

## Effects of $Y_2O_3$ , Ti and Forming Processes on ODS-Iron Based Alloy

Fang Yang<sup>1</sup>, Zhimeng Guo<sup>1\*</sup>, Leichen Guo<sup>2</sup> and Weiwei Yang<sup>1</sup>

<sup>1</sup>Institute of Advanced Materials and Technology, University of Science and Technology Beijing, Beijing 100083, China

<sup>2</sup>School of Engineering, Rensselaer Polytechnic Institute, Troy, NY 12180-3590, USA

### Abstract

Oxide dispersion strengthened (ODS) alloy ferric alloys have been applied to circumstances of high temperature and pressure. In this study, iron based alloys containing  $Y_2O_3$  and Ti have been made. The hydrothermal synthesis and spark plasma sintering (SPS) methods were utilized. The products were examined by Scanning Electron Microscope (SEM) and micro-electronic universal tester to study the effect of the addition of  $Y_2O_3$ , Ti and forming process.

**Keywords:** Mechanical properties; Oxide dispersion strengthened (ODS); Spark plasma sintering

### Introduction

Oxide Dispersion Strengthened (ODS) alloys have been the focus of materials research due to their thermal stability. Their development was promoted for long-life cladding tubes of the fast reactor fuel elements in high-temperature environments [1]. They are highly resistant to heat, corrosion and radiation.

It has been reported that the mechanical properties of ODS iron based alloys have been improved by nanometer oxide dispersion [2], whose strengthening effect is generated during the dispersion phase. The particle size decreases and the length of the dispersion phase increases when the particle space between dispersion phases decreases. The oxide content has a huge influence on the alloys. High content may cause a drop in the mechanical properties because it becomes more difficult to compress [3-5].

When the  $Y_2O_3$  and Ti with an average diameter of less than 10 nm were uniformly distributed in the matrix, the addition of  $Y_2O_3$  and Ti has been reported to increase the tensile strength and the creep properties of the alloy. However, the average size of the original particle is 30 nm [6]. As a result proper treatment should be applied on the oxide powder.

In the present work, Fe based ODS powders were synthesized with hydrothermal synthesis method. The alloys were sintered with Spark Plasma Sintering (SPS) methods. The effects of forming process,  $Y_2O_3$  and Ti content were investigated by Scanning Electron Microscope (SEM) and micro-electronic universal tester.

### Experimental Procedures

The starting materials used were  $Y_2O_3/Fe_2O_3$  composite powder prepared by hydrothermal synthesis method. The powder was ground in a dry vibratory mill for 20 hours and then mixed with 0.8wt% of Ti powder. During spark plasma sintering (SPS), the alloy was put under strictly controlled environment with a minimum of oxygen pollution. The vacuum degree was less than 8 Pa and the powder was sintered at 1050°C for 30 min (Figure 1).

The samples were investigated by Field Emission Scanning Electron Microscopy (FE-SEM) and a 500 MRA Rockwell hardness tester after polishing and etching by a 4 wt% nitric acid-alcohol mixture. Three measurements of the Rockwell hardness (HRB) were averaged. Tensile testing with a CMT 105 micro-electronic tester was performed on the samples. The fracture surface morphology was observed by SEM. The relative densities of samples take drainage method to measure.

### Results and Discussion

#### The effect of forming process

As is shown in table 1, the Fe-1 wt%  $Y_2O_3$  alloy by cold press sintering has a much lower relative density than the hot press sintered sample. But the SPS sample, which has a relative density of 95%, reigns supreme among the three. In conclusion, SPS is a sintering method that can be effectively applied to inhibiting grain diffusion and growth. The alloys manufactured by this method illustrate exceptional hardness and tensile strength.

#### The strengthening effect of $Y_2O_3$

After consolidation by SPS, the tensile strength and hardness of the samples in the ODS alloys increases as the  $Y_2O_3$  content increases in the range of 0-1.0 wt%. When the content of  $Y_2O_3$  was 2 wt%, the tensile strength and hardness decreased with an increase in  $Y_2O_3$  content. The best mechanical properties are obtained when the content of  $Y_2O_3$  is 1.0 wt%, as is shown in table 2.

