Hindawi Publishing Corporation
Journal of Nanomaterials
Volume 2008, Article ID 459093, 4 pages
doi:10.1155/2008/459093

Research Article

Effect of Rare Earth Y on Properties of Nanosized 90W-7Ni-3Fe Composite Powder Fabricated by Spray Drying-Hydrogen Reduction

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Received 6 July 2007; Accepted 11 January 2008

Recommended by Jun Lou

(W,Ni,Fe) composite oxide powder synthesized by spray drying was reduced at 700°C for 90 minutes in H_2 atmosphere. The effect of rare earth Y on H_2 reduction of (W,Ni,Fe) composite oxide powder was studied. Phase composition, crystalline size, and particle morphology of the reduced powder have been measured by X-ray diffraction and scanning electron microscope (SEM). Fsss particle size and special surface area of the reduced powder were also measured and analyzed. The result showed that new phase $Y(Ni_{0.75}W_{0.25})O_3$ appeared in the reduced powder and particle morphology was nearly spherical or polyhedron by Y additions. The higher the rare earth element content was, the bigger the influencing on particle morphology was. When the rare earth Y content was under 0.8%, with the increase of the rare earth element content, d_{BET}, Fsss, and crystal sizes of the reduced powder decreased greatly.

1. INTRODUCTION

Tungsten-based heavy alloys, which are used as kinetic energy penetrators, counter weights, radiation shields, and electrical contacts due to their high density, strength, and ductility, are two-phase composites produced by liquid phase sintering mixed elemental tungsten, nickel, and iron powders. Typical compositions have 90 wt% tungsten with the balance nickel and iron, usually in the ratio of 7 : 3 [1].

Nanostructured tungsten-based alloys possess very high properties, which put high demands on the manufacture process for both powders and bulk alloys. High-energy ball milling, which is a well-known process for preparing amorphous alloyed powders, has been considered as a powderful technique due to relative inexpensive equipment [2]. However, it takes tens of hours to fabricate nanosized W-Ni-Fe composite powder for high-energy milling technique [3], so the milling efficiency is very low. Meanwhile, high-energy milling could bring inclusions. Spray drying process is a useful technique for synthesizing numerous nanostructured materials due to its simplicity and various elements homogeneous dispersibility [4, 5]. Previous studies have discussed preparation and reduction mechanisms of (W,Ni,Fe) composite oxide powder by spray drying [6–8]. In this paper, effects on the reduced powder properties with Y additions were studied by previous reduced conditions optimized at 700°C for 90 minutes.

2. EXPERIMENTAL

The raw materials in this experiment included the $(NH_4)_6H_2W_{12}O_{40} \cdot xH_2O$, $Ni(NO_3)_2 \cdot 6H_2O$, $Fe(NO_3)_3 \cdot 9H_2O$, $Y(NO_3)_2 \cdot 6H_2O$, and surfactant PEG-1000. First, the $(NH_4)_6H_2W_{12}O_{40} \cdot xH_2O$, $Ni(NO_3)_2 \cdot 6H_2O$, and $Fe(NO_3)_3 \cdot 9H_2O$ were added in distilled water by a composition ratio of 90 wt%W-7 wt%Ni-3 wt%Fe and produced solution with 0.2, 0.4, 0.6, 0.8 wt% Y additions by $Y(NO_3)_2 \cdot 6H_2O$ (theoretical content calculation), respectively. Secondly, the solution was changed into solution colloid by adjusting pH value, and $0.5\,g \cdot L^{-1}$ PEG-1000 was added. Finally, the (W,Ni,Fe) precursor oxide powder was fabricated by drying spraying of solution colloid, then the nanosized 90W-7Ni-3Fe composite powder including different Y additions was fabricated by reduction of the (W,Ni,Fe) precursor oxide powder in the hydrogen-atmosphere at 700°C for 90 minutes.

Special surface areas of the reduced powder were measured by the nitrogen adsorption method. The Fsss sizes of the reduced powder were measured by the Fisher testing method. The phase compositions and crystal sizes of the reduced powder were tested and analyzed with Rigaku D/max2550VB$^+$18 Kw X-ray diffraction made in Japan. The reduced powders were dispersed by ultrasonic wave for 30 minutes, whose morphology images were observed with the JSM-5600LV type scanning electron microscope (SEM) made by JEOL cop.

