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Irradiation creep and precipitation in a ferritic ODS steel under helium implantation

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Abstract

Ferritic oxide dispersion strengthened (ODS) steel, PM2000, has been homogeneously implanted with helium under uniaxial tensile stresses from 20 to 250 MPa to maximum doses of about 0.75 dpa (3000 ppm He) with displacement damage rates of 5.5×10^{-6} dpa/s at temperatures of 573, 673 and 773 K. Straining of a miniaturized dog-bone specimen under helium implantation was monitored by linear variable displacement transformer (LVDT) and meanwhile by their resistance also measured by four-pole technique. Creep compliance was almost constant at 5.7×10^{-6} dpa^{-1} MPa^{-1} for temperatures below 673 K and increased to 18×10^{-6} dpa^{-1} MPa^{-1} at 773 K. The resistivity of PM2000 samples decreased with dose and showed a tendency to saturation. Subsequent transmission electron microscopy observations indicated the formation of ordered $Fe_{3-x}Cr_xAl$ precipitates during implantation. Correlations between the microstructure and resistivity are discussed.

1. Introduction

Operation temperatures of future advanced gas cooled reactors within the framework of the International Generation-IV-Initiative Forum [1] will exceed 1173 K with the aim to even reach 1273 K and more. This calls for creep-resistant structural materials not only for piping and heat exchangers but also for in-core or close to core supports or fixtures. Materials with high creep resistance under irradiation at very high temperatures must especially be developed. Oxide dispersion strengthened (ODS) steels may be candidates, since the nano-size oxide dispersoids act as thermodynamically stable obstacles to dislocation movement up to temperature of $T/T_m = 0.9$ [2]. Furthermore, the thermal stresses caused by temperature gradients in components made from ferritic/martensitic ODS steels can be more than three times less compared to nickel-based alloys due to their relatively high thermal conductivity and low thermal expansion coefficient [3]. These advantages add to the overall better radiation resistance of the ferritic/martensitic steels and have stimulated investigations for nuclear fission and fusion application worldwide in the last decade [4–10]. Recently, some studies on phase stability and bubble/void evolution in these steels by means of a dual-ion irradiation have been reported [5, 11–13]. Only a few experiments investigated the mechanical properties of ODS steels after irradiation, while irradiation creep data of ODS steels are rather scarce and are still missing for PM2000. Irradiation creep under simultaneous high helium production is a topic which has not yet been addressed, but may be relevant in advanced nuclear power plants. Another important concern is the microstructural stability, which can be assessed in general by measurement of electrical resistivity and in detail by analytical transmission electron microscopy (TEM). Therefore, in the present work, in situ irradiation creep of the ODS alloy PM2000, and resistivity change were investigated during He-implantation, while microstructural changes were studied by TEM after implantation.

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2. Experimental

The ferritic ODS alloy PM2000 was supplied by Plansee GmbH in the form of 15 mm thick plates of nominal composition (wt%, balance Fe) 20% Cr, 0.5% Ti, 5.5% Al, and 0.5% Y_2O_3. The alloy was manufactured mechanically by alloying in a high energy mill, with the powder consolidated by hot compaction, followed by a hot and cold rolling procedure and a final thermal treatment [8,14] giving a quite uniform dispersion of yttria. Fig. 1 shows the metallographic cut along longitudinal direction (a), TEM micrographs of microstructure (b) and the Y_2O_3 particle size distribution from image analysis (c), of PM2000 in condition as-received. From those pictures, one can summarize that the rolling procedure produced grains with sizes of roughly $1 \times 1 \times (>12)\ \text{mm}^3$, elongated along the rolling direction. The average diameter and number density of the Y_2O_3 particles were (28 ± 8) nm and 5.1×10^{20} particles/m^3, respectively. Dog-bone shaped creep samples of 300 μm thickness were cut by spark erosion perpendicular to the rolling direction in an attempt to have at least a few grain boundaries within the gauge section. For future application of this material, thermo-mechanical treatment will need optimization. The samples were mechanical polished on both sides to 100 μm with grad 2400 paper. The final samples had an overall size of 28 mm in length, 8 mm in width and 0.1 mm in thickness, with a gauge volume of $10 \times 2 \times 0.1\ \text{mm}^3$.

