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Reduced argon bubble formation in oxide dispersion strengthened steels by high-energy mechanical alloying

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ABSTRACT

We report a dramatic suppression of argon bubble formation in oxide dispersion strengthened steels through a high-energy mechanical alloying route. The volume fraction of argon bubbles trapped at the surface of the oxide particles is greatly reduced from 2.1×10^{-3} to 1.4×10^{-4}%, when the milling energy input rate is increased from 20 to 190 kJ/g·hit. It is found that the higher milling energy, associated with a reduction of milling time, yields reduced argon contamination of the ball-milled powders, leading to enhanced microstructural homogeneity in consolidated oxide dispersion strengthened steels.

Oxide dispersion strengthened (ODS) steels have attracted interest on the basis of their superior swelling and creep resistance at high temperatures, and resultant potential applications to structural materials for advanced nuclear fission and fusion reactors [1,2]. Finely dispersed stable oxide particles contributing to the excellent properties of ODS steels are usually introduced into the steel matrix through a powder metallurgical route, which involves a mechanical alloying (MA) process, followed by heat-assisted consolidation such as hot isostatic pressing and hot extrusion [3,4].

One of the critical issues involved in the ODS steel fabrication is the formation of argon bubbles. Argon is generally used as an inert shielding gas to prevent oxidation of powders during MA. However, it is incorporated into the powders and finally forms gas bubbles after consolidation. Several recent studies have shown that it is difficult to avoid argon bubbles in ODS steels [5–7] even with high-temperature degasification (~1200 °C) [8]. Crucially, the argon bubbles, even when present in small proportions, are believed to play a significant role in forming cavity nuclei, which are responsible for reduced ductility or degraded dynamic fracture toughness [4] and creep rupture [9]. Efforts have also been made to replace the argon with hydrogen and nitrogen [6], but the hydrogen is generally considered as increasing the brittleness [10] and the nitrogen causes the formation of unwanted nitrides in ODS steel [6].

Herein, we report that the formation of argon bubbles in ODS steel fabrication can be greatly suppressed by increasing the milling energy during MA. A very high-energy milling technique, which is capable of giving a nearly ten times higher energy input rate compared to the conventional milling processes [11], was employed to fabricate Fe-based ODS steels in conjunction with subsequent hot isostatic pressing. The resultant microstructure was investigated with a focus on argon bubble formation in comparison to ODS steels fabricated by a traditional low-energy milling process.

ODS steel powders with a composition of Fe-14Cr-0.3Mo-0.25Y_2O_3 in wt.% were produced by MA of high purity elemental Fe, Cr, and Mo raw powders together with nano-sized Y_2O_3 particles (40–50 nm) in an argon atmosphere. A home-made, very high-energy ball mill apparatus, specially designed to provide a higher milling intensity, was employed for MA (the details of this system are described elsewhere [11,12]). For a comparative study, MA of an identical powder mixture was performed using a conventional low-energy ball mill machine (Pulverisette-6, FRITSCH, Germany). From the theoretical collision model describing the energy transfer of the grinding ball to the powder [13], the milling energy input rate was determined to be 190 kJ/g·hit for high-energy ball milling carried out at 400 rpm and 20 kJ/g·hit for low-energy ball milling at 300 rpm. The milling times were optimized for each milling condition to ensure compositional and microstructural uniformity of the milled powders as well as a

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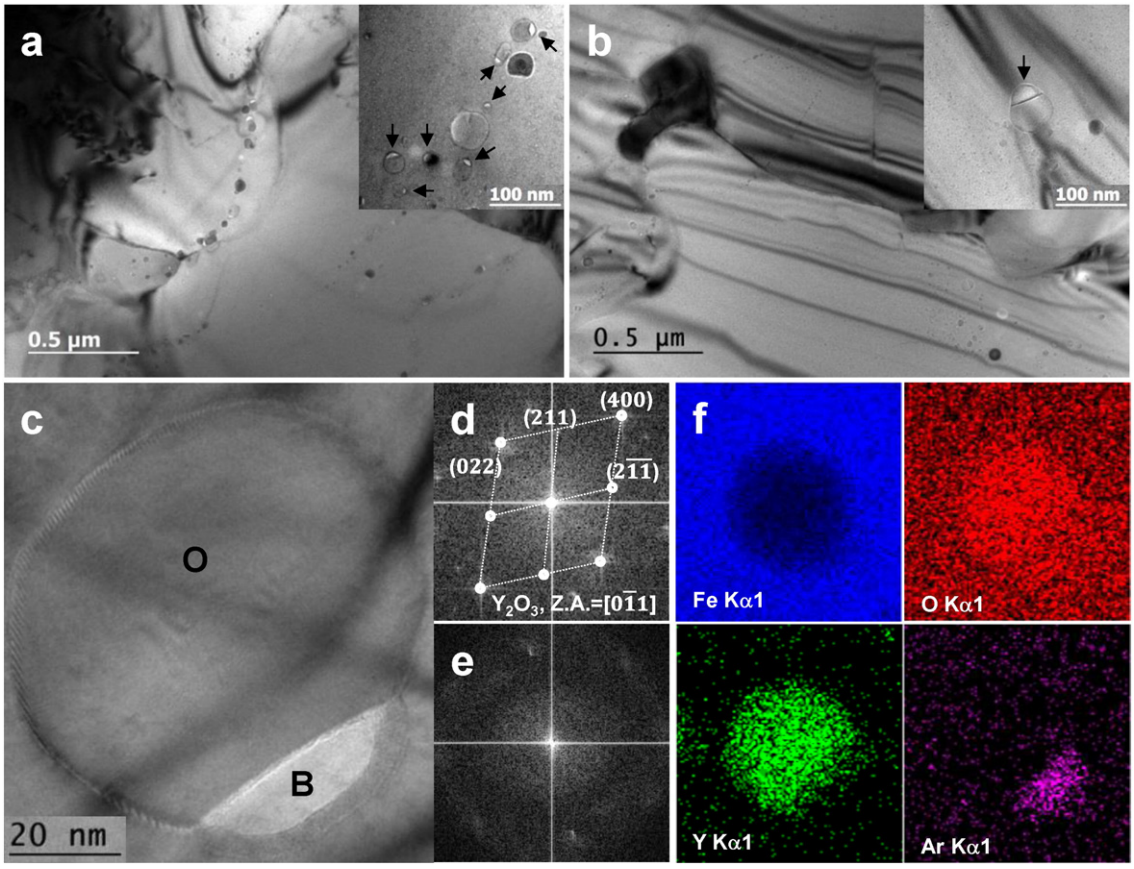

