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The interfacial orientation relationship of oxide nanoparticles in a hafnium-containing oxide dispersion-strengthened austenitic stainless steel

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ABSTRACT

This work reports comprehensive investigations on the orientation relationship of the oxide nanoparticles in a hafnium-containing austenitic oxide dispersion-strengthened 316 stainless steel. The phases of the oxide nanoparticles were determined by a combination of scanning transmission electron microscopy–electron dispersive X-ray spectroscopy, atom probe tomography and synchrotron X-ray diffraction to be complex Y–Ti–Hf–O compounds with similar crystal structures, including bixbyite Y_2O_3, fluorite Y_2O_3–HfO_2 solid solution and pyrochlore (or fluorite) $Y_2(Ti,Hf)_{2-x}O_{7-x}$. High resolution transmission electron microscopy was used to characterize the particle–matrix interfaces. Two different coherency relationships along with one axis-parallel relation between the oxide nanoparticles and the steel matrix were found. The size of the nanoparticles significantly influences the orientation relationship. The results provide insight into the relationship of these nanoparticles with the matrix, which has implications for interpreting material properties as well as responses to radiation.

1. Introduction

Oxide dispersion-strengthened (ODS) alloys, due to their outstanding creep resistance and radiation tolerance [1,2], are promising candidate materials for structural components in prospective energy systems including advanced fission and fusion reactors. The development of ODS alloys has primarily focused on ferritic or martensitic steels due to their advantages in mechanical strength [3,4]. However, austenitic steels have superior corrosion resistance to ferritic steels, and so have a greater potential for use in higher temperature applications. Hence, a series of austenitic ODS stainless steels [5–7], including a hafnium-containing ODS 316 steel [8], were produced in order to enhance the corrosion resistance with little compromise in mechanical strength and radiation tolerance, and to extend the application to higher temperature service conditions.

In ODS alloys, the interfaces between the oxide nanoparticles and the matrix significantly influence material performance. In particular, the challenge is to understand the fundamental physical processes responsible for stabilization of the oxygen-enriched nanoparticles to improve high-temperature strength and creep deformation performance. The orientation between the nanoparticles and the matrix determines the type of the nanoparticle-dislocation interaction, which is directly related to the strengthening mechanisms [9]. Also, the interfaces of dispersive nanoparticles can provide a great number of recombination centers for point defects [2,8]. In addition to point defect annihilation, helium atoms can be trapped by these interfaces to relieve irradiation-induced embrittlement or mitigate the tendency for helium to support void or bubble nucleation [10–13]. These phenomena are driven by the elastic interaction of the stress fields due to the matrix–nanoparticle interfaces and the various point or line defect structures [14,15]. The character of the matrix–nanoparticle interface is, in turn, controlled by the orientation relationship. Multiple advanced investigation methods have been employed to characterize these orientation relationships in ferritic ODS steels [16–19]. A recent high resolution transmission electron microscopy (HR-TEM) investigation in ODS 316 stainless steel discovered the existence of the cubic-on-cubic relation [20]. However, more complex chemical composition and orientation relationships,

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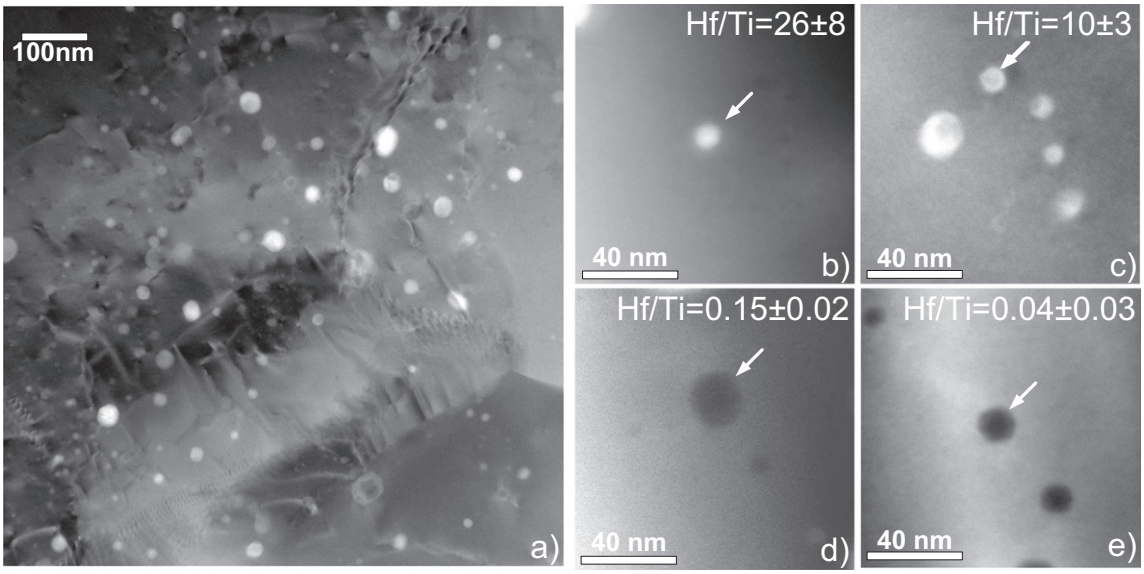

Fig. 1. STEM HAADF images: (a) typical microstructure of ODS 316 stainless steel; (b) through (e) $Y_2(Ti,Hf)_{2-x}O_{7-2x}$ nanoparticles with various Hf/Ti ratios. The discrete Hf/Ti ratios were selected only to show the significant variation of this quantity.

especially those related to the coherency mechanisms, are expected in the ODS 316 stainless steel and not covered by previous studies. In addition, the size effect of the nanoparticles on the orientation relationship is required to better understand the behavior of the nanoparticles as well as to guide the development of future austenitic ODS alloys.

