Electron channeling x-ray spectroscopy for quantitative determination of atom configuration in partially disordered compounds

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Magnesium aluminate spinel (MgO·*n*Al2O3) has potential applications to devices in fusion reactors, because of its excellent radiation resistance. In the course of a study to promote atomistic understanding of radiation induced structural change in MgO·*n*Al2O3, *high angular resolution electron channeling x-ray spectroscopy* (HARECXS) was utilized to determine ion configuration in the irradiated materials. HARECXS takes advantage of characteristic x-ray emission from an illuminated local area of interest as a function of incident electron beam direction over an extended range of angle including several Bragg conditions. The present paper will report HARECXS measurements of ion configuration in MgO·*n*Al2O3 annealed and irradiated with electrons or ions, and emphasize the applicability of HARECXS to heterogeneously disordered materials.

Thinned disc specimens of MgO·*n*Al2O3 with n=1.0 and 2.4 were annealed at 1470 K for 2 days, and then irradiated at 870 K with 900 keV electrons up to fluence of 1.6 x 1027 e/m2. On the other hand, single-crystalline MgO·*n*Al2O3 bulks were subjected to irradiation at 870 K with 1 MeV Ne+ ions up to fluence of 4.5 x 1020 Ne+/m2. The bulk specimens were then mechanically thinned to a wedge shape for cross sectional observation. HARECXS profiles of x-ray intensity were obtained with incident beam rocking between 4*g* and +4*g* (*g*=400) Bragg conditions. The results were analyzed on the dynamical scattering formulation derived by Rossouw *et al.* to locate crystallographic positions of the component ions quantitatively.

The theoretical simulations demonstrated that the HARECXS profiles are quite sensitive to ion replacement in the crystal lattice. Accordingly, the experimental HARECXS clearly distinguishes the stoichiometric n=1 and the nonstoichimetric n=2.4 compounds in terms of cation configuration. The former almost forms the normal spinel configuration, where Mg^{2+} and Al^{3+} ions occupy the tetrahedral (IV) and the octahedral (VI) sites respectively in the approximate *fcc* lattice of O^{2-} ions. In the latter compound, in contrast, the cation configuration is partially disordered. Mg^{2+} ions are located more in the VI sites than in the IV positions. 900 keV-electron irradiation induces simple cation disordering between the IV and the VI sites. The radiation-induced disordering is less significant in $MgO\cdot2.4Al_2O_3$ than in $MgO\cdot Al_2O_3$, since structural vacancies involved in the former enhance reordering. On the other hand, 1 MeV Ne^+ ion irradiation causes ion replacement depending on depth from the irradiated surface. The simple disordering occurs in defect free regions near the irradiated surface, in a similar way to the case of electron irradiation. The compound of n=2.4 is more stable than n=1 in this range of depth. In contrast, fully disordering proceeds in the peak-damaged regions in both the compounds without any significant difference. Thus HARECXS is applicable even to heavily damaged areas containing extended lattice defects.