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TEM examination of microstructural evolution during processing of 14CrYWTi nanostructured ferritic alloys

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Abstract

A transmission electron microscopy (TEM) study was carried out on the co-evolution of the coarser-scale microstructural features in mechanically alloyed (MA) powders and hot isostatic press (HIP) consolidated Fe–14Cr–3W–0 and 0.4Ti–0.25Y_2O_3 nanostructured ferritic alloys (NFAs). The pancake shaped nanoscale grains in the as-MA powders are textured and elongated parallel to the particle surface. Powder annealing results in re-crystallization at 850 °C and grain growth at 1150 °C. The grains also recrystallize and may grow in the alloys HIPed at 850 °C, but appear to retain a polygonized sub-grain structure. The grains are larger and more distinct in the alloys HIPed at 1000 and 1150 °C. However, annealing resulted in bi-modal grain size distribution. Finer grains retained a significant dislocation density and populations of small precipitates with crystal structures distinct form the matrix. The grains and precipitates were much larger in alloys without Ti.

1. Introduction

A promising approach to achieving high creep strength, radiation damage resistant alloys is to create a very high density of fine-scale features that act as dislocation obstacles, serve as the dominant nucleation site for small helium bubbles and promote vacancy-interstitial recombination. A high density of coherent, nanometer-scale Y–O–Ti nanoclusters (NCs) can be produced in so-called nanostructured ferritic alloys (NFAs) by mechanical alloying (MA) Fe–Cr–Ti powders with Y_2O_3 followed by hot consolidation [1–7]. In a companion paper we show that MA dissolves the Y_2O_3 and that the NCs subsequently precipitate as coherent transition phases during high temperature consolidation treatments [7]. The NC number density decreases and size increases, between 850 and 1150 °C; and at higher temperatures Ti is a necessary ingredient in their formation. MA also produces a high density of dislocations and nanocrystallite grain substructures. These structures evolve during consolidation and subsequent heat treatments by processes of recovery, recrystallization and grain growth in a way that is expected to interact strongly with the co-evolution of NCs due to dislocation and boundary pinning mechanisms.

In order to optimize the array of key mechanical properties, ranging from creep strength to fracture toughness, it is necessary to first understand, and then control, these co-evolutions. Such understanding requires application of a variety of characterization techniques. We have focused on the use of small angle neutron scattering (SANS), and more recently three-dimensional atom probe tomography (APT), to characterize the NCs, which are difficult to study with typical transmission electron microscopy (TEM) techniques. However TEM, along with X-ray diffraction (XRD), is well suited to characterize the coarser-scale features including dislocations, grain/subgrain structures and larger Y–Ti oxide types of particles. Thus the objective of this study was to use TEM to characterize changes in the coarser-scale microstructures in both annealed and consolidated powders.

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2. Experimental

The pre-alloyed Ar gas atomized powders, provided by ORNL, with a nominal composition of Fe–14Cr–3W, were MA with (0–0.4)Ti and $0.25Y_2O_3$ in a high energy SPEX 8000 for 8 h in an Ar gas atmosphere at room temperature. The master alloy and Y_2O_3 powders were 45–150 and <1 μm, respectively. Milled 14CrYWT powders were canned and de-gassed prior to hot isostatic pressure (HIP) consolidation at 200 MPa for 3 h at 850, 100 and 1150 °C. The MA 14CrYW (without Ti) was also HIP consolidated at 1150 °C. The MA14-CrYWT as-milled powders were annealed at 850 and 1150 for 3 h.

Thin, electron transparent foils were prepared from the powders using a FEI Model 235 Dual-Beam Focused Ion Beam (FIB) instrument and micro-pick-up system. Standard 3 mm TEM discs were cut from the consolidated alloys were cut and ground to a thickness of 0.2 mm prior to thinning in a twin-jet electro-polisher with $HClO_4 + 95\%CH_3OH$ electrolyte at −20 °C. The FIB powder foils were mounted on carbon film supported copper grids and, along with the thinned discs from the consolidated alloys, were examined in a 200 kV JEOL 2010HR transmission electron microscope.

