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THE EFFECT OF ADDITIVE ELEMENT ON THE PROPERTIES OF MECHANICALLY ALLOYED Fe-Y_2O_3 ALLOYS

In the present work, we have examined the effect of Ti on the properties of Fe-Y_2O_3 alloys. The result showed that the addition of Ti was effective for improving mechanical properties. This is due to the reduction of oxides by Ti during mechanical alloying and hot-consolidation. In particular, iron oxides are effectively reduced by the addition of Ti. Compared to the pristine Fe-Y_2O_3 alloys, titanium-added alloys exhibited fine and uniform microstructures, resulting in at least 60% higher tensile strength.

Keywords: Mechanical alloying, ODS alloys, Nanoparticles, Oxide-dispersion Strengthening, High-energy ball milling

1. Introduction

ODS (oxide-dispersion strengthened; ODS) alloys have various interesting mechanical properties such as good creep resistance and thermal stability for high-temperature applications [1,2]. Such enhanced properties can be attributed to fine and uniform distribution of dispersing oxide particles in a metallic matrix phase. In particular, ferrous ODS alloys such as ferritic or ferritic/martensitic steels have been considered as one of the most promising candidate materials for cladding alloys in Generation IV (Gen-IV) nuclear reactors which will be deployable in near future [3-5]. However, the reported properties of these alloys are still insufficient for the practical application to GEN-IV reactors. Several alloy compositions have been investigated for the optimization of the material properties, in particular, to form and retain nano-sized dispersoids. It has been reported that a small addition of several elements such as Ti or Al in the ferrous ODS alloys is beneficial for multi-component systems which usually contain more than 7~8 elements [6]. The exact cause of the improvement is unclear. It is thus important to clarify whether the beneficial effect is due to the addition of Ti alone or a combined effect with other additive elements.

For this purpose, we examined the effect of additive Ti on the microstructure and properties in simple binary Fe-Y_2O_3 alloy systems.

2. Experimental

The starting materials were elemental Fe (>99%, ~200 μm) and Y_2O_3 (<25 nm, Aldrich), Ti (99.8%, 50 μm) powders. Prior to mechanical alloying, the powders were mixed to give four different compositions. The examined compositions were Fe- 1% and 2 wt.% Y_2O_3 with or without the addition of 0.5 wt.% Ti. Detailed compositions examined in the present work are shown in Table 1. Mechanical alloying, or high-energy ball-milling, was performed using a Szegvari attrition mill (model S1) at a rotating-arm speed of 200 r.p.m. Both milling balls and vial were hardened stainless steel. Th diameter of ball was 6.4 mm. The balls were charged into the vial with 50% vol. % (5 kg). The ball-to-powder weight ratio was 20:1. Milling was carried out in a Ar atmosphere for 2~25 h. Mechanically alloyed powders were hot-consolidated by hot isostatic pressing(HIP) at 1100°C for 1 h with the pressure of 110 MPa. For this, the milled powders were put into a steel tube (Φ24×70 mm), and then evacuated in vacuum for 400°C for 1 h, followed by sealing prior to HIPing. The HIPed samples were machined by wire-cutting for tensile test and characterization. The phases and lattice parameters after milling and HIPing were identified by XRD using a Ultima IV X-ray diffractometer. Microstructures were examined by an optical microscopy (OLYMPUS BX51M), a SEM (VEGA II LMU), and a TEM (JEOL 200). Tensile test was carried out for specimens with a gauge length of 4 mm (cross-section: 1.4×1.2 mm^2) at room temperature with a strain rate of 10-5/s.

