Asian Journal of Chemistry;   Vol. 24, No. 9 (2012), 3945-3947

ASIAN JOURNAL OF CHEMISTRY

www.asianjournalofchemistry.co.in

Preparation and Magnetic Properties of Ultra-Fine Grained γ-Ni-22Fe/Al_2O_3 Composite†

YIN LIU*, JUN TAO, LINYI ZHENG, CHUYANG XU, FANFEI MIN and MINGXU ZHANG

School of Materials Science and Engineering, Anhui University of Science and Technology, Huainan 232001, Anhui Province, P.R. China

*Corresponding author: E-mail: yinliu@aust.edu.cn

AJC-11279

Ultra-fine grained γ-Ni-22Fe alloy dispersed Al_2O_3 matrix composites were prepared by a mechano-chemical process and subsequent combining with hot-pressing sintering of nanometer Ni-22Fe alloy/Al_2O_3 mixed powder. The microstructure was investigated by using XRD, TEM as well as AFM. Magnetic properties were measured with using vibrating sample magnetometer (VSM) at room temperature. The results indicated that the Ni-22Fe/Al_2O_3 composite possessed high densities (relative density *ca.* 98 %) and sub-micrometer-sized matrix dispersed with γ-Ni-22Fe particles of sizes below *ca.* 500 nm. The composite containing the ferromagnetic Fe-22Ni alloy particles exhibited ferromagnetic properties. The specific saturation magnetization and coercivity of Ni-22Fe/Al_2O_3 composite are 78.8 emu g^{-1} of Ni-22Fe, 105Oe, respectively, which points functionally structural composite material.

Key Words: Ni-22Fe alloy, Ceramic-matrix composites, Preparation, Magnetic properties.

INTRODUCTION

Alumina, as an excellent construction ceramics, possesses the attractive properties of good hardness, chemical stability, refractory character and low density. However, it is brittle at low temperature for the lack of fundamental dislocation mobility and of insufficient slip systems. From the standpoint of material design, it is undoubtedly a good method to develop ceramic-matrix composite with nanosized or sub-micrometer-sized metal or alloy dispersion because of their high strength and multi-functional properties[1-3]. For example, Al_2O_3/Ni nanocomposites exhibited not only high fracture strength of 1 GPa but also peculiar magnetic properties due to the dispersion of nanosized Ni magnetic particles[4]. Among the metal or alloy, the nickel-iron (Ni-Fe) alloys in the permalloy rang, from about 10-65 wt % iron, are probably important soft magnetic alloys[5], which have been widely used in industry, such as recording heads, transformers or magnetic shielding materials. Among them, γ-Ni-22Fe alloy has been particularly emphasized because of its higher permeability, lower coercivity and relative high saturation magnetization. Therefore, it would show synergetic combination of improved mechanical properties and excellent magnetic functionality by Ni-xFe/Al_2O_3 composite, which may realize a functionally structural composite material[6,7].

Recently many techniques have been used to prepared ultra-fine grained metal/ceramic-base composites or nanocomposites, such as hot-pressing of the fine ceramic and metal powder mixture[8], gelcasting process[9], spark plasma sintering technique, *etc.*[10]. In this paper, ultra-fine grained Ni-22Fe/Al_2O_3 composite were prepared by mechano-chemical method combining with hot-pressing technique and magnetic properties of Ni-22Fe/Al_2O_3 composite were investigated.

EXPERIMENTAL

The nanocrystalline γ-Ni-22Fe alloy was prepared by using the mechano-chemical alloying process as described elsewhere[11]. The reagent grade NiO and Fe_2O_3 powder were mixed stoichiometrically and ball milled by high-energy ball milling. Then the milled powders were reduced and *in situ* alloying at 600 ºC for 0.5 h to form nanocrystalline γ-Ni-22Fe alloy.

Commercially available alumina (α phase; purity: > 99.99 %) was ball-milled in alcohol for 5 h to minish particle size. Then the nanocrystalline γ-Ni-22Fe alloys was chosen to be 8 and 12 wt % in the corresponding composite systems. Weighted powders were mixed by high-energy ball milling. The re-milled powders were subsequently reduced in H_2 at 600 ºC for 10 min to eliminate possible surface oxidation of nanocrystalline γ-Ni-22Fe alloy. Then disk-shaped bulk

†Presented to The 5th Korea-China International Conference on Multi-Functional Materials and Application.

samples (with the size of 60 mm diameter and 3.5 mm thickness) were obtained by hot-pressing at 1400 and 1450 ºC for 40 min under applied uniaxial stress of 30 MPa in high-purity argon atmosphere. For comparison, a monolithic alumina sample was prepared under the identical condition.

The microstructure was examined by X-ray diffraction with CuK_α radiation. Mean grain size was evaluated based on Scherrer formula. The main constituent elements were determined by energy dispersive X-ray spectroscopy (EDS) equipped in transmission electron microscope (TEM). Microstructural characteristics such as morphology and particle size were further confirmed by atomic force microscope (AFM). The densities of specimens were investigated based on Archimedes principle. The relative densities of the specimens were around 0.98. Magnetization measurements were measured with a LakeShore 7307-9309 vibrating sample magnetometer (VSM) under an external field between -10 and 10 kOe at room temperature.