3. Results and discussion

The MA U14YWT powders have 'asteroid' shaped geometries with diameters ≈40–100 μm. Fig. 1 shows a cross-section TEM image of powder microstructures, as-MA and annealed at 850 °C and 1150 °C for 3 h. The pancake shaped grains in the as-MA powders shown in Fig. 1(a) are highly textured (texturing is also seen in the diffraction pattern) and elongated in directions parallel to the powder surface. The grains have typical thicknesses and diameters of 25–50 and 50–200 nm, respectively. The grains also contain a high dislocation density, which is difficult to quantify with TEM. However, the TEM results are reasonably consistent with XRD measurements showing characteristic crystallite size of about 20 nm and a lattice strain of 0.6% [8]. It should be noted that is difficult to distinguish distinct grain from subgrain dislocation structures on this size scale and we will refer to all such features as 'grains'.

Fig. 1(b) shows the effect of annealing at 850 °C. The grains appear to be much more isotropic with diameters of ≈50 nm, similar to the thicknesses of the as-milled grains. The diffraction pattern shows a significant reduction in texture, signaling that recrystallization has occurred. However, little or no grain growth occurs at 850 °C. There is no XRD data for the 850 °C anneals, but at 1000 °C the nominal XRD crystallite size is about 30 nm and the lattice strain of 0.05% is much lower than in the as-milled condition [8]. Fig. 1(c) shows that grain growth occurs during annealing at 1150 °C, resulting in a large range of grain sizes. Larger precipitates with diameters up to 20 nm are observed both in the grains and on the grain boundary.

Fig. 2 shows the corresponding microstructures of U14YWT alloys HIPed at 850, 1000 and 1150 °C. The image shown in Fig. 2(a) for HIPing at 850 °C is very difficult to interpret. However, there appears to be a wide range grain or polygonal subgrain sizes from about 25 to more than 500 nm. A significant density of dislocations is also observed and there are no obvious indications of small precipitates (although some of the smallest features may be larger oxide particles). As shown Fig. 2(b), recrystallization and grain growth occurs in the alloy HIPed at 1000 °C with both fine (<10 nm) and coarser (>30 nm) grain regions. The sizes of fine grains are reasonably consistent with the XRD crystallite sizes for annealed powders. The grains shown in Fig. 2(c) are generally larger in the alloy HIPed at 1150 °C, but there is still a wide range of sizes; and other parts of the microstructure are similar to those shown in Fig. 2(b) for the alloy HIPed at 1000 °C. The dislocation densities appear to be low in the larger grains in this case.

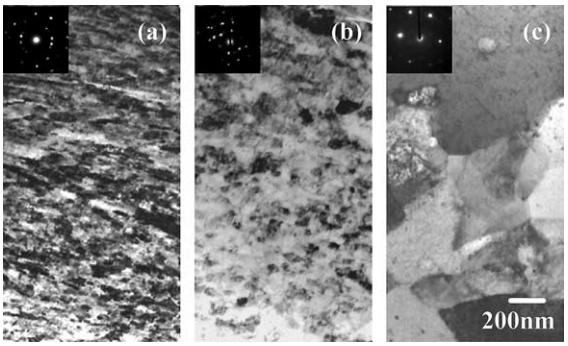

Fig. 1. TEM micrographs of cross-sections of the U14YWT powders: (a) as-MA; (b) annealed at 850 °C and (c) thermal annealed at 1150 °C.

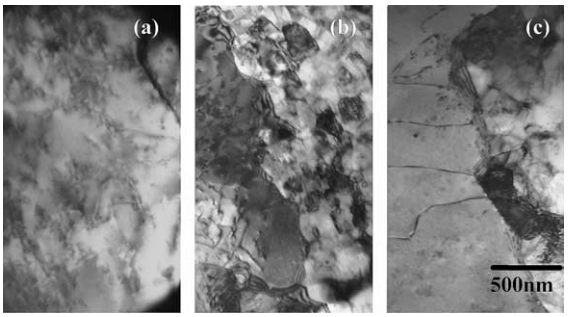

Fig. 2. TEM micrographs the HIPed U14YWT: (a) 850 °C; (b) 1000 °C and (c) 1150 °C.

