

Microstructure and tensile properties of oxide strengthened ferritic steel fabricated using as-milled powders with dissolved oxygen



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HIGHLIGHTS

- The work proved the “O-Vacancies Pairs Mechanism” and applied it in experimental.
- Hard and soft microstructures’ separation during hot processing was identified.
- Precipitate type particles were excellent in strengthening metal–matrix composites.
- Bimodal distribution of oxides exhibited two strengthening mechanisms.

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ABSTRACT

Pre-alloyed ferritic powders were milled for 10 h in the air atmosphere to introduce O atoms into the steel matrix. Then the milled powders which contained dissolved O were used to fabricate ferritic steels by hot processing. The precipitation of oxide, microstructural evolution and tensile properties of ferritic steels were studied. Results show that about 1.4 wt.% of oxygen was introduced into powders, and the powder surface has a higher O content (about 2–4 wt.%) than the inner region (about 1 wt.%). During hot processing Cr₂O₃ and Y₂TiO₅ were precipitated as the main second-phases. Surface regions of powders in which Cr₂O₃ largely precipitated were elongated, cracked then gathered, and dispersed in the steel matrix along the hot working direction. Tensile results show that the ultimate tensile strength of as-prepared steel in room temperature was 1601 MPa and was still high (1335 MPa) at 550 °C. The excellent tensile strength under 550 °C can be ascribed to the reinforcement of high content of oxide particles in the matrix. The tensile strength suffered a great reduction at temperatures between 550 °C and 650 °C, but it decreased slower at higher tensile temperatures.

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1. Introduction

The oxide dispersion strengthened (ODS) steels containing nanometer sized precipitates in the steel matrix have been chosen as potential materials using in some key parts of fast fission reactors and future fusion reactors. This is due to their superior resistance to irradiation-induced swelling and excellent mechanical properties in high temperature environment [1–3]. Preparation of ODS steels is traditionally achieved by mechanical alloying of ferritic powders with a small amount of Y₂O₃ powders, subsequent

hot working and heat treatment [4–6]. The Y₂O₃ powders were decomposed as Y and O solute atoms during milling. However, the break of high energy Y–O bond in Y₂O₃ by milling consumes a lot of time and energy. Besides, alloys fabricated using powders prepared at long milling time typically developed pores during high temperature annealing. As a result the mechanical properties of alloys are degraded greatly [7].

In recent years, much research concentrated in exploration of suitable substitution of Y₂O₃ has been done in order to improve the fabrication of ODS steels. For examples, Brocq et al. [8] used YFe₃ + Fe₂O₃ powders as Y and O carriers to fabricate ODS steels while Wen et al. [9] used YH₂ + Fe₂O₃ powders. Both methods induce the nucleation of fine Y–Ti–O precipitates that dispersed in the matrix. The sizes of these precipitates are in the range of 50–100 nm. On the other hand, Zhang et al. [10,11] mechanically milled the Y pre-alloyed gas-atomized ferritic powders with Fe₂O₃

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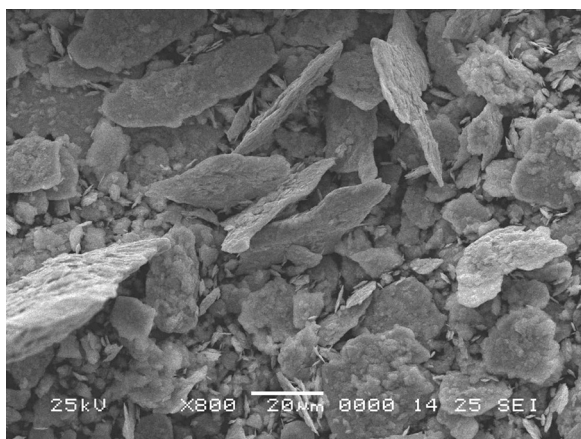


Fig. 1. The morphology of particles of powder milled for 10 h.

powders. Several phases composed of Ti, Y, Cr and O in varied particle sizes were formed in consolidated steels. However, the aforementioned methods, which used Fe_2O_3 as the substitute for O carrier, neither reduced the challenges during fabrication, nor shortened and the milling time (still longer than 40 h). As reported by Fu et al. [12], if vacancies exist in the matrix of ferritic steels, the formation energy of O-vacancy pairs will almost be reduced to zero. Namely, the concentration of O atoms can reach as much as that of the vacancies. A large number of vacancies can be generated in the matrix of ferritic powders during mechanical milling. As a result, the ferritic powders can absorb O atoms from the air. In this work, we milled pre-alloyed ferritic powders in the air, which act as the O carriers, in order to obtain dissolved O in ferritic powders. The ODS ferritic steels was then fabricated by subsequent hot working and heat treatment.

