

Interfacial strained structure and orientation relationships of the nanosized oxide particles deduced from elasticity-driven morphology in oxide dispersion strengthened materials

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Abstract

Fe–Cr–Ti–Y₂O₃ oxide dispersion strengthened materials have a high density of nano-oxides (Y, Ti, O) that are stable at high temperature. In this work based on transmission electron microscopy it is shown that the interfacial strained structure and orientation relationship can be determined by using the elasticity-driven morphology of the nanosized particles after coarsening at 1573 K. In a Fe–14 Cr–1 W–0.3 Ti–0.3 Y₂O₃, most of the coarsened phases found are Y₂Ti₂O₇ particles adopting a cubical shape. They are embedded with a cube-on-cube orientation relationship with the matrix, while a few Y₂O₃ particles are embedded in both cube-on-cube and cube-on-edge orientation relationships with the matrix. This morphological transformation could reveal the coherent misfitting character of the nanosized parent phases. The kinetics of the elasticity-driven morphology is different for both phases. Based on the supersphere approach, the elastic and interface energy calculation is proposed. The corresponding value of the (100) interface energy is 260 mJ m⁻² for Y₂Ti₂O₇ particles and 350 mJ m⁻² for Y₂O₃ particles. The particle evolution during thermal annealing and (Y, Ti, O) coarsening resistance system is then discussed.

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1. Introduction

The interfacial structure of nanoscale phases has a great influence on the final microstructure of materials since it governs particle nucleation and growth. Oxide dispersion strengthened (ODS) materials are reinforced by nano-oxides homogeneously distributed within a ferritic matrix. Thus, understanding of the interface configuration of the nanophases is a key parameter to explain particle nucleation, growth and coarsening during processing. When a second phase is nucleated, the precipitate/matrix interface is classified as either coherent, partially coherent, or incoherent. If there is a difference in the lattice parameters between the particle and the matrix phases when the

particle is coherent or partially coherent, a misfit strain is created at the interface. It is well established experimentally that in many cubic lattice metals the equilibrium morphology of the second-phase particles with a cubic lattice is governed by the elastic stress induced by the misfit strain [1]. Thus, during growth and coarsening, the shape of an individual particle in the alloy is dictated by the elastic energy, which is proportional to the volume of the particle and the square of the lattice misfit ε^* [2]. When $|\varepsilon^*| > 0$ the morphology of the precipitates evolves from spherical to cubical with faces parallel to $\langle 100 \rangle$ [3]. For values of ε^* less than a critical value, which depends on intrinsic properties of the material such as the surface energy density, the particles remain spherical [4]. The shape transitions have been discussed in terms of the change in the balance of the interface energy of the matrix/precipitate interfaces and the elastic strain energy of precipitates [5]. Since the interface energy

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is proportional to the second power of the precipitate size and the elastic strain energy is proportional to the third power of the precipitate size, for smaller precipitates the interface energy is predominant while for larger precipitates the elastic energy becomes predominant. If the interface energy is isotropic, the particle will be spherical. On the other hand, the cubical shape evolution of larger precipitate is then explained by the effects of elastic anisotropy of on the cubic materials [1,6–8] by adopting the system symmetry. This shape dependence of the elastic energy is treated in the original work of Eshelby [9].

As the interface energy of the coherent precipitate is lower than for the incoherent precipitate [10], the nucleation of coherent phases is favored. In fact, the nanoscale oxide particles in ODS materials have a chance to be coherent with the matrix and to evolve to a cubic shape during growth if the interface is misfit strained. In the present paper the morphological evolution of nanoscale oxides in an ODS material during coarsening is studied. The evolution is explained by the superspherical approach [5,11] and by considering the nature and coherency of the phases.

2. Materials and experimental details

The behavior of nanoscale oxides during coarsening is studied in the new ODS material developed at CEA, which was obtained by mechanical alloying of a pre-alloyed Fe–Cr–W–Ti powder with Y_2O_3 powder in an attritor [12,13]. The mechanical alloying was performed by Plansee under a hydrogen atmosphere with prealloyed powders from Aubert & Duval. The studied alloy was hot extruded at 1373 K and then annealed at 1323 K. Its chemical composition is presented in Table 1.

In this paper, two conditions have been considered: the as-received material and the material annealed for 1 h at 1573 K. Transmission electron microscopy (TEM) observations were performed on a JEOL 2010F and on a JEOL 2100 microscope operating at 200 keV. The JEOL 2100 is equipped with a Gatan imaging filter (GIF) for energy filtered transmission electron microscopy (EFTEM) image acquisition.