The microstructures of Fe-0 wt%  $Y_2O_3$  and Fe-1 wt%  $Y_2O_3$  samples are shown in figure 2. When there is no  $Y_2O_3$  addition, the grain size of the iron matrix has no difficulty in increasing during the sintering insulation stage. As the content of  $Y_2O_3$  increases, the grain size reduces because the nanoparticles in the matrix hinder the movement of the

Sample	Relative density (%)	Hardness (HRB)	Tensile strength (MPa)	Elongation (%)
Fe-1.0 wt% $Y_2O_3$ (cold press sintering)	83.35	64.3	379.0	4.5
Fe-1.0 wt% $Y_2O_3$ (hot press sintering)	92.0	84.3	518	4
Fe-1.0 wt% $Y_2O_3$ (SPS)	95.0	91.2	536.7	2.0

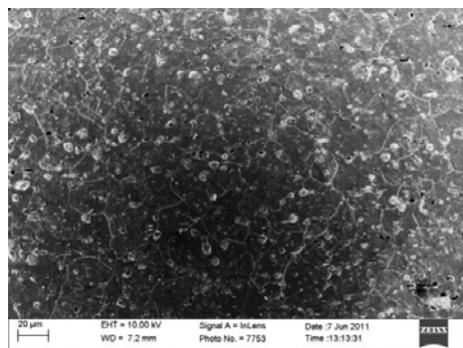
Table 1: Mechanical properties of the samples by different forming processes.

\*Corresponding author: Zhimeng Guo, Institute of Advanced Materials and Technology, University of Science and Technology Beijing, Beijing 100083, China, E-mail: [zmguo@ustb.edu.cn](mailto:zmguo@ustb.edu.cn)

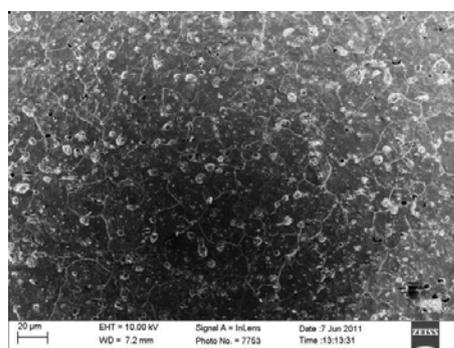
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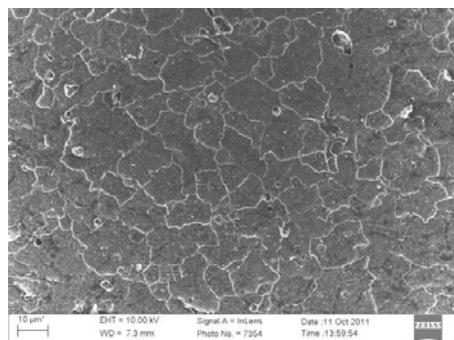
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a)



b)



c)

**Figure 1: SEM images of Fe-1 wt%  $Y_2O_3$  samples: (a) cold press sintering; (b) hot press sintering; (c) spark plasma sintering (SPS).** From Figure 1 (a) Magnified 1000 times, the grain size ranges from 10  $\mu m$  to 20  $\mu m$ . The structure isn't dense enough with a considerable amount of pores. The sample demonstrated in (b) is denser. However, some vacancy defects are still obvious. The average grain size is 10  $\mu m$ . The sample in (c), which was sintered by SPS, exhibits a configuration much denser compared to (a) and (b) in which there is no obvious pore. Its average grain size is 10  $\mu m$ .

grain boundaries in recovery and recrystallization. Therefore, by the addition of  $Y_2O_3$ , the microstructure of ODS-Fe materials presents unique characteristics such as small grain size [7]. It can be concluded that the tensile strength and hardness of the samples increases as the content of  $Y_2O_3$  increases. However, it should be noted that the content of  $Y_2O_3$  needs to be controlled within a range [8]. When the content of  $Y_2O_3$  was 1 wt%, the small particle space hampered the process of dislocation, resulting in optimum tensile strength and consequently the best dispersion strengthening.