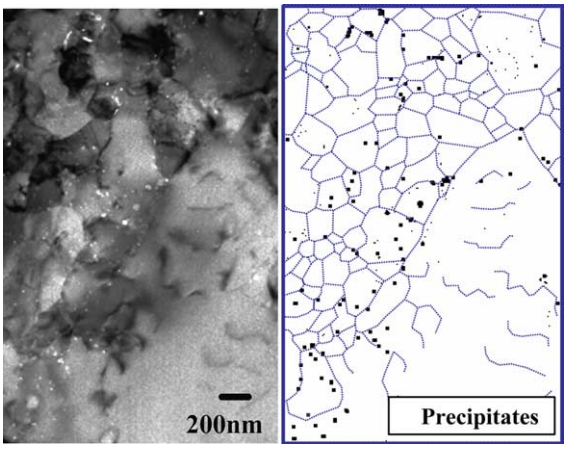

Fig. 3. Dark field TEM micrograph of U14YWT HIPed at 1150 °C and a schematic illustration of precipitate distribution.

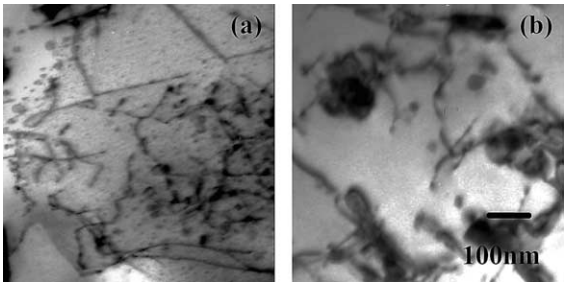

Fig. 4. TEM micrographs of U14YWT and U14YW HIPed at 1150 °C.

The dark field image of the 14YWT alloy HIPed at 1150 °C in Fig. 3 shows precipitates form on grain boundaries and dislocations. These precipitates are primarily observed in the fine grain regions, with diameters ranging from less than 10 to about 20 nm. There also appears to be a high density of similar, but smaller, precipitates in the matrix with diameters <10 nm. Fig. 4(a) shows a higher magnification bright field image of the precipitates in 14YWT HIPed at 1150 °C. Fig. 4(b) shows that the U14YW alloy, without Ti, has large dislocation free grains with much larger ≈100 nm grain boundary precipitates along with a low density of 20–40 nm precipitates in the interior of the grain.

4. Summary

The co-evolution of various microstructural features in NFAs, including grain sizes, dislocation structures and precipitates, has been investigated using TEM. Recovery/recrystallization occurs in both the powders and HIPed alloys at 850 °C. Grain growth occurs at higher HIPing and annealing temperature and there may even be grain growth in the alloy HIPed at 850 °C. In general, the grain structures tend to be larger in the HIPed alloys compared to the annealed powders, indicating a role of pressure induced deformation in these evolution processes. The grain sizes are generally bimodally distributed for all HIPed alloys and annealed powders at consolidation temperatures above 850 °C. The smaller grains retain a significant dislocation density and/or subgrain structure while the larger grains have a low dislocation density.

The observable precipitates that form have not been analyzed in detail. However, they are generally larger than the NCs observed in SANS [7], and dark field imaging indicates the presence of a crystal structure distinct from the matrix. Larger precipitates form on dislocations and particularly on grain boundaries. Significant dislocation structures are maintained in fine grain regions along with finer-scale precipitates. These observations indicate a strongly interactive co-evolution of these various features. Consistent with SANS observations of the NCs, the precipitates are much coarser in the 14YW alloy without Ti.

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