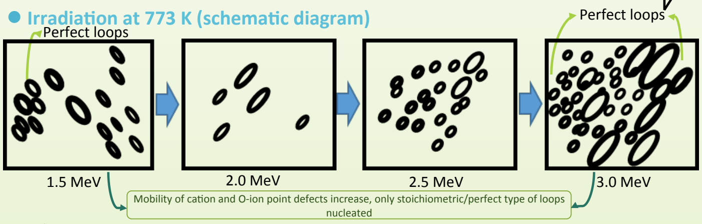
Fig. 4: BF-TEM images showing defects in YSZ with different e⁻ energy and irradiation temperatures. Here P and O in the blue arrow are used to indicate the perfect loops and O-interstitial loops respectively.

4. Defects in YSZ as a function of e⁻ energy and irradiation temperature:

● Irradiation at 300 K (schematic diagram)



● Irradiation at 773 K (schematic diagram)



– Defects on YSZ can be produced at and beyond 1.5 MeV e⁻ irradiation. Threshold displacement energy for Zr can be bracketed from this assumption as:

$$E_d = \frac{2E_e(E_e+2m_ec^2)}{mc^2} = \frac{2147.7E_e(E_e+1.022)}{A}$$

(Incident e⁻ energy: E_e; Atomic mass: A)

For E_e= 1.25 MeV For E_e= 1.5 MeV

Thus, 66 < $E_{d,Zr}$ ≤89 in eV

5. Defects in YSZ with e⁻ subsequent to 200 MeV Xe^{+14} ion irradiation:

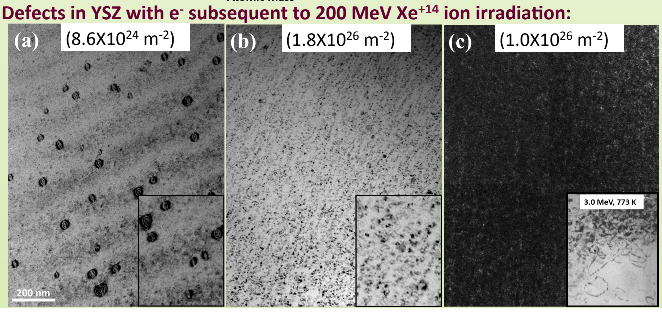


Fig. 5: BF-TEM images showing defects in Xe^{14+} ion irradiated YSZ subsequently irradiated with 3 MeV e⁻ energy at 300 K (a), 573 K (b) and 773 K (c). Inserted micrographs in (a) and (b) show the defects in respective irradiation conditions, while inserted image in (c) shows defects in e⁻ irradiated YSZ.

– Loops formed in ion-tracks induced by Xe ion irradiation prior to electron irradiation.

Conclusions

A series of *in situ* irradiation experiments were performed on YSZ at different electron energy ranging from 1.25 to 3.0 MeV and irradiation temperature, from 300 to 773 K, and the following conclusions are drawn:

- e⁻ energy of 1.25 MeV or lower is not enough to form defect clusters in YSZ.
- Two types of dislocation loops: perfect dislocation loops and/or O-interstitial type ones, were formed at 1.5, 2.0, 2.5 and 3.0 MeV e⁻ irradiation.
- The ratio of the displacement rate of O and cation sublattices induced by incident electron energies, controls the evolution of dislocation loops.
- Threshold displacement energies for Zr sublattices is bracketed as 66< $E_{d,Zr}$≤89 eV.
- Ion-tracks induced by heavy ion irradiation work as nucleation sites for dislocation loops.

Presented @ Joint ICTP/IAEA Workshop on Fundamentals of Vitrification and Vitreous Materials for Nuclear Waste Immobilization
6 - 10 November 2017
Trieste, Italy