Characterization of Precipitates in 12Cr F/M ODS Steel

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1. Introduction

Ferritic/Matenstic (F/M) oxide dispersion strengthened (ODS) alloys are attractive candidates for cladding materials of GEN-IV sodium-cooled fast reactor (SFR) because of their superior high temperature creep resistance. These F/M ODS steels are expected to be applied at high temperatures above 650ºC due to the nano-oxide particles dispersed in the F/M matrix, which acts as pinning points against dislocation motions. The high temperature creep strength and irradiation resistance of F/M ODS steels depend considerably on the size and number density of oxide particles. Great efforts have been done to make finer dispersoids with higher number density.

Y_2O_3 is most commonly added as reinforcement oxide, due to its stability at high temperature. Y_2O_3 dissolves into alloy matrix during MA (mechanical alloying) and precipitates as nano-oxide particles during HIPing or hot extrusion [1]. Some other elements like Ti or Al are also added to form finer precipitates like $Y_2Ti_2O_7$ and $YAlO_3$ with Y_2O_3 [2]. This study focuses on the characterization of precipitates in 12Cr F/M ODS steel.

2. Methods and Results

2.1 Experimental procedure

The 12Cr ODS steel investigated in this study was prepared by MA of argon gas atomized element powders along with 0.3 wt% Y_2O_3 powder in a Pulverisette-5 planetary mill at 200 rpm in high purity Argon gas atmosphere for 12h, with a ball to powder ratio of 15:1. After ball milling, the MA powders were transferred to a 304 stainless steel can and degassed at 500ºC for 1h. The degassed powders were consolidated by HIPing at 1150ºC under 100MPa pressure for 4h.

Specimens were taken from the hipped rod. Carbon extraction replicas were prepared from the mechanically polished surface. The surface was electroetched and a carbon film was coated and carbon replicas were removed by electroetching with a solution of 5% perchloric acid and 95% ethanol. Thin foil TEM samples were prepared by jet-polishing at 20V and 253K with the same etchant for electropolishing. The precipitates obtained from carbon extraction replicas and microstructure of the ODS steel were characterized by using a FE2100F transmission electron microscope (TEM) and an energy dispersive spectroscope (EDS) attached to the TEM. Precipitates in the extruded commercial ODS steel MA956 (Fe-20Cr-0.3Y_2O_3) were also investigated to make comparison.

2.2 Microstructure

Fig. 1 shows the TEM image of the precipitates obtained from a carbon extraction replica of the 12Cr F/M ODS steel. Particles are homogeneously distributed and have a size range from several to about 50nm. The precipitate size distribution was calculated from the particles in the area of analysis from TEM images. The mean particle size (diameter) and number density in the 12Cr F/M ODS steel are 9.1 ± 0.7 nm and $2.8 \pm 0.4 \times 10^{21}$ m^{-3}, respectively (Assume that all particles are sphere in shape and were extracted from a 100nm thick layer of matrix surface).

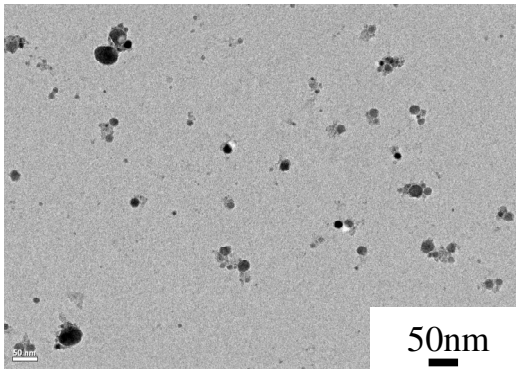


Fig. 1. Precipitates in the 12Cr F/M ODS steel (Extraction replica sample).

The size distributions of precipitates in the 12Cr F/M ODS and MA956 (containing mainly $Y_3Al_5O_{12}$ particles) are shown in Fig. 2. The distribution peak in the 12Cr F/M ODS steel appears at a size smaller than 5nm, while that for MA956 is at the size of about 15nm. Also, there is large difference in distribution fraction for small particles ranging from 5 to 10nm. These results indicate that the 12Cr ODS F/M steel sample has a much smaller mean particle size and a higher number density of fine particles than commercial MA956.