2. Materials and experimental procedure

Gas atomized pre-alloyed ferritic powders with the chemical composition of Fe–13.91Cr–4.03W–0.32Ti–0.19Y (wt.%) were milled by a planetary ball mill with 500 mL jars for 10 h at the speed of 300 rpm with a 10:1 ball to powder weight ratio (BWR). The metallic air valve in the milling bowl was not utterly closed, so that the air can enter into the bowl during milling. The as-milled powders were transferred into mild steel cans, which then were evacuated to a vacuum of ~ 1 Pa and sealed. The sealed cans were heated at 1150 °C for 30 min and then extruded. The as-extruded bars were rolled at 1000 °C followed by an annealing at 1200 °C for 1 h.

The O/N contents were analyzed using a TCH-600 H/N/O determinator. Phase structure was determined by a Rigaku D/max 2550VB+ X-ray diffractometer (XRD). Particle size of powders was measured by MICRO-PLVS Laser Particle Size Analyzer. The JXA-8530F electron probe microanalyzer (EPMA) was used to measure the elemental contents on the cross-section of powders. Microstructures were observed by Nava Nano SEM 230 scanning electron microscope (SEM) and JEOL-2100F transmission electron microscope (TEM). Tensile tests were carried out using Instron-3369 from ambient temperature to 750 °C under Ar atmosphere with a strain rate of 10^{-3} s^{-1} , using dog-bone type rectangular cross-section specimens with the gauge size of 8 mm \times 3 mm \times 2 mm.

3. Results

3.1. Powder characterization

Fig. 1 shows the morphology of as-milled ferritic powders. The nearly spherical shaped particles were kneaded into flaky shape,

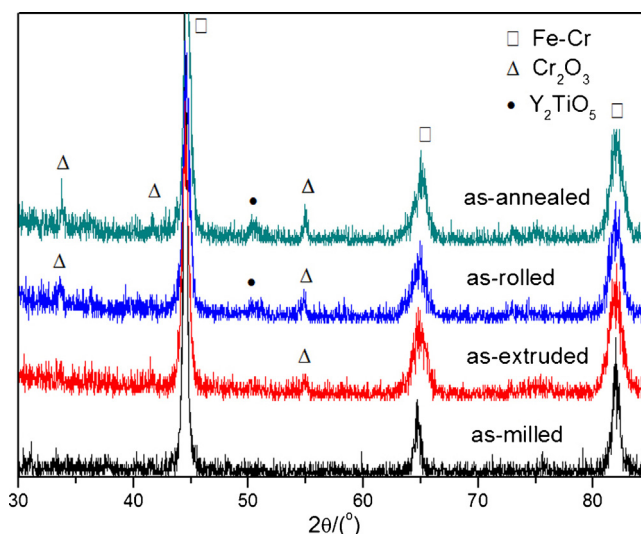


Fig. 2. XRD patterns of ferritic samples.

and the mean diameter of particles was reduced from 72 to 36 μm during milling. The average O and N contents in as-milled powders were measured as 1.4 wt.% and 0.01 wt.%, respectively. This O content largely exceeded the equilibrium O solubility in iron [13]. The XRD curve of as-milled ferritic powders, in Fig. 2, shows only evident Fe–Cr ferritic diffraction peaks without other diffraction peaks, indicating no detectable oxides in as-milled powders at the scale of observation. Therefore, it may be concluded that the O atoms were mainly dissolved in the ferritic matrix or existed in other defects induced by milling.

Fig. 3 shows the EPMA results of as-milled powders. Fig. 3a gives the backscattered electron graph of the cross-section of two particles of as-milled powders. Fig. 3b gives the elemental weight percentage distribution of the two particles along the white line in Fig. 3a. The O content distribution curve shows that the O content at the surface region of particles is as high as 2–4 wt.%, while it is about 1 wt.% in the inner region of particles. Other elemental content distribution curves indicate metallic elements are distributed nearly homogeneously in particles of as-milled powders. It has been measured that the O content in ferritic steel has the same value of that in as-milled powders, which was 1.4 wt.%. Fig. 2 indicates that: (1) Cr_2O_3 oxides largely precipitate after extrusion; (2) Y_2TiO_5 oxides are measurable after hot rolling; and (3) the both oxides contents increase after annealing.

3.2. Microstructural evolution

Fig. 4 shows the back scattered electron images and energy dispersive spectrometer (EDS) results of the as-annealed steel. Regional differences in the microstructure are readily observed in Fig. 4a that two kinds of typical regions were arranged roughly along the rolling direction. One kind is the clearest ones (marked as rectangle b for example) dispersed with less and smaller black spots and the other is the darkest ones (marked as rectangle c for example) with more and larger black spots. The clearest region, magnified in Fig. 4b, is the matrix homogeneously dispersed with fine black spots ($< 500 \text{ nm}$) both in grains and on grain boundaries and the average grain size of ferritic matrix is about 2–5 μm . The darkest region, magnified in Fig. 4c, is the matrix dispersed with coarser black spots ($< 3 \mu\text{m}$). The grain boundaries of ferritic matrix of this region were not well etched that the grain size can only be roughly estimated as 1–10 μm . The difficulty to observe the grain boundaries may be attributed to the preference of solution used for etching to react within interfaces between ferritic matrix and

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