超高圧電子顕微鏡法による立方晶安定化ジルコニアの微細構造発達に関する研究

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Yttria-stabilized cubic zirconia (YSZ) with a fluorite-type structure has attracted enormous attention due to its exceptional radiation tolerant quality against extreme radiation environment, and it is, therefore, considered to be matrices of nuclear reactor fuel and transmutation target for actinides. In these applications, YSZ should subsist against severe adverse environment regarding high temperature and intense radiation fluxes of different kinds. Despite excellent radiation tolerance, fundamental understanding of radiation damage and defect kinetics is desired for the further improvement and development of radiation tolerant material.

The production rate of point defects in YSZ under irradiation differs in cations and oxygens. Reported defect formation and growth in YSZ is, therefore, dependent on irradiation conditions. The present study investigated the nucleation-and-growth of extended defects in YSZ through in situ high voltage transmission electron microscopy under irradiation. Microstructure evolution in pristine and ion-irradiated YSZ was investigated as functions of electron energy and irradiation temperature to gain insights into the kinetic behavior of point defects, under which production rate and mobility of point defects of oxygens and cations change. This dissertation consists of six chapters.

Chapter 1 describes the research goal of this study and step-wise arrangement of the chapters is also outlined.

Chapter 2 reviews theoretical and experimental investigations on radiation damage in ceramics, with an emphasis on fluorite structure oxides. Distinct responses of defect formation under different irradiation particle and temperature are discussed. Elastic displacement cross-section and threshold displacement energies of anion and cation sublattices are also compiled.

In Chapter 3, the basics of transmission electron microscopy (TEM) including imaging and diffraction techniques are firstly described. Experimental details for specimen preparation technique of 8 mol% Y$_2$O$_3$ doped zirconia (YSZ) specimen, and ion or electron irradiation conditions are described in detail. In situ TEM conditions are also described together with the explanation of data acquisition procedures.

Chapter 4 describes in situ TEM results on the nucleation-and-growth of defects in 8
mol% pristine YSZ as functions of electron energy and irradiation temperatures. *In situ* TEM experiments showed that electron irradiation with 1.25 MeV or lower does not induce defect clusters in YSZ. On the other hand, electron irradiation ranging from 1.5 to 3.0 MeV forms oxygen interstitial-type and/or perfect type dislocation loops. The microstructure was developed by the mutual interaction of those defects, and the ratio of displacement damage in oxygen and cation sublattices has found to be a principal factor. Displacement cross-sections of oxygen and cation sublattices are evaluated by simulations. Threshold displacement energy is also determined experimentally from the energy-dependent microstructure evolution to be around 80 eV for cations.

Chapter 5 presents results on the interaction of Frenkel defects induced by 3.0 MeV electrons with the columnar defects of ion tracks induced by 200 MeV Xe ions. Microstructure evolution was significantly different from pristine YSZ specimens. Ion-tracks are found to act preferential nucleation sites of dislocation loops, and change the nature of dislocation loops and threshold temperature for perfect dislocation formation in ion-irradiated YSZ.

In chapter 6, all the experimental and simulation works done in the wake of this study are summarized as conclusions. Possible directions of research for further understanding of defects’ behavior in YSZ are also highlighted.