TABLE 1

Compositions and preparation conditions of examined samples (wt.%)

Specimen No.	Compositions	Milling time
No. 1	Fe-1Y_2O_3	24h
No. 2	Fe-1Y_2O_3-0.5Ti	24h
No. 3	Fe-2Y_2O_3	24h
No. 4	Fe-2Y_2O_3-0.5Ti	24h

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3. Results and discussion

3.1. Mechanical alloying

The starting Fe powders with the size of ~200 μm were enlarged several times during the initial stage of mechanical alloying for 1 h where cold welding is a dominating event over fracturing. Particle fracturing started after milling for 2 h. The particle size of Fe powder was reduced and stabilized to 15 μm after mechanical alloying for 24 h where the steady-state of mechanical alloying was reached after the fracturing-dominated intermediate stage. The mean particle size after attrition milling for 24 h was ~15 μm with a quite homogeneous size distribution for all examined compositions. The result of X-ray diffraction (XRD) for the milled powders showed that elemental crystalline peaks of Y_2O_3 and Ti completely disappeared after mechanical alloying for 24 h (Fig. 1). It has been reported that the high-energy ball-milling of Fe or Ni powders with Y_2O_3 resulted in a complete dissolution of the latter phase by mechanical alloying, forming a metastable solid solution. Fig. 1 also shows the broadening of crystalline peaks of Fe, indicating a grain refinement and a partial formation of metastable phases. The grain size which were estimated from the Scherrer's equation was about 15 nm for the powders which were mechanically alloyed for 24 h.

3.2. Microstructures and properties after hot-consolidation

No visible change was observed by XRD after the subsequent HIPing of the mechanically alloyed powders at 1100°C for 1 h (Fig. 1) except a sharpening of diffracted peaks of Fe, indicating a crystallization at elevated temperatures. However, a closer examination by XRD showed that crystalline peaks of mechanically alloyed powders commonly shifted to lower angles after HIPing. The calculated lattice parameter of the mechanically alloyed Fe phase slightly increased from 2.884 ±0.003 to 2.865 ±0.004Å after HIPing, depending on compositions (Fig. 2). This is attributed to both the formation of a thermodynamically stable solid solution and the grain growth. The crystalline peaks of both Ti and Y_2O_3 are absent due to both the dissolution of Ti within the Fe-based matrix and a very fine size of Y_2O_3 with a small volume fraction.

Fig. 3 shows the optical micrographs of HIPed specimens. It can be clearly seen from the micrograph that, compared to the pristine Fe-Y_2O_3 (specimens No. 1,3), the Ti-added specimens (No. 2,4) contain much less particles (the dots-like phase). Furthermore, the volume fraction of particles is less for the Ti-added specimens. The particles were identified as Fe-Y-O or Fe-Y-Ti-O oxides. A closer examination by SEM also demonstrated that the

