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Characteristics of Fe-Ni Nanopowders Prepared by Electrical Explosion of Wire in Water and Ethanol

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In this work, we prepared Fe-Ni alloy nanopowders by wire electrical explosion in deionized water and ethanol. Particles size and morphology of the as-synthesized nanoparticles prepared in water and ethanol were observed by transmission electron microscopy. In both cases, the as-synthesized nanoparticles were in nearly spherical shape and their size distribution was broad. The particles prepared in the water were in core-shell structure due to the oxidation of Fe element. X-ray diffraction was used to analyze the phase of the nanopowders. It showed that the nanopowders prepared in water had γ-Fe-Ni solid solution and FeO phase. The samples obtained in ethanol were in two phases of Fe-Ni solid solution, γ-Fe-Ni and α-Fe-Ni. Bulk samples were made from the as-synthesized nanopowders by spark plasma sintering at 1000 °C for 10 min. Structure of the bulk sample was observed by scanning electron microscope. Magnetic properties of the as-synthesized nanopowders and the bulk samples were investigated by vibrating sample magnetometer. The hysteresis loop of the as-synthesized nanopowders and the sintered bulk samples revealed a ferromagnetic characteristic.

Keywords : magnetic materials, magnetic properties, nanopowder, electrical explosion of wire (EEW)

1. Introduction

Electrical explosion of wire (EEW) is a very simple and low-cost method to produce various kinds of nanosized powders [1-3]. The explosion of wire in gas atmosphere such as air, Ar, N_2, etc. has been investigated to synthesize metallic, oxide, nitride, carbide materials and so on [4-6]. Properties of powders obtained by EEW depend on many conditions of electrical explosion process, which include wire properties such as wire dimension (diameter and length) and wire material, capacitor bank voltage, characteristics of the electrical circuit, and properties of the ambient medium. In general, it was shown that the capacitor bank voltage and wire dimension could affect on the particles size and size distribution. The ambient environment of the explosion process affects not only on the size, shape and size distribution but also on the chemical reactions between wire materials and active medium. Therefore, it is one of the most important parameters in electrical explosion process, which affects on the properties of produced materials. Ambient environment of EEW process in liquid showed a significant effect on the properties of synthesized materials. Influences of the medium on preparation of the copper nanofluids by wire explosion process were presented in the Ref. [7]. The research revealed that the exploding medium has a remarkable effect on properties of product. The pure copper nanopowder was prepared by using an ethylene glycol as medium of explosion process, while mixed copper and copper oxide were obtained using the deionized water and cetyl trimethylammonium bromide (CTAB) solution. Properties of Au colloid depended on the explosion medium in the liquid were reported in the Ref. [8]. With noble materials, no reaction can happen between them and medium but quality of product depends on the enhancement plasma formation and surface modification of nanoparticles.

Iron-nickel alloys are very important soft materials, which have been widely used such as recording heads, transformers or magnetic shielding materials. Their properties much depends on the synthetic method and processing. The objectives of the present work were to investigate the influence of ambient medium on properties of Fe-36Ni alloy (36 wt.% Ni), one of attractive materials for maximum resistivity and very low coefficient of expansion, known as Invar. Deionized water and ethanol were used as exploding environment for investi-

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gation. The structural, magnetic properties of the synthesized nanopowders and sintered bulk sample prepared by spark plasma sintering (SPS) process were also characterized.

2. Experimental

Fe-36Ni alloy wire with diameter of 0.2 mm was exploded in deionized water and ethanol. A stainless steel beaker filled with 500 mL liquid was used as the explosion chamber. A schematic diagram and detail mechanism of the EEW process in liquid has already been reported in the Ref. [9]. Briefly, a capacitor was charged to 3 kV and then released a high density current through the Fe-Ni alloy wire with 27 mm in length. The current density through the wire in EEW can be more than 10^{10} A/m^2 [10]. When a high-density current pulse passes via the wire, the wire is heated due to Joule heating. As a result, the material boils up at a burst, a bright light flashes, and a mixture of superheated vapor and boiling droplets of the exploding wire material forms and a shockwave scatter to the ambient atmosphere. The nanopowders are created when the vapor was cooled down by liquid.