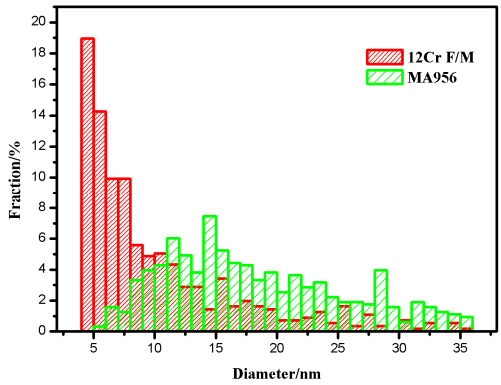
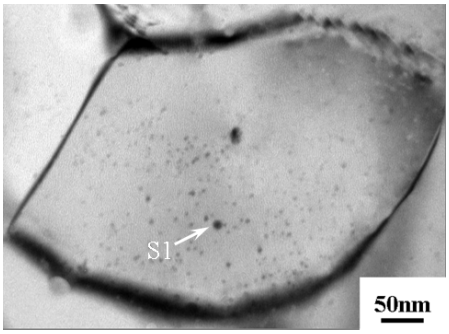
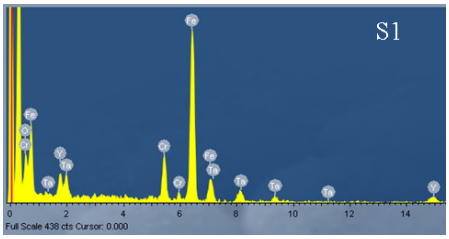


Fig. 2. Comparison of the size distributions of precipitates in the 12Cr F/M ODS steel and MA956 ODS steel.

Fig. 3 shows the microstructure of the 12Cr ODS steel and the EDS analysis of the precipitation marked by S1. Small nano-clusters with a size less than 5nm were observed, indicating that the specimen contains high density of the nano-clusters, which were not revealed in carbon extraction replica due to the resolution limit. The composition of the precipitate marked by S1 was identified to be Y-Ta-O type.



(a)



(b)

Fig. 3. (a) Microstructure of the hipped 12Cr F/M ODS steel and (b) EDS analysis of the precipitation marked by S1.

Fig. 4 shows the HRTEM image of the nano-particles. HRTEM study indicated that the nano-particles were mainly monoclinic $YTaO_4$ particles. Most observed precipitates were coherent with the matrix with a habit plane (110) and with $(100)_P//(110)_M$.

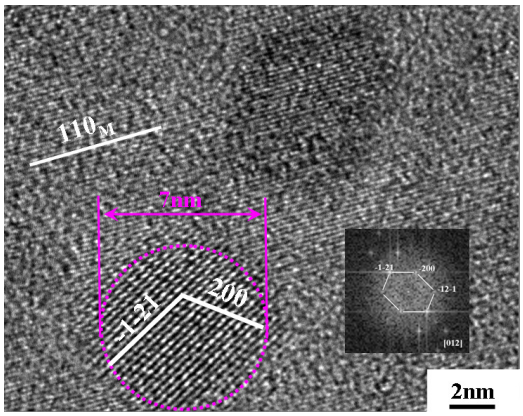


Fig. 4. HRTEM image with FFT of the fine nano-particle (circled by the pink ring) in the hipped 12Cr F/M ODS steel. The white lines in the figure show the lattice planes.

3. Conclusions

Precipitates in newly developed 12Cr F/M ODS steel were characterized by using HRTEM and EDS. Fine nano-particles with high number density were observed. Nanometer sized oxide particles were identified to be a monoclinic $YTaO_4$, and they were coherent with the matrix by $(100)_P//(110)_M$. The nano-sized precipitates with high number density show prospect of obtaining high mechanical properties.

Acknowledgements

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