3. RESULTS AND DISCUSSION

3.1. Effect of rare earth Y on phase composition of 90W-7Ni-3Fe composite powder

Figure 1 showed curves between the Y additions and phase compositions of the reduced composite powder at 700°C for 90 minutes. It was seen from Figure 1 that the phase compositions of the reduced powder were W and γ-(Ni,Fe) without the rare earth Y addition, new phase $Y(Ni_{0.75}W_{0.25})O_3$ was appeared when adding 0.4 wt% Y. With the increase of Y additions from 0.4 wt% to 0.8 wt%, intensity of new phase $Y(Ni_{0.75}W_{0.25})O_3$ raised in X-ray diffraction graph.

3.2. Effect of rare earth Y on properties of 90W-7Ni- 3Fe composite powder

Figure 2 showed curves between the d_{BET} particle sizes, Fsss particle sizes, and crystal sizes of the reduced powder and the Y additions. The crystal sizes of the reduced powder were decreased with increase of the Y additions from Figure 2(a). High Y addition has better effect on inhibition for crystal size than low Y addition. The d_{BET} and Fsss particle sizes of the reduced powder with some Y additions were much smaller than those of the reduced powder without Y addition from Figures 2(b) and 2(c). Even if 0.2 wt% Y was added, the d_{BET} and Fsss particle sizes of the reduced powder declined obviously. With the increase of water vapor produced during reduction process, tungsten oxide and water vapor synthesized volatiling $WO_x \cdot H_2O$, that is $WO_2(OH)_2$, which deposited on the surface of metal tungsten powder by vapor transference, and led to growth of particles [9]. The process sequenced as

$$\begin{array}{ccccccc} WO_3(\text{solid}) & \longrightarrow & WO_{2.9}(\text{solid}) & \longrightarrow & WO_2(\text{solid}) & \longrightarrow & W(\text{solid}) \\ & & & & \downarrow & & \uparrow \\ & & & & WO_2(OH)_2(\text{gas}) & & \end{array}$$

When rare earth Y was added, new phase $Y(Ni_{0.75}W_{0.25})O_3$ formed and adsorbed on the surface of tungsten particles. Meanwhile, new phase $Y(Ni_{0.75}W_{0.25})O_3$ can effectively prevent $WO_2(OH)_2$ from producing and decreasing vapor transference, which inhibited effectively W particles from growing up.

3.3. Effect of rare earth Y on morphology images of 90W-7Ni-3Fe composite powder

SEM morphology images of the reduced composite powder were listed in Figure 3. From Figure 3, the reduced W-Ni-Fe