The nanopowders were collected from the dispersion by centrifuging, dried under vacuum for 5 h. These dried nanopowders were used for phase analysis, magnetic measurement and making bulk sample. X-ray diffraction (XRD) with Cu-Kα incident radiation was used to analyze the phase of nanopowder. The shape and size of the as-synthesized nanoparticles were determined by transmission electron microscopy (TEM). Bulk samples were made from the as-synthesized nanopowders by SPS at 1000 °C for 10 min. Surface of the bulk samples was observed by scanning electron microscope (SEM). Vibrating sample magnetometer (VSM) was used to analyze the magnetic properties of the nanopowders and the sintered samples at room temperature.

3. Results and Discussion

3.1. Explosion in deionized water

Fig. 1 shows the XRD pattern of nanopowders prepared by EEW in the deionized water. Two phases were indexed in this pattern: γ-Fe-Ni phase and FeO phase. Main phases of the nanopowder were γ-Fe-Ni solid solution and FeO. The FeO phase was found in the XRD patterns, resulting from an oxidation of iron element. The FeO formed because Fe element reacted with oxygen diluting in water when the Fe vapor condensed. It was noted that there was no evidence for the formation of NiO phase in the XRD result. It means that Ni element did not oxidize

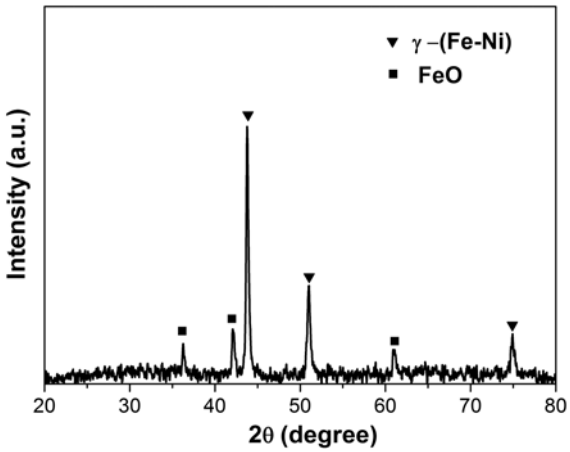

Fig. 1. XRD pattern of Fe-Ni nanopowder prepared by EEW in water.

when condensing from vapor state. It could be possible that because the Fe content in this alloy is around 64 wt.%, which is much higher than Ni content. In addition, Fe element is more active than Ni. Therefore, the Fe can react with oxygen and forms the oxide phase but the Ni element does not react with it.

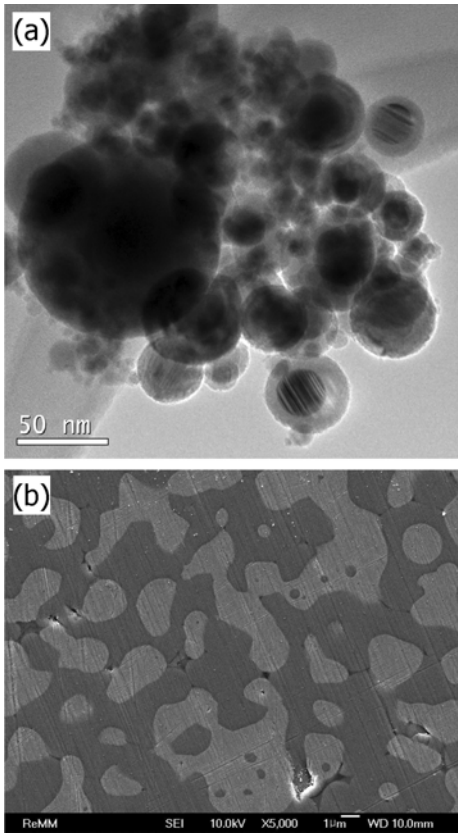

Fig. 2. (a) TEM photograph of Fe-Ni nanopowder prepared by EEW in water and (b) SEM photograph of sintered sample.