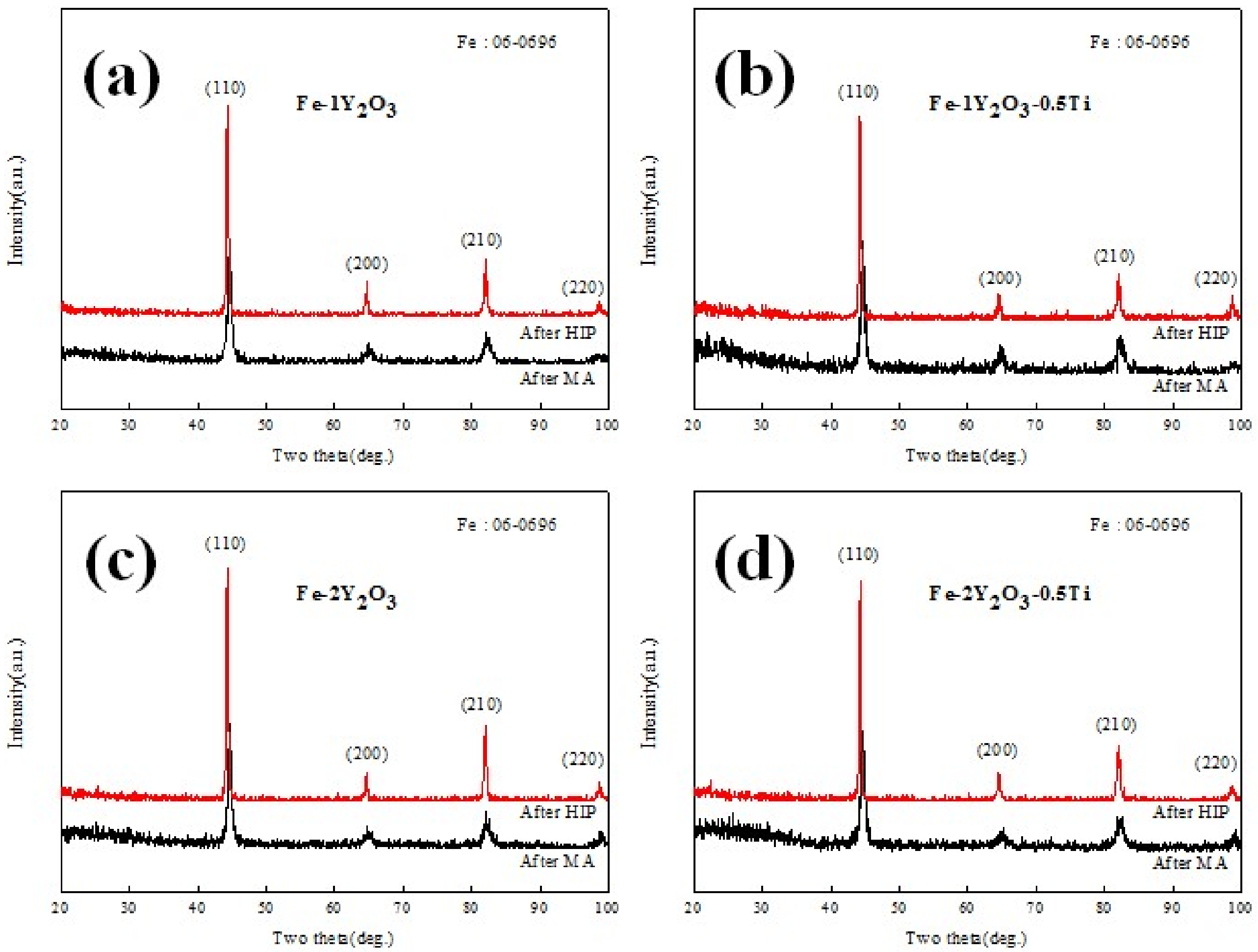

Fig. 1. XRD patterns after MA and HIPing for specimen No.1~4 : (a)~(d)

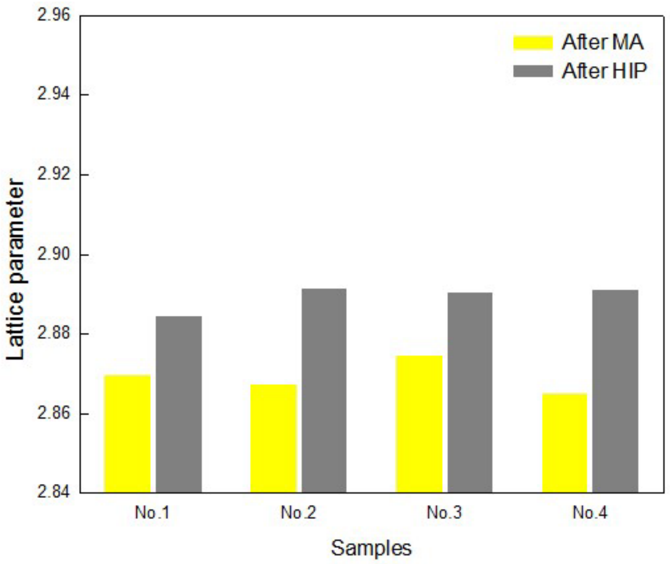

Fig. 2. Lattice parameter of Fe after MA and HIPing for specimen No.1~4 : (a)~(d)

