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Effect of milling temperature on nanoclusters and ultra fine grained microstructure of oxide dispersion strengthened steel

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ABSTRACT

The effect of ball milling temperature on the mechanical alloying and subsequent microstructural evolution of ferritic oxide dispersion strengthened steel was studied. The mixtures of Fe–14Cr–3W–0.4Ti powder and Y_2O_3 were ball milled for 40 h at room-temperature, −70 °C, and −150 °C. As milling temperature decreased, the fraction of low angle grain boundaries increased. Final grain size after consolidation was greatly affected by the ball milling condition. The size of nanoclusters seems to be dependent on milling temperature too. Ultra-fine grain size with homogenous dispersion of Y–Ti–O nanoclusters was achieved by very low temperature milling.

1. Introduction

The advanced oxide dispersion strengthened (ODS) steels offer great advantage in creep and irradiation resistance for high temperature reactor applications [1–4]. Recent progress shows that ultra-fine grained microstructure with a high density of Y–Ti–O nanoclusters can be produced by a controlled mechanical alloying (MA) and consolidation process [5–8]. However, these processes are not easy to control due to their very complexity. Under poor ball milling conditions, a mixture of coarse and fine grained structure, so called bimodal microstructure, is obtained. This bimodal structure originates from inhomogeneous Y_2O_3 distribution in the material [9], which results in inferior creep and irradiation resistance. In contrast, MA with sufficient milling force and time can induce uniform grain size with homogeneous Y_2O_3 distribution [9]. However, MA produces much heat from friction, vibration and plastic deformation. If the heat cannot be discharged in time, it will directly affect the efficiency of milling and the quality of the resulting powder [10]. Thus, the final mechanical properties are expected to depend on the cooling condition. Unfortunately, however, quantitative study on the effect of ball milling temperature is not yet available. The present work was undertaken to clarify the effect of ball milling temperature on the mechanical alloying and subsequent microstructural evolution of Fe-alloy + Y_2O_3 powder. In particular, grain size distribution and nanocluster formation were examined carefully.

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2. Materials and experimental procedure

A pre-alloyed metal powder with chemical composition of Fe–14Cr–3W–0.4Ti (wt.%) was prepared by the gas-atomization process. As-received powder particle size was 50–100 μm. The pre-alloyed powder was mixed with 0.3 wt.% Y_2O_3 powder of 30–50 nm particle size, and then put into a milling chamber. MA was performed in a high-purity Ar (99.999 wt.%) atmosphere for 40 h at room-temperature, −70 °C, and −150 °C with a temperature deviation of ±10 °C. Dry milling, whereby liquid nitrogen flows around the outside of the chamber, was utilized to avoid direct contact with the powder material. Milling temperature was monitored using a thermocouple inserted into the wall of the milling chamber. After milling, the Ar pressure level was checked to ensure that the seal was intact. The mechanically alloyed powders were filled into a mild steel can which was evacuated to a vacuum of ∼1 Pa at a temperature of 400 °C, sealed and consolidated by hot isostatic pressing at 1100 °C. Consolidated samples were hot rolled at 1150 °C with a thickness reduction of 50%, and subsequently annealed at 1100 °C. In order to investigate the morphology and microstructure of the materials, a JSM-7001F with an EDAX-TSL system was used for scanning electron microscopy (SEM) and electron backscattering diffraction (EBSD). The bright field images were taken to observe grain structure in a JEOL JEM-2200FS with Cs corrector. Regarding the imaging of NCs, energy filtered TEM (EFTEM) was performed at 300 kV on the same TEM machine with a Gatan image filter. Fe–M jump-ratio images were taken under beam condition of 10 eV slits at 46 and 62 eV. High quality images for NCs were revealed in dark contrast mode for sufficiently thin regions. Samples containing interested region were fabricated by dual beam focused ion beam (FIB, Helios Nano-Lab) to observe the microstructures of tensile specimens. In order to remove the FIB damage, low angle (4°), low energy (5 kV) Ar ion milling step was applied.

3. Results

3.1. EBSD analysis on the microstructure of MAed powder before and after consolidation

Table 1 summarizes the average particle size of MAed powders milled at room temperature, −70 °C, and −150 °C with respect to

Table 1
Average particle size of powders milled at room temperature, −70 °C and −150 °C.

Milling temperature	Milling duration (h)		
	0 (μm)	10 (μm)	40 (μm)
Room temperature		80 ± 40	70 ± 30
−70 °C	50 ± 20	35 ± 12	25 ± 10
−150 °C		15 ± 8	8 ± 5

milling time. The average powder particle size decreases as ball milling temperature decreases. The room temperature sample exhibited a coarse irregular shape while the −70 °C sample revealed a mixture of coarse and fine particles. The powders seemed to experience a high degree of flattening. In contrast, milling at −150 °C produced the finest particles along with reduced irregularity. This is mainly due to the suppression of cold welding and enhanced brittle fracture at low temperature.

