Control of an Excess Oxygen Concentration of MA Powders for ODS Steels

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1. Introduction

Oxide dispersion strengthened (ODS) alloys are being considered as the candidate core materials in GEN-IV reactors mainly due to their excellent creep resistances and irradiation resistances at high temperatures [1]. In the ODS alloys, the mechanical properties are mainly determined by the relevant factors such as the alloying elements, oxide particles, grain size and so on. The precipitates imply their amount, type, size, morphology, stability and distribution. Many studies have focused on the formation of fine precipitates, thus improving the mechanical properties [2].

This study focuses on the formation of fine oxide particles by a control of the excess oxygen concentration, thus expecting the improved mechanical properties of ODS alloys.

2. Methods and Results

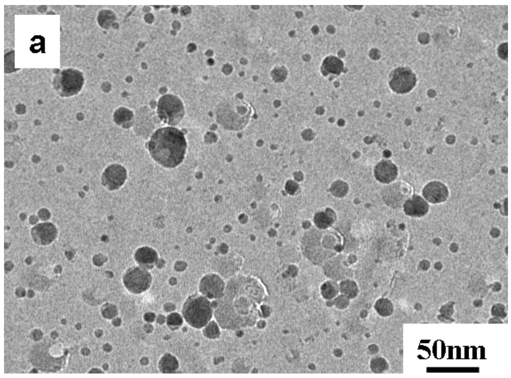
2.1 Experimental procedure

Fe-Cr-Ni ODS steels were designed and fabricated by mechanical alloying and hot isotropic pressing (HIPing) processes. The metal powders along with Y_2O_3 powders were mechanically alloyed by using the Pulverisette-5 planetary mill at 200 rpm in high purity argon gas atmosphere for 12 hr. A ball to powder ratio of 15:1 was applied. After the ball milling process, the MA powders were transferred to a steel can, and the oxygen concentration was controlled. Degassing was performed at 500ºC for 2 h, and then hipped at 1150^{o}C under a pressure of 100 MPa for 4 h. The precipitates taken from the carbon extraction replicas were examined by using a transmission electron microscope (TEM) with an energy dispersive spectroscope (EDS). The carbon extraction replicas were prepared by means of a mechanical polishing, etching with a mixed solution of 5% perchloric acid and 95% ethanol, carbon coating and removing the replicas by an electrochemical etching with a mixed solution of 90% methanol and 10% hydrochloric acid. The size distribution of the precipitates was measured by using an image analyzer.

2.2 Microstructure

Fig. 1 shows the TEM images of the precipitates obtained from carbon extraction replica samples in H0, H1 and H2 ODS steels. H0 was fabricated without reduction process, while H1 was reduced by 5vol.% H_2 and 95vol.% Ar mixed gas under 1atm pressure and H2 was reduced by flowing 5vol.%H_2 and 95vol.% Ar mixed gas for 2h before HIPing. Precipitates in H0 have an apparent larger size than those in H1 and H2, indicating than reduction has a significant effect on the size of precipitates in ODS steels. The mean particle sizes and particle number densities of the three ODS steels are shown in Tabel 1. The precipitate size distribution was calculated from the particles in the area of analysis from TEM images. The precipitates were assumed to be extracted from a 100 nm thick layer of the matrix surfaces when calculating the number densities. H2 has a mean size (9nm) smaller than half of that in H0. Since H0 and H2 have the same composition, it is expected that hydrogen reduction by flowing 5vol.% H_2 and 95vol.% Ar mixed gas can reduce the mean particle size of ODS steels. H2 has a smaller mean particle size and a higher number density than H1, indicating that reduction by flowing gas is more effective than reduction under a sealed atmosphere.

Excess oxygen is an important alloying element of ODS steels and controlling excess oxygen concentration turned up to be a key point to achieve high mechanical properties. These results show that hydrogen reduction by flowing gas is an effective method to control the excess oxygen concentration in ODS steels.