The scanning transmission electron microscopy (STEM), combined with spectroscopies, such as electron dispersive X-ray spectroscopy (EDS) or electron energy loss spectroscopy (EELS), is powerful for the characterization of dispersed inclusion phases with diameters around 5 to 100 nm [21–23]. On the other hand, atom probe tomography (APT) was successfully utilized to analyze the oxygen-enriched nanoclusters with extremely small size (<5 nm) in ODS steels [24]. Therefore, the combination of STEM and APT techniques provides a method for chemical composition investigations that are capable of covering inclusions with a wide range of dimensions. In addition, the high energies and high intensities of X-rays produced by synchrotron radiation provide the opportunity to collect diffraction data from inclusion phases of low volume fractions, such as the oxygen-enriched nanoparticles in ODS steels, a task which is impossible with conventional X-ray sources. This paper reports the chemical composition and structure characterization by the joint efforts of STEM, APT and synchrotron X-ray diffraction (XRD) followed by an HR-TEM investigation of ODS nanoparticle size influence on the orientation relationship of oxide nanoparticles within a 316 stainless steel matrix.

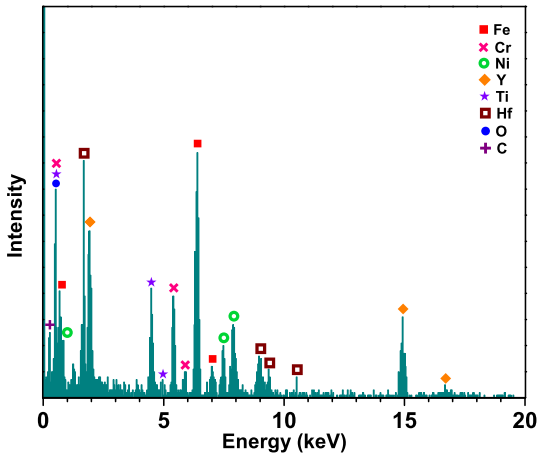

Fig. 2. A typical EDS result of an oxide nanoparticle showing the enrichment of Y, Ti, Hf, and O.

2. Description of experiments

The SUS316 stainless steel, Fe–16.16Cr–13.66Ni–2.33Mo–1.82Mn–0.08Ti–0.75Si–0.08(Nb+Ta)–0.05C by wt.%, was ball milled with 0.35 wt.% Y_2O_3, 0.1 wt.% Ti and 0.6 wt.% Hf for 24 h. The product was annealed at 1150 °C for 2 h before hot extrusion, and was finally annealed at 1100 °C for 1 h. The material was manufactured by Prof. Somei Ohnuki's research group at Hokkaido University [20]. The grain size of the ODS alloy 316 is approximately 0.5 m, and the oxide particle density is 6.6×10^{21} m^{-3} with an average size of 9.4 nm according to the manufacturer. The alloy was mechanically polished to 100 m and then punched into 3 mm TEM discs. Finally, the alloy was thinned to electron transparency by electropolishing with a solution containing 5 vol.% perchloric acid and 95 vol.% methanol at −14 °C.

The APT specimens were fabricated from in-situ lift-out specimens extracted from the surface of the electropolished 3 mm TEM disc specimen and then annular milled in a Dualbeam FEI Nova 200 Nanolab focused ion beam/scanning electron microscope (FIB/SEM) [25]. APT characterization was performed in an energy-compensated CAMECA Instruments Inc. local electrode atom probe (LEAP 4000X HR). Due to the poor electrical and thermal conduction of these materials, the specimens were analyzed in laser-mode at a specimen temperature of 30 K, a pulse repetition rate of 200 kHz, a focused laser beam energy of either 50 or 100 pJ, and a data collection rate between 0.5% and 4% ions per field evaporation pulse depending on the standing voltage applied to the specimen. The position of the laser beam on the apex of the specimen was adjusted automatically during the experiment to account for the field evaporation of material from the apex of the specimen and specimen drift. These conditions resulted in individual LEAP datasets containing up to 200 million atoms. Surface regions that contained damage from the gallium ion beam were not used for analysis. Data analysis was performed with the use of CAMECA Instruments Inc. Integrated Visualization and Analysis Software (IVAS 3.6.6). The proxigrams [26] were calculated according to the isosurfaces defined by 8% decomposed oxygen concentration. Thus, the full width at half maximum (FWHM) of the oxygen concentration in proxigrams was regarded as the size of the oxygen-enriched nanoclusters.