### The effect of Ti element

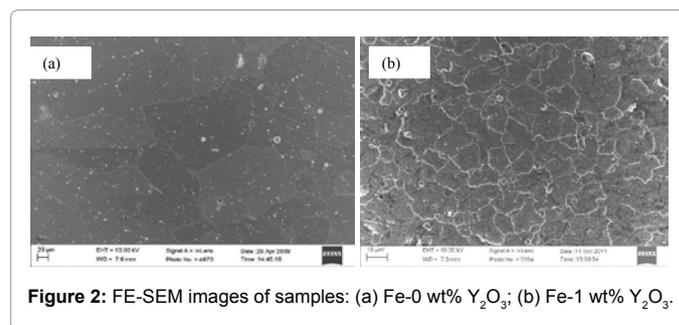
Table 3 gives the mechanical properties of Fe-1 wt%  $Y_2O_3$  and Fe-1 wt%  $Y_2O_3$ -0.8 wt% Ti samples prepared by SPS. It is clear that the tensile strength and hardness of the samples were further improved with the addition of Ti element.

The FE-SEM images of Fe-1 wt%  $Y_2O_3$  and Fe-1 wt%  $Y_2O_3$ -0.8 wt% Ti prepared by SPS are shown in figure 3, respectively. It can be seen that the presence of Ti led to a distinct decrease in grain size and has little effect on the size of the dispersed particles.

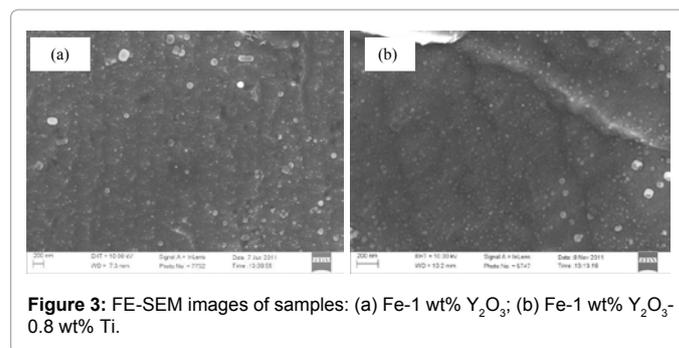
Ti refined the alloy in the Oxide Dispersion Strengthening (ODS) phase by dissolving  $Y_2O_3$  and precipitating it during the mechanical alloying process. The presence of Ti led to the formation of a new oxide  $Y_2Ti_2O_7$ , resulting in a more refined dispersion of the oxide [3,9].

### Conclusion

- (1) SPS is an effective sintering method to inhibit grain diffusion and growth and sample has a relative density of 95% by SPS.
- (2) With an increase of their  $Y_2O_3$  content, the mechanical



**Figure 2: FE-SEM images of samples: (a) Fe-0 wt%  $Y_2O_3$ ; (b) Fe-1 wt%  $Y_2O_3$ .**



**Figure 3: FE-SEM images of samples: (a) Fe-1 wt%  $Y_2O_3$ ; (b) Fe-1 wt%  $Y_2O_3$ -0.8 wt% Ti.**

Sample	Relative density (%)	Hardness (HRB)	Tensile strength (MPa)	Elongation (%)
Fe-0 wt% $Y_2O_3$	96.5	53.0	327.0	5.0
Fe-0.5 wt% $Y_2O_3$	95.3	65.7	425.0	4.8
Fe-1.0 wt% $Y_2O_3$	95.0	91.2	536.7	2.0
Fe-2.0 wt% $Y_2O_3$	94.6	72.0	402.7	2.5

**Table 2: Mechanical properties of the samples with different  $Y_2O_3$  content.**

Sample	Relative density (%)	Hardness	Tensile strength (MPa)	Elongation (%)
Fe-1 wt% $Y_2O_3$	95	91.2 (HRB)	536.7	2
Fe-1 wt% $Y_2O_3$ -0.8 wt% Ti	94	38 (HRC)	713	0

**Table 3: Mechanical properties of Fe-1 wt%  $Y_2O_3$  and Fe-1 wt%  $Y_2O_3$ -0.8 wt% Ti prepared by SPS.**

properties of samples of iron-based alloys increased in the content range of 0-1 wt% and decreased when it was over 1 wt%. The best mechanical properties were obtained with the addition of 1 wt%  $Y_2O_3$ .

(3) With the addition of Ti element, the tensile strength and hardness of the samples were improved.

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