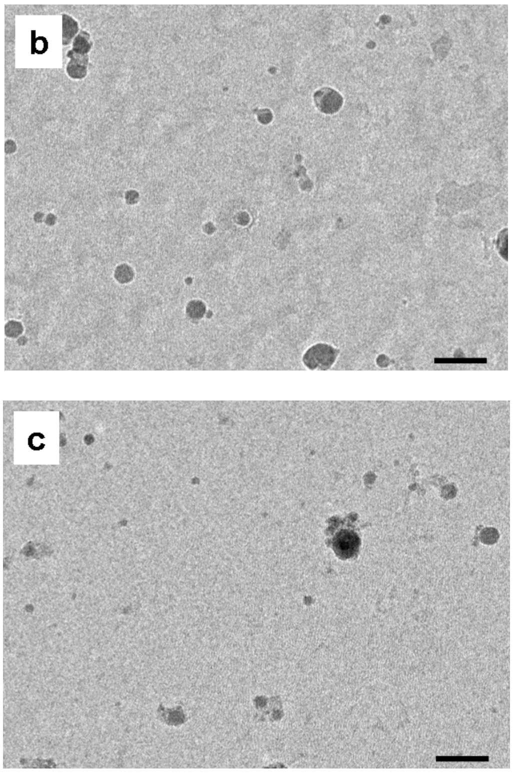


Fig. 1. Precipitates in (a) H0, (b) H1 and (c) H2 ODS steels. (Carbon extraction replica samples)

Table 1: Mean particle size and number density in H0, H1 and H2 ODS steels

	Mean size/nm	Number density/m^{-3}
H0	22 ± 4	$3.6 \pm 0.8 \times 10^{21}$
H1	14 ± 2	$1.7 \pm 0.6 \times 10^{21}$
H2	9 ± 1	$2.8 \pm 0.6 \times 10^{21}$

Oxide precipitates in the ODS hipped bars prepared by MA + HR + HIPing processes (H1 and H2) were much different from that of normal MA + HIPing/Hot extrusion processes (H0). Fig. 2 shows the EDS analysis of oxide precipitates in these ODS steels. H1 contains large particles (>100nm), which is Cr-rich precipitates, and fine Y-Ti-O and Y-Si-O particles. K1-H1 and K1-H2 only contain fine Y-Ti-O and Y-Si-O particles, indicating that large Cr-O precipitates were eliminated by hydrogen reduction. This should be achieved due to the reduction of excess oxygen concentration, since Cr-rich particles have a high oxygen concentration.

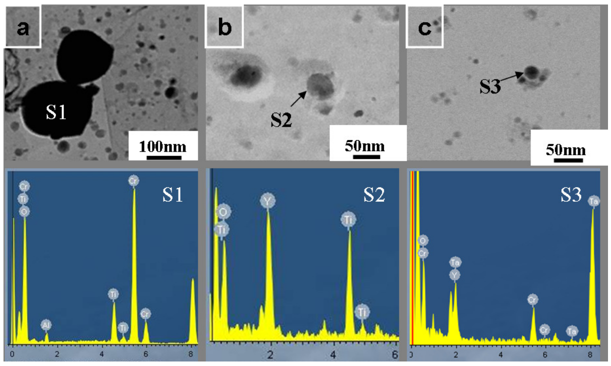


Fig. 2. EDS analysis of the precipitates in (a) H0; (b) H1 and (c) H2

Table 2: Precipitates contained in different ODS steels

	H0	H1	H2
Cr-rich	√	×	×
Y-Ti-O	√	√	√
Y-Si-O	√	√	√

3. Conclusions

In order to reduce the size of precipitates in ODS steel, especially irregular large Cr-rich precipitates, hydrogen reduction process by 5vol.%H_2 and 95vol.% Ar mixed gas was introduced before degassing. The observation of oxide precipitates in ODS steels prepared with and without hydrogen reduction indicated that HR process significantly reduced the mean particle size of the precipitates and eliminated large Cr-rich particles in ODS steels, which is prospective for preparing ODS steel of high mechanical properties.

Acknowledgements

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