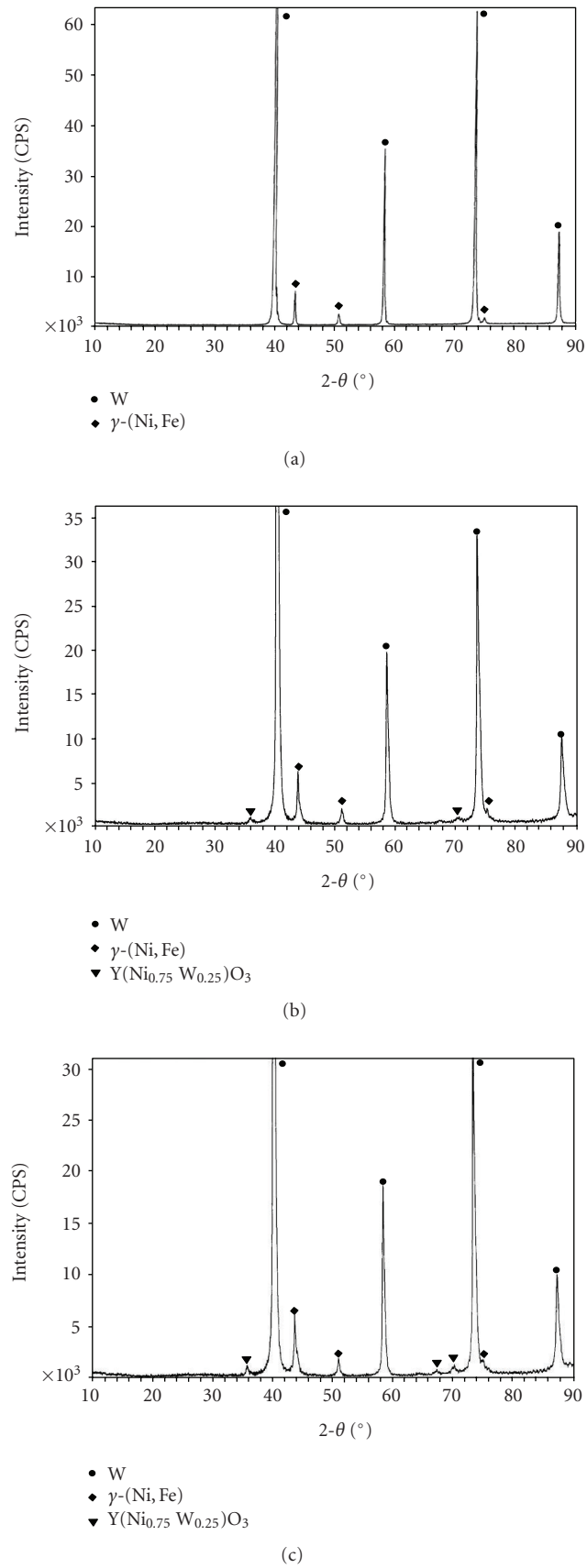
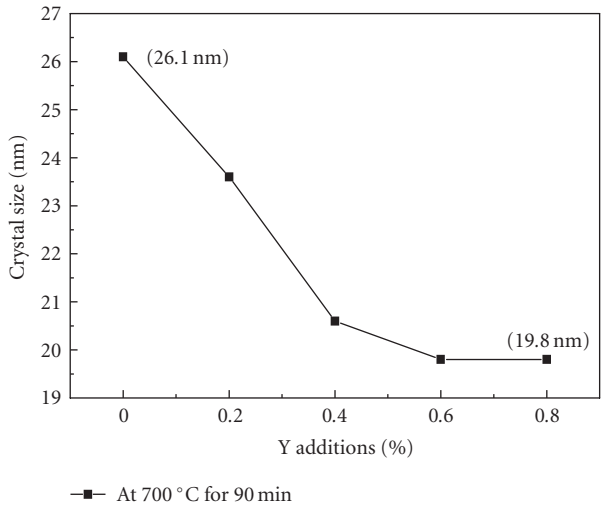
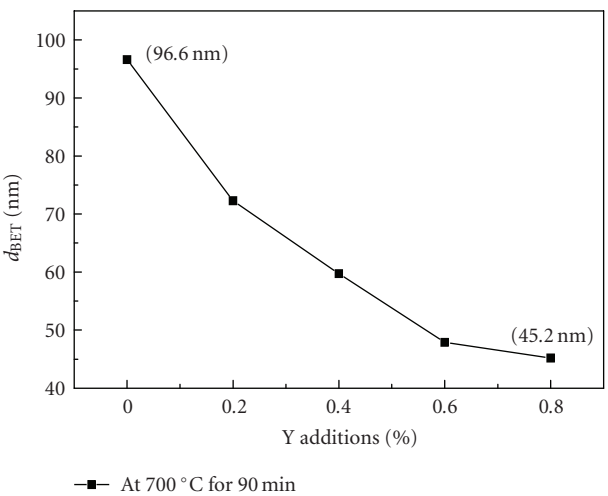


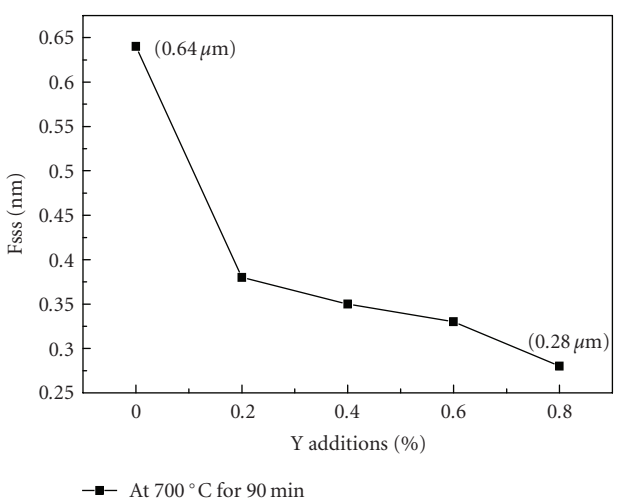
Figure 1: X-ray diffraction images of W-Ni-Fe composite powder reduced: (a) no Y addition; (b) 0.4 wt% Y; (c) 0.8 wt% Y.