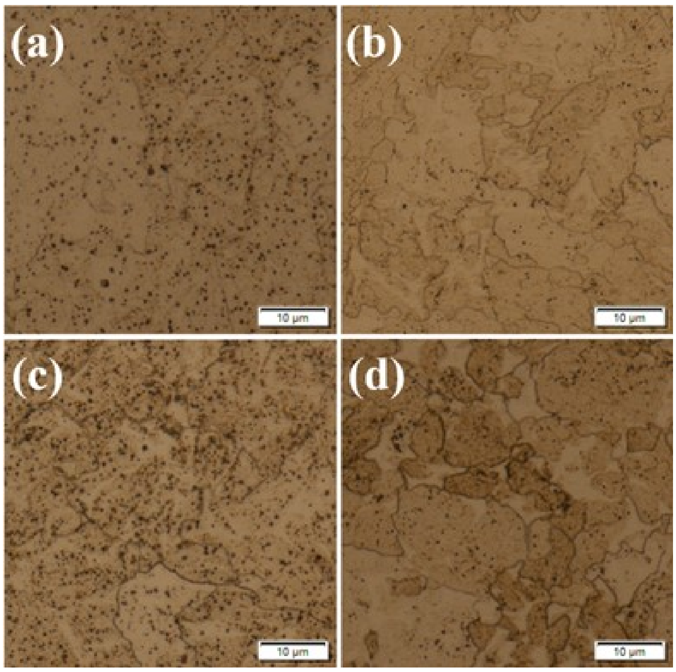

Fig. 3. OM micrographs after HIPing for specimen No.1~4 : (a)~(d)

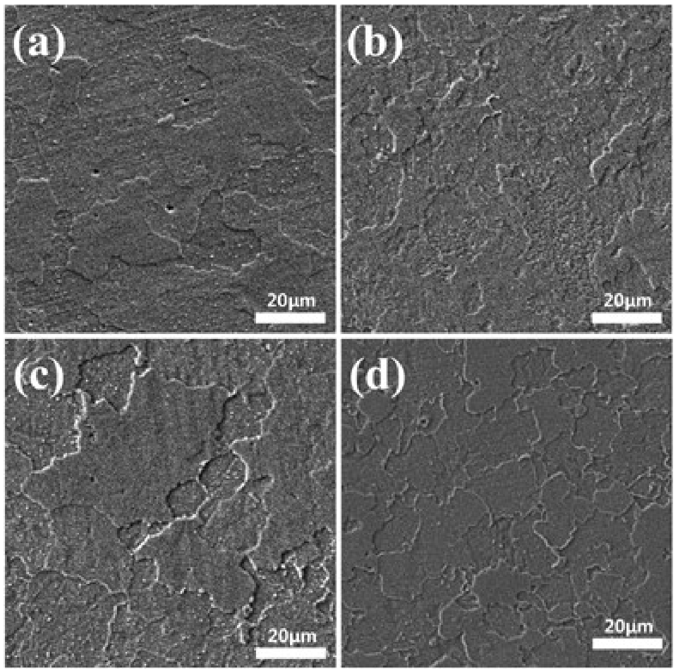

Fig. 4. SEM micrographs after HIPing for specimen No.1~4 : (a)~(d)