Fig. 1. Under-focused TEM micrographs of the (a) "L72h" and (b) "H100m" samples, (c) a high-magnification TEM micrograph of a coarse oxide particle (marked by "O") decorated by a bright contrast area (marked by "B"), (d) and (e) the corresponding fast Fourier transformation images of the region "O" and "B" in (c), respectively, and (f) EDS maps for the BTO particle in (c).

complete alloying of each metallic element. Through preliminary experiments, 100 min and 72 h were adopted for high-energy and low-energy ball milling, respectively. The milled powders were commonly degassed at 300 °C for 3 h under a vacuum of 10^{-4} Torr in a pre-sealed low carbon steel can, followed by consolidation by means of hot isostatic pressing (HIP, KOBELCO HP800) at 1165 °C for 3 h under 100 MPa. The two types of ODS steel samples prepared by low-energy and high-energy ball milling are hereafter referred to as "L72h" and "H100m", respectively. Both samples exhibited little difference in their densities (7.683 and 7.693 g/cm^3 for "L72h" and "H100m", respectively). The formation behavior of nano-sized oxide particles and argon bubbles in the fabricated ODS steels was comprehensively studied using a field emission transmission electron microscope (FE TEM, JEOL JEM-2100 F, Japan) equipped with an energy dispersive spectroscope (EDS). The TEM investigations were performed with an accelerating voltage of 200 kV and a beam size of ~1.3 nm was used for local EDS measurements. The presence of argon bubbles was determined by a series of in-focus and under-focus observations [7] and the highest contrast was obtained from the under-focus adjustment of 1–1.5 μm. To obtain the volume fraction of argon bubbles for each sample, a quantitative analysis was carried out using a number of TEM bright field (BF) images (analyzed images = 20 pictures per sample, measurements = 3 times per image, total measurement area = 13.5 μm^2).

Figs. 1a and b are under-focused TEM BF images showing the formation characteristics of oxide particles in the ODS steels prepared at different MA conditions. Each sample shows an apparently bimodal distribution of fine particles (less than 10 nm in size) and abnormally coarse particles (over 20 nm in size). Note that small bright spots (marked by arrows in the insets of Figs. 1a and b) are clearly visible, indicating argon bubbles trapped onto the coarse particles, whereas few of these spots are seen near the fine particles. A high-magnification TEM image for a coarse oxide particle is presented in Fig. 1c. The coarse particle "O" corresponds to a *C*-type cubic Y_2O_3 phase (space group *Ia*3, $a = 1.059$ nm), as identified by the fast Fourier transformation (FFT) image in Fig. 1d. Both the FFT image (Fig. 1e) and EDS mapping by the Ar Kα signal (Fig. 1f) taken from the bright region "B" confirm the presence of argon bubble. It is also found that these argon bubbles are predominantly present at the interfaces of oxides in the ODS steels, based on our TEM observations, consistent with a previous study [5]. It should be noted that the formation of these coarse bubble-trapped oxide (hereafter described as BTO) particles in the ODS steels is highly sensitive to the applied energy input rate in the MA. Numerous BTO particles are arrayed in a line in the "L72h" sample and these BTO lines are readily found throughout the sample (Fig. 1a). On the contrary, the BTO particles are almost negligible in the "H100m" sample and the overall distribution of oxide particles is more homogeneous (Fig. 1b).

Table 1 shows the average particle sizes and number densities of oxide particles for the "L72h" and "H100m" samples. Owing to the same HIP condition, no significant variation in average particle size of both the fine and coarse oxide particles was observed with the applied energy input rate in the MA, but the number density of coarse particles

Table 1
Average particle sizes and number densities of overall oxide particles for the "L72h" and "H100m" samples.

	Fine particles		Coarse particles	
Samples	L72h	H100m	L72h	H100m
Average particle size (nm)	3.8	4.1	32.7	27.0
Number density (m^{-3})	1.55×10^{22}	1.86×10^{22}	6.44×10^{20}	3.57×10^{20}