Characterization and In-Situ Ion-Irradiation of MA957 ODS Steel

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INTRODUCTION

Oxide dispersion strengthened (ODS) Ferritic/Martensitic steels produced by mechanical alloying with Y_2O_3 particles are considered as possible cladding materials for GEN-IV nuclear reactors. They are expected to achieve high creep strength and be resistant to radiation swelling at the high temperatures (350-700ºC) and doses (~200 dpa) expected in these reactors thanks to the incorporation of a very high density of fine-scale oxide particles in the matrix. Since these alloys derive their good properties from their special microstructure (especially the oxide dispersion), it is necessary to assess the stability under irradiation of the microstructure in general and the nano-sized particles in particular. The use of *in-situ* ion irradiation in a transmission electron microscope (TEM) provides a unique approach to follow the microstructure evolution of the material under irradiation.

EXPERIMENT

Material Characterization Prior to Irradiation

MA957 ODS alloy (Fe–14Cr–1Ti–0.3Mo–0.25Y_2O_3) obtained from CEA (Saclay, France) was characterized in the as-received condition using 15 keV synchrotron radiation x-ray diffraction (XRD) at Argonne National Laboratory (ANL). The XRD spectrum (Fig.1) did not show diffraction peaks associated with the Y_2O_3 particles initially introduced, but showed instead the presence of Y_2O, indicating that the original particles transformed during processing of the alloy. The TEM observations showed two populations of oxide particles (one of fine particles (few nms) and one of larger particles (few hundreds of nms)), a lamellar grain-structure in the direction of extrusion and an isotropic and small-size grain-structure in the transverse direction (Fig.1).

In Situ Ion Irradiations

TEM samples were irradiated in situ in the Intermediate Voltage Electron Microscope at ANL using 300 keV self-ion (Fe) irradiation at 25ºC (~21 dpa) and 1 MeV Kr ions at 500ºC (~200 dpa), under a vacuum of 10^{-7} Torr. The evolution was followed in situ by systematically recording bright-field, dark-field and diffraction-pattern micrographs at successive doses.

RESULTS

300 keV Self-Ion Irradiation at 25 ºC to 18 dpa:

Irradiation at room temperature showed high density of irradiation damage in the form of "black dot damage" and developing loops, as well as particle amorphization and degradation of particle interfaces as shown in Fig.2.

1 MeV Kr Irradiation at 500 ºC to 200 dpa:

Despite the very high damage level attained, the overall microstructure was remarkably stable. It was possible to discern the same grain structure present at the beginning of irradiation. Detailed examinations using through focus imaging showed no voids formed. The large particles appeared to be stable to amorphization at 500ºC, as expected (Fig.3); no halo was observed around the particles.

In both irradiations direct observation of the small precipitate population (<5 nm) after irradiation was not possible due to the large density of defect clusters. The use of energy filtering imaging technique will be useful to discern whether or not particle dissolution has occurred.

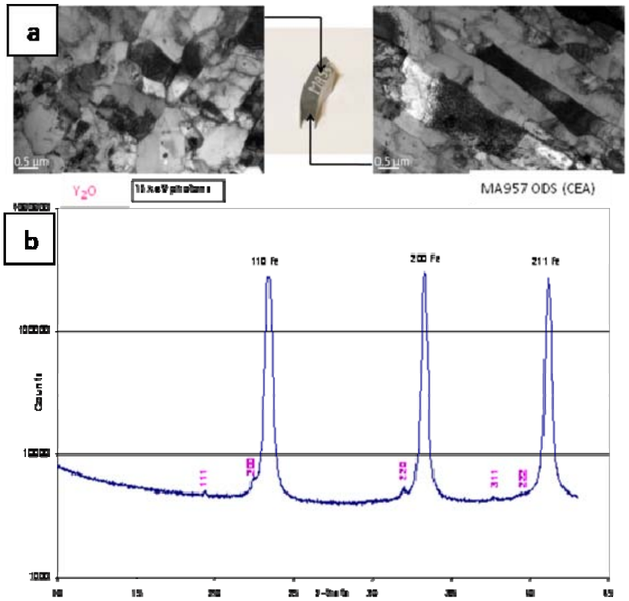

Fig. 1. (a) Bright Field TEM images of the grain microstructure in MA957 ODS steel in transversal direction (left) and (right) circumferential direction and (b) XRD spectrum from 15keV synchrotron irradiation showing Fe matrix peaks and oxide peaks.

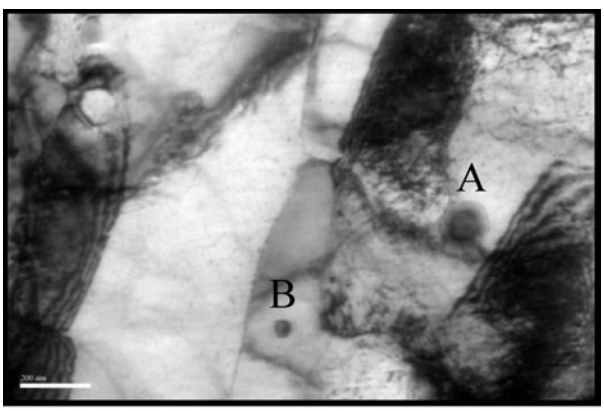

Fig. 2. Bright Field TEM micrograph of MA957 irradiated with 300 keV Fe ions at 25˚C to ~18 dpa: some oxide particles exhibit a halo, indicating dissolution at the surface (particle A), while other oxide particles are more stable (particle B). The difference in behavior most likely comes from the difference in local chemical composition.

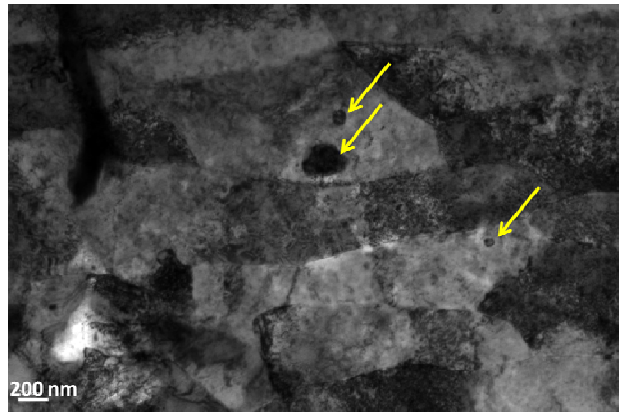

Fig. 3. Bright Field TEM micrograph of MA957 irradiated with 1 MeV Kr ions at 500˚C to ~200 dpa. Despite a large amount of damage the alloy retains its grain morphology after 200 dpa and the large precipitates remain crystalline.