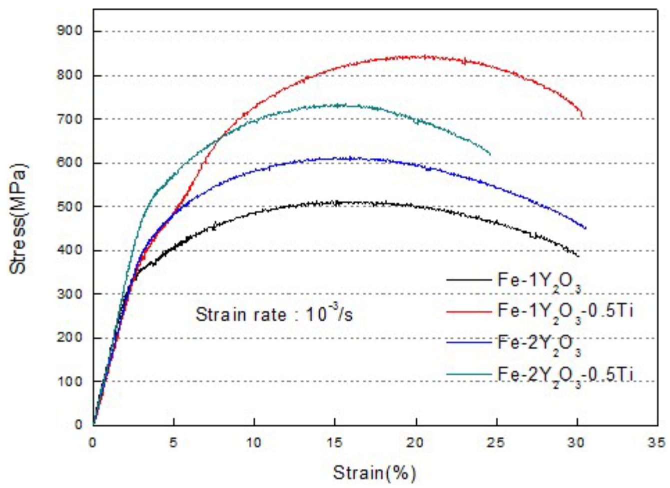

Fig. 5. Tensile curves for alloys after HIPing

Ti-added specimens (Fig. 4b,d) have more reduced grain size of the matrix phase with more uniform distribution than those of the pristine Fe-Y_2O_3 (Fig. 4a,c). Due to such fine and homogeneous microstructures, the Ti-added specimens exhibited better mechanical properties than Fe-Y_2O_3.The results of tensile test (Fig. 5) indicated that the addition of Ti to Fe-Y_2O_3 markedly improved both yield strength and tensile strength. In particular, the enhancement by the addition of Ti was apparently visible for Fe-1%Y_2O_3 without diminishing ductility (specimens No. 1,3). Hardness test also showed the same beneficial effect by Ti.

To clarify the cause of the observed property improvement by the addition of Ti, dispersed oxide particles were examined by a TEM combined with EDS mapping technique. The mean size of dispersing oxides was ~20 nm for Fe-1% Y_2O_3 (+0.5 Ti), whereas it was much larger for Fe-2% Y_2O_3 (+0.5 Ti). We have exaggeratedly increased the content of Y_2O_3 to 2% to confirm this effect. In this Fe-2% Y_2O_3 (+0.5 Ti) system, the oxides were over-grown (Figs. 6,7). As shown in Fig. 6, the size of oxides in HIPed Fe-2% Y_2O_3 are generally large, varying from several hundreds to tens nm. The EDS mapping result showed that oxides are ternary Fe-Y-O phase. For the Ti-added specimen, on the other hand, the number of oxides larger than 100 nm was less frequently observed, although some large ones were occasionally visible (Fig. 7). Furthermore, the oxides are quaternary Fe-Y-Ti-O. One interesting feature observed in the present work is the composition of the large particle (lower right in the micrograph) whose lower part is exempt from Y element. In summary, the addition of Ti to Fe-Y_2O_3 reduced the number of large oxide particles whose composition is slightly different from that of smaller ones.

4. Conclusions

In the present work, we examined the effect of additive Ti on the microstructure and mechanical properties of ODS Fe-Y_2O_3 alloys. The addition of Ti resulted in the reduction of grain size of matrix phase as well as dispersing oxides with uniform distribution. Furthermore, large particles have a complex composition exempted from Y. It might to be attributed to the interplay between formation and reduction of Fe-, Y-, and Ti-based oxides which can be estimated from the Ellingham diagram. A detailed explanation is under investigation. In this short work, we demonstrated that the Ti-added Fe-Y_2O_3 ODS alloys exhibited an improved mechanical strength, due to homogeneous and fine size of both matrix and oxide phases.

Acknowledgments

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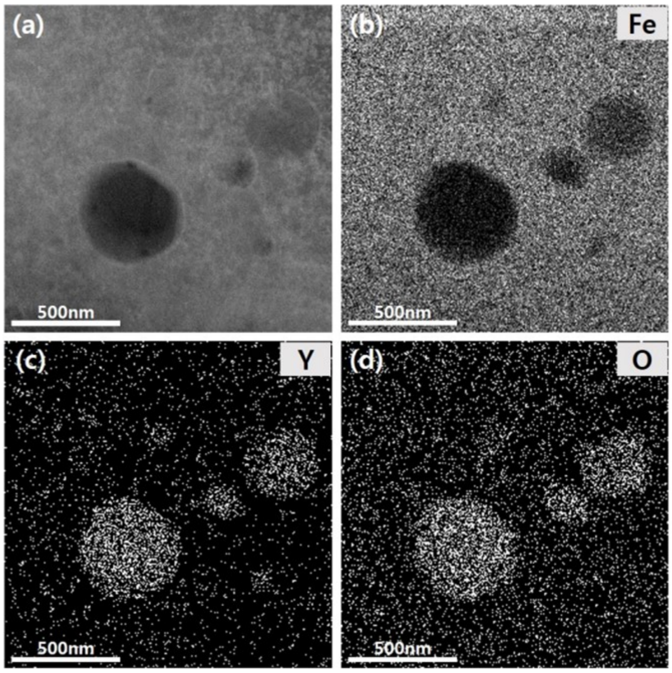

