

Investigation of Nano-sized Oxide Particles in ODS Steels

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1. Introduction

Oxide dispersion strengthened alloys are considered as good candidate materials for key components of future nuclear systems [1]; owing to their good radiation resistance and their excellent high temperature mechanical properties such as tensile property and creep rupture property. The superior characteristics of the oxide dispersion strengthened alloys are mostly attributed to the nano-sized oxide particles that are usually dispersed evenly within the metal matrix, and maintain their microstructure and the size distribution even under a significant stress at high temperature. Even the interface between particles and matrix is known to accommodate the radiation-induced defects such as vacancies or cavities, and can act as sinks for the defects.

In this study the behavior of the nano-sized oxide particles within ferritic or austenitic Fe-based steel is investigated by using electron microscope.

2. Experiments

2.1. Sample preparation

Oxide dispersion strengthened (ODS) steel samples were prepared through mechanical alloying (MA) with elemental metal powders less than 100 μm in size and Y_2O_3 powders of about 30 nm in size, followed by hot isostatic pressing (HIP), hot rolling, and a consequent heat treatment. The nominal composition of the ferritic and austenitic ODS steel samples was Fe-12Cr-1.1W-0.2V-0.14Ta-0.3Y₂O₃ in wt.%, and Fe-17Cr-12Ni-2.5Mo-0.3Ti-0.3Y₂O₃ in wt.%, respectively.

Elemental metal powders and yttria powders mixture was mechanically alloyed by using a planetary ball mill (Pulverisette-5) at 200 rpm in an Ar atmosphere for 12 hours with a ball to powder ratio of 15:1. The mechanically alloyed powders were degassed at 500 °C for 1 hour, and then hot isostatically pressed at 1150 °C under 100 MPa of Ar for 4 hours.

2.2. TEM sample preparation and observation

TEM (transmission electron microscope) samples of 3 mm diameter discs were prepared from hot rolled and then heat treated plate specimen by slicing, mechanical polishing, and twin-jet polishing. Twin-jet polishing was carried out at 25 V in 5% perchloric acid and 95% methanol mixture at -40 °C. Observation was carried out

by using JEOL FE2100F high-resolution TEM equipped with EDS, operated at 200 keV.

3. Results

3.1. Ferritic ODS steel

One example of nano-sized oxide particles within Fe-12Cr-1.1W ferritic steel matrix is shown in Fig. 1. HRTEM micrograph of an YTaO_4 particle with a size of 5 nm as well as the iFFT image of the particle and of the matrix are shown. The inter-planar spacing along the two major directions was estimated as 2.90 and 2.02 Å respectively with the inter-planar angle of 75.2° that appears to matched well with $d_{\text{YTaO}_4(1-21)}$ (2.94 Å) and $d_{\text{YTaO}_4(051)}$ (2.02 Å).

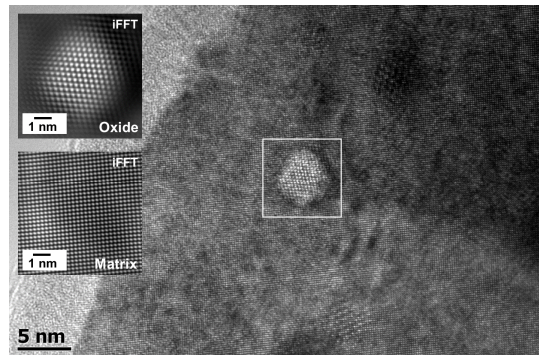


Fig. 1. Oxide particle within Fe-12Cr-1.1W ferritic steel matrix; HRTEM micrograph of YTaO_4 particle with iFFT images of the particle and the matrix respectively

Most oxide particles of fine sizes in this alloy specimen appears to maintain their microstructural stability with respect to the size distribution and the specific crystallographic orientation relationship with the matrix phase. They also demonstrated a good thermal stability up to 1150°C. Ledges were frequently observed at the particle/matrix interface on (HR)TEM micrographs.

3.2. Austenitic ODS steel

One example of nano-sized $\text{Y}_2\text{Ti}_2\text{O}_7$ oxide particles within Fe-17Cr-12Ni-2.5Mo austenitic ODS steel specimen is shown in Fig. 2. TEM analyses showed most $\text{Y}_2\text{Ti}_2\text{O}_7$ oxide particles within the austenitic ODS steel matrix reveal contrast-free line(s) at a specific \mathbf{g} vectors such as \mathbf{g}_{200} of which Ashby-Brown criteria [2]

could explain well. This means that most oxide particles within austenitic steel matrix maintain (semi-) coherent interface with the matrix.

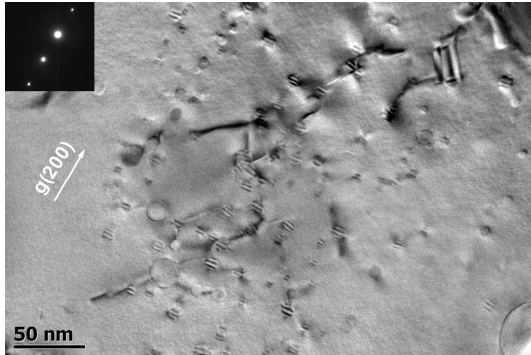


Fig. 2 Oxide particles within Fe-17Cr-12Ni-2.5Mo austenitic steel matrix; contrast-free line(s) within the fine particles demonstrating their coherent crystallographic relationship of the particles with the matrix phase

It is also well known that (semi-) coherent interface has about two times pinning effect on moving dislocations [3], presumably resulting in more effective strengthening consequence when compared with the incoherent one.

4. Summary

Nano-sized oxide particles in ODS steels were investigated by using (HR)TEM.

In case of Fe-12Cr-1.1W ferritic ODS steel the orientation relationship and interfacial structures of $YTaO_4$ nanoparticles were investigated by using (HR)TEM imaging techniques. Several specific orientation relationships were found between the $YTaO_4$ particles and the matrix. The thermal stability of the oxide particles was confirmed up to 1150°C.

In case of Fe-17Cr-12Ni-2.5Mo austenitic ODS steel most $Y_2Ti_2O_7$ oxide particles revealed to be maintaining the (semi-) coherent crystallographic relationship with the matrix.

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