Fig. 1 shows SEM photo of as-received powder and EBSD photo indexing the ferrite phase of as-received Fe–14Cr–3W–0.4Ti powder before MA. Very coarse grains with an average diameter of ∼18 μm were observed in the EBSD photo. Many grain boundaries appear to be low angle grain boundaries below 15° but a quantitative analysis was not carried out because one powder particle contains only a few grains.

In contrast to the as-received powder, MAed powders showed a fine microstructure. However, among the MAed powders, the room temperature sample contained relatively coarse and elongated grains (see Fig. 2). The relatively large grains (>500 nm) comprised more than 50% of the total volume of the grains. The grain size histogram of the room temperature sample suggests a bimodal grain size distribution: one broad and higher peak in the small grain size regime, another small peak in the large grain size (around 1 μm) regime (see Fig. 2d). As milling temperature decreases, the bimodal distribution changes to a single distribution at the smaller grain size regime; i.e., the larger grains disappear. The −150 °C sample in particular exhibited a very fine grain size, with an aspect ratio of less than 2. It is clear that the grain size is much more uniformly distributed in cryomilled powder. Although there is a difference in grain size distribution with regard to milling temperature, the average grain size of all powders did not exceed 1 μm. It is noted that the number fraction of low angle boundaries increases as milling temperature decreases (see Fig. 2e). Notably, the number fraction of low angle boundaries between 2° and 5° is 1.7 times higher at −150 °C than at room temperature. In contrast, the number fraction of high angle boundaries is slightly higher for room temperature milled powder than for powder milled at −150 °C.

Powders ball milled at different milling temperatures were consolidated by hot isostatic pressing, and then hot rolled and subsequently annealed at 1000 °C. Microstructures of the resulting ODS steels were examined by EBSD technique again as shown in Fig. 3. Room temperature milled ODS steel (RT steel, hereafter) showed very coarse grained microstructure with inhomogeneous grain size distribution and −70 °C milled ODS steel also exhibited bimodal structure. In contrast, −150 °C milled ODS steel (−150 °C steel, hereafter) showed fine homogeneous grain size distribution (see Fig. 3c and d). Compared with Fig. 2, it is noted that room temperature milled powders experienced significant grain growth during consolidation and subsequent hot working, while −150 °C powders maintained a nearly constant grain size distribution. It is not usual that such ultra-fine grained structure can be maintained even after 1100 °C consolidation process. This striking difference in microstructure evolution might be due to the fact that very low temperature milling induced homogenous dispersion of Y_2O_3 throughout the microstructure, and then homogeneous precipitation of nanoclusters [9]. It was reported roughly one fourth of all nanoclusters were located at grain boundaries preferentially [7]. Thus, these grain boundaries decorated with a high number density of nanoclusters can be effectively pinned by a Zener effect. This result stresses that fine homogeneous distribution of Y_2O_3 is much more important than the initial grain size of ball milled powder in order to obtain fine microstructured ODS steel.

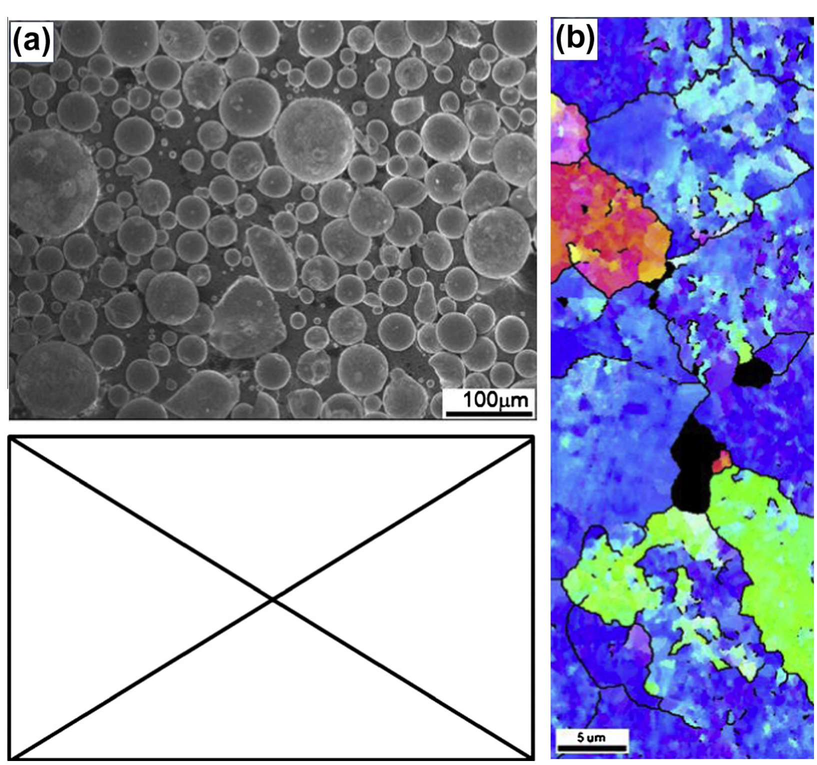

Fig. 1. Micrographs of as-received powder before milling; (a) SEM photo of powder particles and (b) EBSD photo indexing ferrite grains. Black areas in Fig. 1b are porosities.