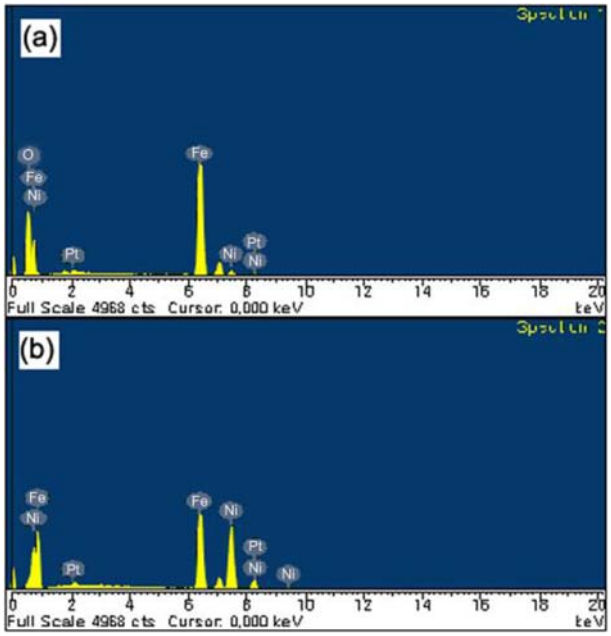

Fig. 3. (Color online) EDS results of sintered sample: (a) dark phase and (b) light-white phase.

Microstructure of the nanoparticles prepared in the water was shown in Fig. 2a. From this image, we can see that the nanoparticles were nearly spherical in shape and had a core-shell structure. We could determine that the core phase was γ-Fe-Ni and the shell phase was FeO, which resulted in the oxidation of Fe. The histograms of size distribution extracted from TEM image presents Gaussian approximation of size. The particle sizes are in the range of 15-65 nm with a mean particles size of 33 nm.

Bulk sample was made from the as-synthesized nanopowders by SPS at 1000 ºC for 10 min. The microstructure of bulk sample surface was observed by SEM and shown in Fig. 2b. From the graph, we can see that the bulk sample was in dense with few pores. There were two distinguishable areas in the graph: the dark phase mixed with the light white phase. Based on the EDS data, the dark phase is formed by the oxide iron phase, which was indicated in previous investigation of XRD and TEM results and the light white phase is Fe-Ni alloy (seen in Fig. 3).

Magnetic properties of the as-synthesized nanopowder and bulk sample were investigated at room temperature using a VSM with an applied field 10,000 Oe ≤ H ≤ 10,000 Oe. Fig. 4 shows their magnetic hysteresis loops, which are the typical loops of soft magnet. The values of the saturation magnetization and coercivity are listed in Table 1. As can be seen in the table that the sintered sample had smaller coercivity and higher saturation magnetization in comparison with the as-synthesized powder. The lower coercivity of the sintered sample than the powder could be explained by the particle-particle interaction. In the non-compaction state, the nanopowders are loose so they are difficult to interact each other. The compaction and sintering improve contact between particles and fix them up which causes the lowering coercivity and higher saturation magnetization.

