The response of complex ceramic oxides exposed to ion-irradiation, compared using two TEM characterisation techniques; bulk, *ex-situ*, and thin crystal, *in-situ*.

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The response of materials exposed to high energy particles has been an active area of research for several decades, due both to potentially improved and detrimental effects on the material properties. For the nuclear industry ion-irradiation has been used to simulate recoil damage from alpha decay, and exposure to neutrons in fission and fusion reactors [1]. Whilst there are many changes to the host material caused via the impact of accelerated ions the particular focus of this research is on the crystalline to amorphous transition. The amorphisation of the host material can lead to detrimental effects on its properties such as swelling, embrittlement, and micro-cracking leading to eventual structural failure.

The complex ceramic oxides chosen for this study, Ln_2TiO_5 (Ln = lanthanides and yttrium), have several uses within the nuclear industry. The compound Dy_2TiO_5 has been used within Russian WWER type reactors for two decades due to its good resistance to irradiation induced swelling and structural failure [2]. Of particular interest are the cubic symmetry compounds with defect fluorite structure, which gives good radiation response. Previous studies have indicated that the series of Ln_2TiO_5 compounds may take on a variety of crystal symmetries depending on the lanthanide size and fabrication conditions used [3, 4].

Previous to any ion-irradiation exposure the materials of interest were tested for homogeneity of stoichiometry and crystallography. Characterisation was carried out via backscattered electron imaging (Z contrast) to highlight any variations in elemental composition. This was followed up with multiple spot analyses using energy dispersive x-ray spectroscopy to confirm the homogeneous nature of the material plus the stoichiometry. X-ray diffraction was used to determine the long range symmetry of the test materials plus confirm the single structure nature. Any materials found to have more than one crystal structure type or greater than 5% secondary phase were rejected for further ion-irradiation based experiments.

The preliminary study of ion-irradiation response was carried out using the in-situ approach where the test materials were exposed to 1 MeV Kr^{2+} ions and monitored for their transition from crystalline to amorphous state. The *in-situ* ion-irradiation was carried out using the intermediate voltage electron microscope (IVEM)-Tandem facility at Argonne National Laboratory. The critical dose of irradiating ions, D_o, required to render the Ln_2TiO_5 completely amorphous was determined by monitoring selected area electron diffraction patterns for loss of diffraction spots (Bragg maxima) and replacement with diffuse rings (refer to Figure 1).

Further bulk Se^+ ion-irradiation was carried out at the Australian National University using the TANDEM, heavy ion accelerator. The damage penetration depth was characterised *ex-situ* using cross-sectional TEM. The cross-sectional damage depth profile of the bulk sample was compared with simulation, SRIM (Stopping Range of Ions in Matter), based calculations and a critical dose of amorphisation value attained.

By using these two TEM characterisation approaches the thin crystal *in-situ* results can be compared with the more "realistic" bulk approach.

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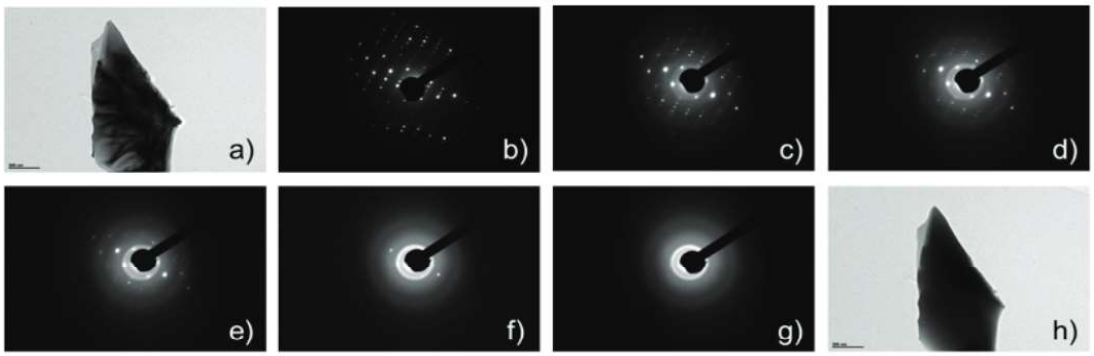

Figure 1. A series of bright field electron micrographs and selected area electron diffraction patterns showing the effects on crystal structure with exposure to 1 MeV Kr^{2+} ions; (a) the unirradiated grain, (b – g) from unirradiated diffraction pattern to sequentially increasing dose of ions until completely amorphous indicated by loss of diffraction maxima, (h) amorphous grain.

References

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