(a)

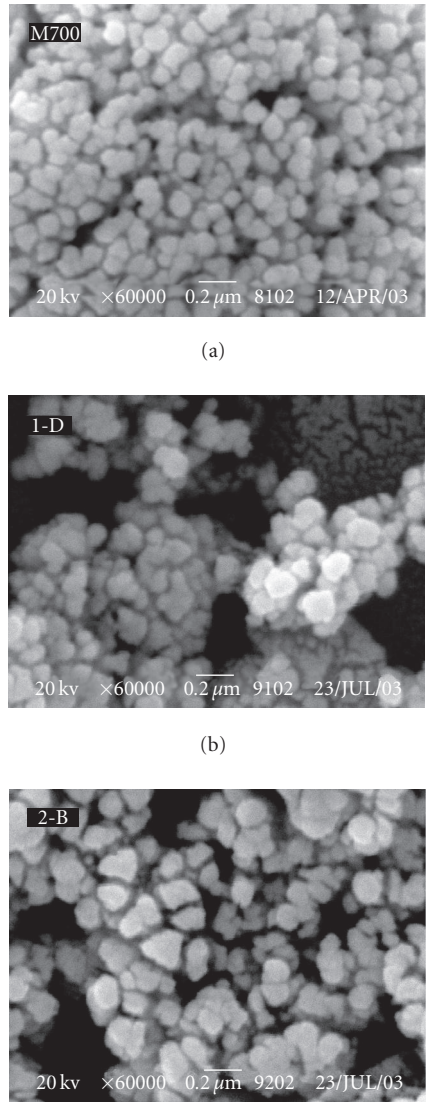


(b)



(c)

Figure 2: Properties curves of the reduced powder for different Y additions: (a) crystal sizes; (b) d_{BET}; (c) Fsss.



(a)

(b)

(c)

Figure 3: Morphology images of the reduced powder for different Y additions (SEM): (a) no Y addition; (b) 0.4 wt% Y; (c) 0.8 wt.% Y.

composite powder by rare earth Y additions was more homogeneous dispersibility than that of no Y addition. Meanwhile, with the increase of Y addition, effect of rare earth on dispersibility of the reduced powder enlarged obviously. It was also seen clearly from Figure 3 that, powder particle morphology with rare earth Y addition has changed, the particle morphology was spherical without rare earth Y, but the powder particle morphology was nearly spherical with 0.4 wt% Y and polyhedral with 0.8 wt% Y. The higher the Y additions were, the greater the effect on powder particle morphology was. Because energy state of some crystal interface has changed when some rare earth Y were added, which forced crystal quickly growth up at some directions and inhibited other directions from growthing up. With the increase of rare earth Y, tendency of preferential orientation growthing was more obvious [10, 11].

4. CONCLUSION

(1) The phase compositions of the reduced powder were W and γ-(Ni,Fe) without the rare earth Y addition, but new phase $Y(Ni_{0.75}W_{0.25})O_3$ was appeared by adding 0.4 wt% Y. With the increase of Y additions from 0.4 wt% to 0.8 wt%, intensity of new phase $Y(Ni_{0.75}W_{0.25})O_3$ raised in X-ray diffraction graph.

(2) With increase of Y additions in the range of 0.8 wt%, the crystal, d_{BET}, and Fsss sizes of the reduced W-Ni-Fe composite powder were decreased from 26.1 nm, 96.6 nm, 0.64 μm to 19.8 nm, 45.2 nm, 0.28 μm, respectively.

(3) Rare earth Y possessed great influence on powder particle morphology. The particle morphology is spherical without rare earth Y, but the particle morphology is nearly spherical by adding 0.4 wt% Y or polyhedron with 0.8 wt% Y addition. The higher rare earth content is, the bigger influencing on particle morphology is.

ACKNOWLEDGMENTS

The authors thank the National Natural Science Foundation of China (Grant no. 50774098) and the Creative research group of National Natural Science Foundation of China (Grant no. 50721003) for the financial support.

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