Fig. 6. TEM/EDS results of the Fe-2Y_2O_3 alloy (specimen No. 3) after HIPing

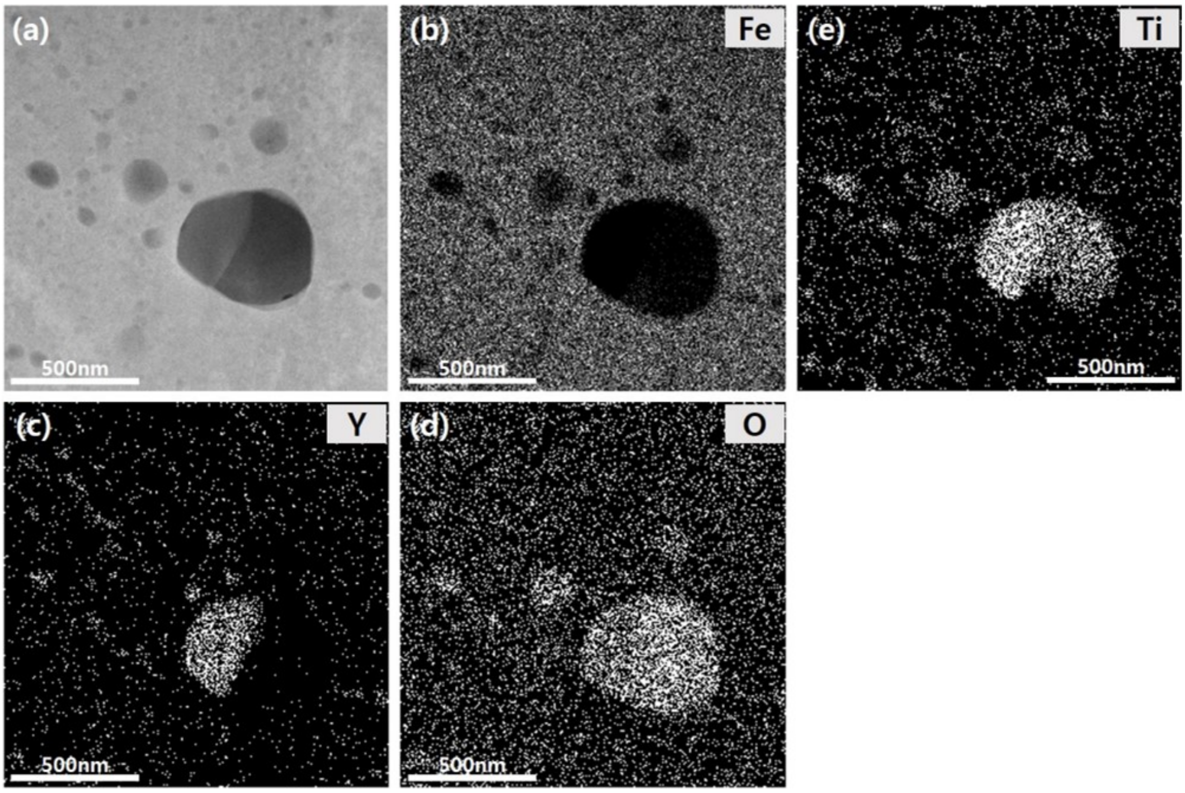

Fig. 7. TEM/EDS results of the Fe-2Y_2O_3-0.5Ti alloy (specimen No.4) after HIPing

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