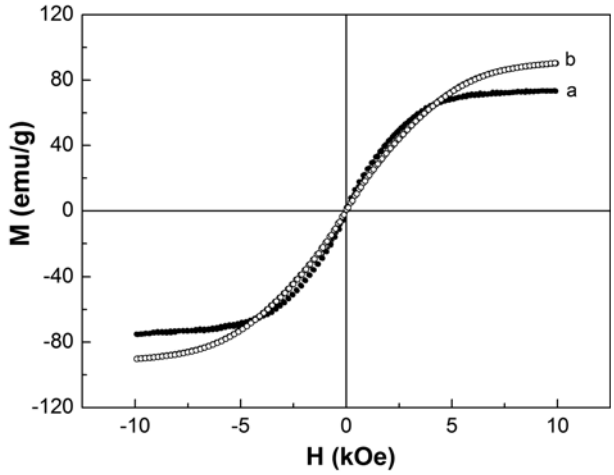

Fig. 4. Hysteresis loops of (a) as-synthesized nanopowder prepared by EEW in water and (b) sintered sample.

Table 1. Coercivity and saturation magnetization of nanopowder prepared by EEW in water.

Sample	Hc (Oe)	Ms (emu/g)
Nanopowder	50.22	73.30
SPS bulk sample	14.26	90.59

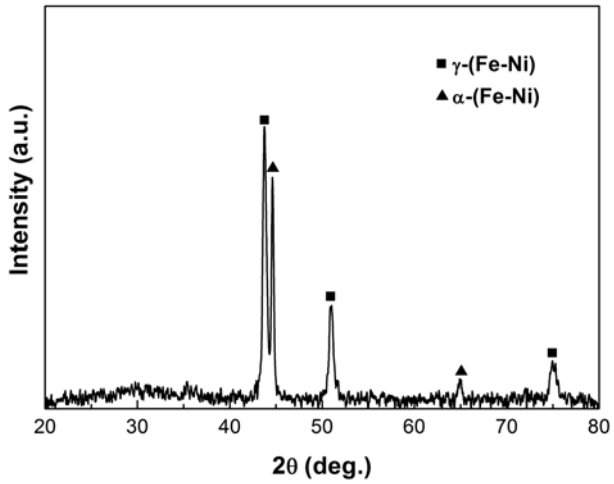

Fig. 5. XRD pattern of Fe-Ni nanopowder prepared by EEW in ethanol.

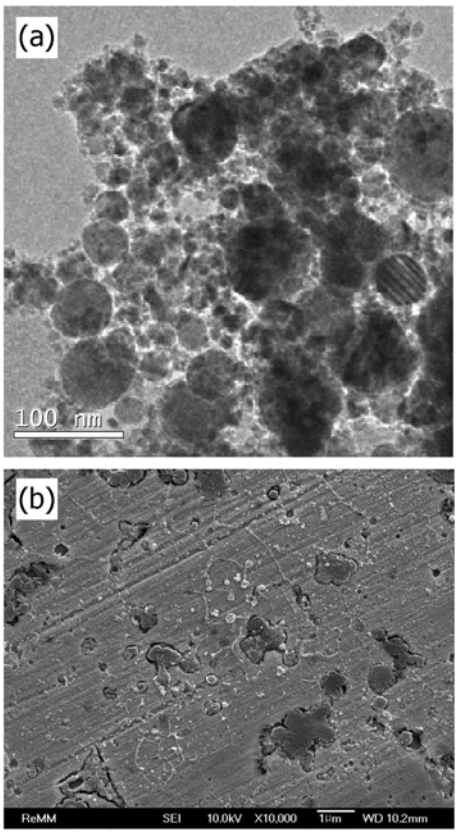

Fig. 6. (a) TEM photograph of Fe-Ni nanopowder prepared by EEW in ethanol and (b) SEM photograph of sintered sample.

3.2. Fe-Ni nanopowder prepared in ethanol

Fig. 5 shows the XRD pattern of the nanopowder obtained by EEW in the ethanol. It can be seen from the pattern that two phases of Fe-Ni alloy presented in the pattern. The γ-Fe-Ni phase and α-Fe-Ni phase co-existed in the material. Unlike case of preparation in water, no oxide phase was detected in the sample prepared in the ethanol.