3. Results

3.1. Morphological evolution of spherical nanoparticles during coarsening

TEM observations of the ODS material in the as-received conditions indicated that the precipitates are finely distributed. Their average diameter is 1.1 nm and their density is $2.9 \times 10^{23} \text{ m}^{-3}$ (Fig. 1a and c). Furthermore, the shape of these nanosized particles appears spherical at

the scale of Fig. 1a. However, at high magnification (Fig. 2a), the shape of the nanosized particles is not clearly distinguishable; moreover the high resolution TEM images (Fig. 2b), where only the matrix planes appear, highlight the real difficulty of characterizing the particles (appearing as black shadows) since they are really small.

After annealing, TEM observations indicate that particles are larger (Fig. 1b and c) and a morphological evolution of the precipitates from an aggregate shape (assumed to be spherical for theoretical interpretation) (Fig. 2a) to cube is clearly observed in Fig. 1b. It indicates that particles are not elastically separated from the matrix. The faces of the cubes are parallel to the $\langle 100 \rangle$ directions, conforming to the Maheshwari [2] predictions. The average equivalent radius ($r_{eq} = a/\sqrt{\pi}$ where r_{eq} is the equivalent radius for a cubic shape particle and a the edge dimension of the cube) of the cubical particles is 2.2 nm and their density is $3.4 \times 10^{22} \text{ m}^{-3}$. The change in particle interspacing indicates consuming particles by a coarsening process (Ostwald ripening). Some spherical particles still remain in the microstructure (Fig. 1b).

The EFTEM investigations confirm that the cubical particles are (Y, Ti, O)-type. The resulting energy-filtered images are presented in Fig. 3. The dark regions on the Fe $L_{2,3}$ image (Fig. 3a) show the local Fe deficiency and give a clear view of all kinds of precipitates in the matrix. The other two images acquired by the filtering of Ti $L_{2,3}$ (Fig. 3b) and O K (Fig. 3c) edges exhibit the same pattern of the bright spots which correspond to the oxide particles. This suggests that all oxide particles in the range of 1–10 nm have roughly the same chemical composition and are (Y, Ti, O) particles, even if no Y map can be acquired in filtered imaging since the Y $N_{2,3}$ edge is compromised by plasmon excitation. The Y $M_{4,5}$ edge has a delayed profile and the Y L_3 edge provides only a weak signal [14]. The particles are too small to offer a diffraction signal in conventional TEM. However, the HRTEM images (Figs. 4, 5a–d and b–d) show the atomic plane spacing and lattice structure of the particles and indicate that at least three types of oxides are present in the matrix: the $Y_2Ti_2O_7$ -type pyrochlore oxide (face-centered cubic (fcc), $a = 10.1 \text{ \AA}$) (majority phase) and the Y_2O_3 -type yttria oxide (body-centered cubic (bcc), $a = 10.6 \text{ \AA}$ and fcc, $a = 5.2 \text{ \AA}$), the lattice structures of which are represented in Fig. 7. The lattice structure of the Y-pyrochlore oxide is closely related to the fluorite structure except that there are two cation sites and one-eighth of the anions are absent (8a). Anion vacancies occur in a pyrochlore by virtue of the oxygen deficiency inherent in an $A_2B_2O_7$ compound [15]. The Y and Ti metal cations occupy the $16d(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ and $16(0, 0, 0)$ sites respectively, and the oxygens are in the $48f(x, \frac{1}{8}, \frac{1}{8})$ and $8b(\frac{3}{8}, \frac{3}{8}, \frac{3}{8})$ positions. The lattice structure of bcc yttria oxide is a bixbyite structure, where the Y^{3+} ions occupy the $8b(\frac{1}{4}, \frac{1}{4}, \frac{1}{4})$ and $24d(u, 0, \frac{1}{4})$ ($u = -0.0327 \text{ \AA}$) Wyckoff positions, and O^{2-} the $48(e)$ general positions [16]. The lattice structure of the fcc yttria oxide corresponds to a modified fluorite-type structure with one-fourth of the anion sites

Table 1
Chemical composition of the studied material (wt.%).

Cr	W	Ti	Mn	Si	Ni	C	Y_2O_3	Fe
14	1	0.3	0.3	0.3	0.15	0.5	0.3	Bal.

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