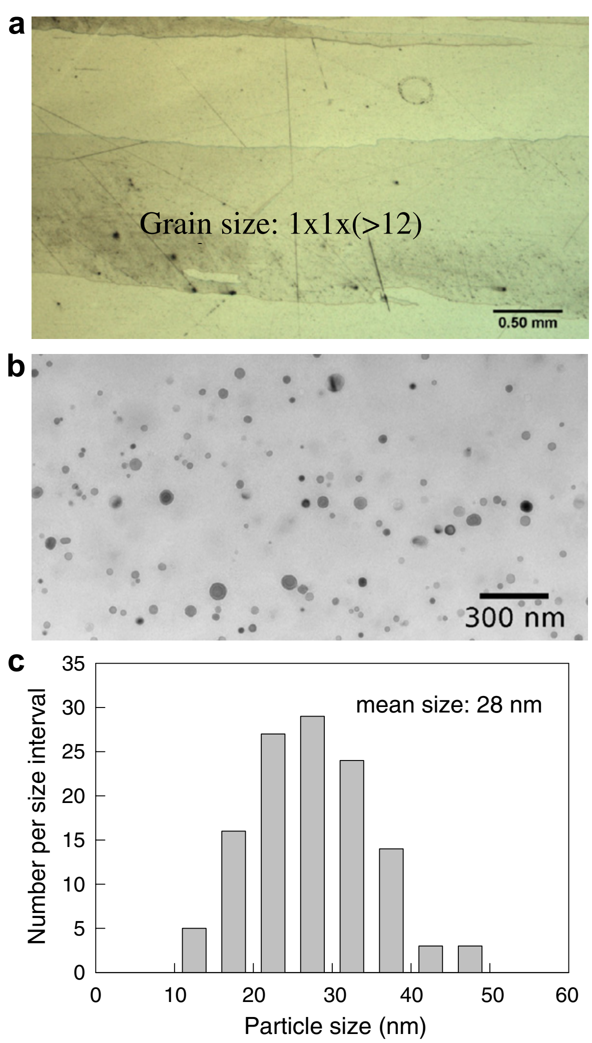

Fig. 1. Microstructure of as received PM2000 alloy, cut along longitudinal direction (a), TEM micrograph of microstructure (b), and size distribution of Y_2O_3 particles from image analysis (c).

In situ creep under He-implantation was performed at the compact cyclotron of Forschungszentrum Juelich. Details of the experimental set up are described in Ref. [15]. With 24 MeV $^4He^{2+}$ ions passing through a magnet scanning system and a degrader wheel with 24 Al-foils of variable thicknesses, the 0.1 mm thick samples were 3D-homogeneously implanted under constant uniaxial stress. Typical implantation rates were 0.023 ppm per second. The concurrent production of displacement damage was calculated by TRIM and SRIM for displacement threshold energy of 40 eV and a binding energy of 3 eV, giving per implanted He-atom 294 displacements on the front side and 194 on the back side, averaging to 244 displaced lattice atoms. With an average beam current density of 6.1 μA/cm^2, a displacement rate of about 5.5×10^{-6} dpa/s (displacements per atom per second) is derived. The irradiation creep strains were monitored by LVDT (linear variable displacement transducer) while the resistance was derived by a four-pole technique during beam-off periods. The implantation was continued until the strain rate became constant (stationary creep). Then the implantation of the same specimen was continued at a different stress in the range of 20–250 MPa. To minimize systematic errors from dose effects on the microstructure, e.g., by accumulation of irradiation defects, applied stress was changed alternatively to higher and lower values as indicated in Fig. 2. For each specimen, the temperature was fixed at 573, 673 or 773 K, respectively. The temperature distribution along the gauge region was monitored by an infrared pyrometer under 45° from the backside of the specimens. Finally, TEM specimens were prepared from the implanted gauge sections (see Ref. [16] for details) and TEM examinations were performed with a JEM 2010 at PSI.

3. Results

3.1. Irradiation creep

Fig. 2 shows the strain $\varepsilon(\sigma) = \frac{\Delta l}{l_0}$ of PM2000 during implantation as a function of the displacement dose at 573 (a), 673 (b) and 773 K (c), respectively. At all three temperatures, a contraction of the specimen against the applied tensile stress occurs at the beginning of irradiation, but already after 0.05 dpa, creep in the stress direction is observed. Each stress change caused, aside from elastic strain, also a short transient stage before stationary creep was reached. Those transient strains are similar to observations in other materials [17] and are ascribed to