Fig. 6a and b illustrated the TEM graph of the nanoparticles obtained by EEW in the ethanol and SEM surface image of sintered body prepared by SPS of the nanopowders. From this graph, nearly spherical particles were observed and no core-shell structure presented in this sample because no oxide phases was detected in XRD result. In XRD analysis, we knew that nanopowder formed in two phases of Fe-Ni solid solution but we cannot distinguish in TEM photograph. The average size of particles estimated from TEM image is ~25 nm, smaller than that of particles prepared in water. From XRD and TEM results, we can reveal that the ambient liquid is much affects on the microstructure of product. The liquid is not simple as a medium for cooling during

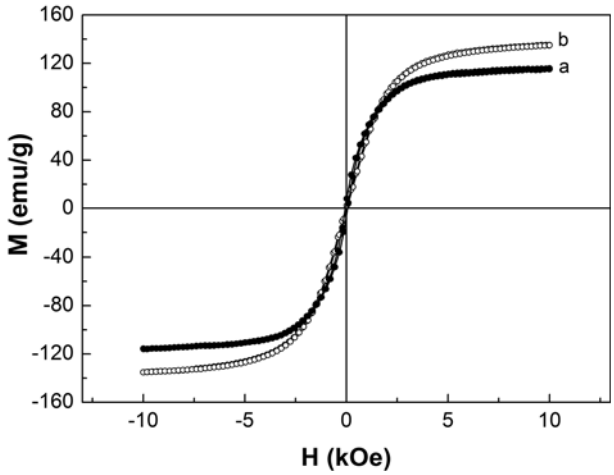

Fig. 7. Hysteresis loops of (a) as-synthesized nanopowder prepared by EEW in ethanol and (b) sintered sample.

vapor state of materials. More researches need to be done to explain why the ambient liquid medium can affect on the properties of product. Many previous studies only attributed on the phenomenon of influences but no papers gave mechanism of this process. Uncertainty in time-dependent parameters, such as specific resistance, thermal conductivity, density, and temperature of the wire and liquid during phase transitions, makes it difficult to provide a self-consistent description of process [11].

Fig. 7 presents the hysteresis loops of the nanopowder prepared by EEW in ethanol and its bulk sample obtained by SPS. The shape of loops is typical loop of ferromagnetic materials. It is similar to the case of preparation in the water, the bulk sample have higher value of saturation magnetization and smaller value of coercivity (Table 2). The coercivity value decreased from 59.55 Oe to 27.53 Oe and the saturation magnetization increased from 116.17 emu/g to 137.15 emu/g. Two reasons can attribute to this result. First, particles in sintered sample are closer than those in powdered sample. The better contact between nanoparticles in sintered sample can cause the better magnetic properties. Second, the change of alloy phase also attributes to enhance its magnetic characteristics. Nanopowder has two phases of γ-Fe-Ni and α-Fe-Ni but the sintered sample was only in γ-Fe-Ni phase without presence of α-Fe-Ni phase. When nanopowder was sin-

Table 2. Coercivity and saturation magnetization of nanopowder prepared by EEW in ethanol.

Sample	Hc (Oe)	Ms (emu/g)
As-synthesized powder	59.55	116.17
Sintered sample	27.53	137.11

tered at high temperature, the α-Fe-Ni phase transformed to γ-Fe-Ni phase.

4. Conclusion

The Fe-Ni alloy nanopowders were prepared by EEW in the deionized water and ethanol. This research showed the properties of Fe-Ni alloy much depends on the ambient medium of EEW process. Microstructural and magnetic properties of nanopowder changed when water and ethanol were used as explosion environment. Preparation in water, besides the main phase was γ-Fe-Ni, the FeO phase existed due to the oxidation of Fe element. Preparation in ethanol, no oxide phases presented in the samples. Two phases of γ-Fe-Ni and α-Fe-Ni coexisted. In both cases, the sintered samples decreased the value of coercivity and increased the saturation magnetization.

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