RESULTS AND DISCUSSION

Synthesis and microstructure of nanocrystalline γ-Ni-22Fe alloy: Fig. 1 shows a typical XRD pattern of nanocrystalline γ-Ni-22Fe alloy reduced in H_2 atmosphere and *in situ* alloying at 600 ºC for 0.5 h. All diffraction peaks are identified to be a face central cubic (fcc) crystalline structure (γ phase). The average grain size of sample evaluated based on Scherrer formula:

$$d = \frac{0.9\lambda}{\beta\cos\theta}$$

where λ is the X ray wavelength, β is the full width at half-maximum and θ is the Bragg angle corresponding to the diffraction peak. The grain size of obtained nanocrystalline γ-Ni-22Fe alloy powders is around 20 nm at 600 ºC. Fig. 2 gives a typical TEM image of specimen at 600 ºC for 0.5 h. One can see that the grain sizes of specimen are around 30 nm, which is consistent with XRD analysis results. There are a few of large agglomerate with particles size of 100 nm.

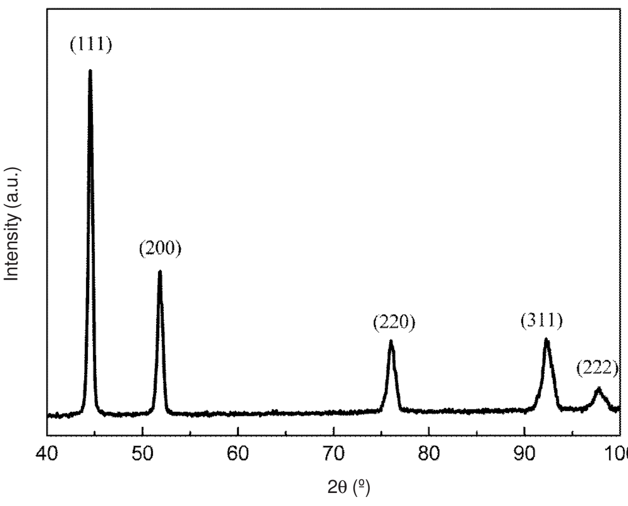

Fig. 1. XRD patterns of nanocrystalline γ-Ni-22Fe alloy by mechanochemical alloying reduced at 600 ºC for 0.5 h

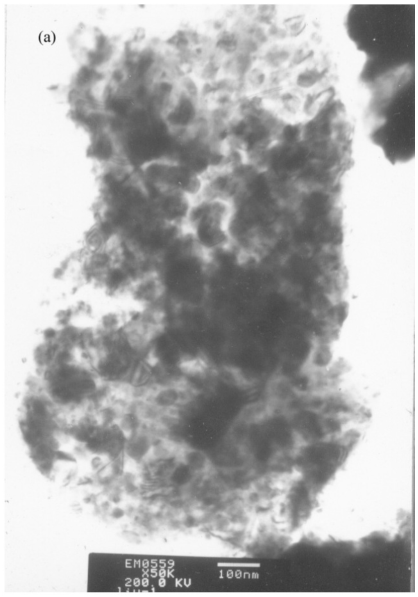

Fig. 2. TEM micrograph of nanocrystalline γ-Ni-22Fe alloy reduced at 600 ºC for 0.5 h

Microstructure of Ni-22Fe/Al_2O_3 composite by hot pressing sintering: Fig. 3 shows XRD patterns for the bulk composite samples 12 wt % (Ni-22Fe)/Al_2O_3 and monolithic alumina sample sintering at 1450 ºC for 40 min compared to the pattern of the alumina sample, it is observed that there are three additional reflection peaks locating at 43º, 51º and 76º for 12 wt % (Ni-22Fe)/Al_2O_3 composite, all of which are identified to originate from γ-Ni-22Fe alloy, indicating that there are only two phases in the composites and no byproduction such as $NiAl_2O_4$ was observed as shown in Fig. 3.

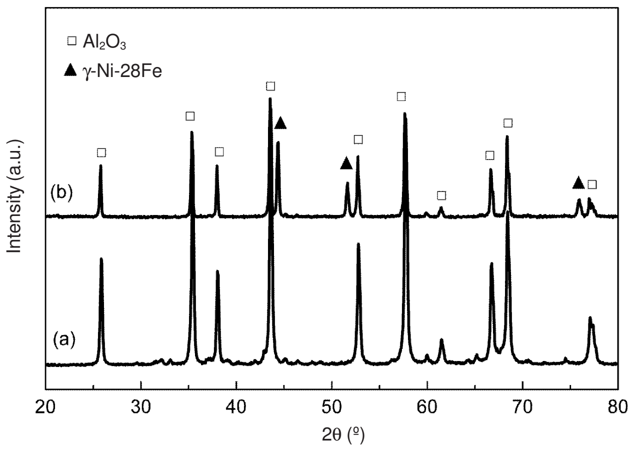

Fig. 3. XRD patterns for (a) monolithic α-Al_2O_3, (b) 12 wt % (Ni-22Fe)/Al_2O_3 composite

Fig. 4 shows typical AFM images of 12 wt % (Ni-22Fe)/Al_2O_3 composite sintering at 1450 ºC for 40 min. It can be seen that some black spots identified to be Ni-22Fe alloy, appearing on the alumina matrix grain boundaries. The particle size of γ-Ni-22Fe alloy is around *ca.* 500 nm and alumina matrix has the mean grain size of several micronmeter.

Magnetic properties of Ni-22Fe/Al_2O_3 composites: Fig. 5 show a hysteresis loop of 12 wt % (Ni-22Fe)/Al_2O_3 composite at room temperature. Magnetic measurements reveal that

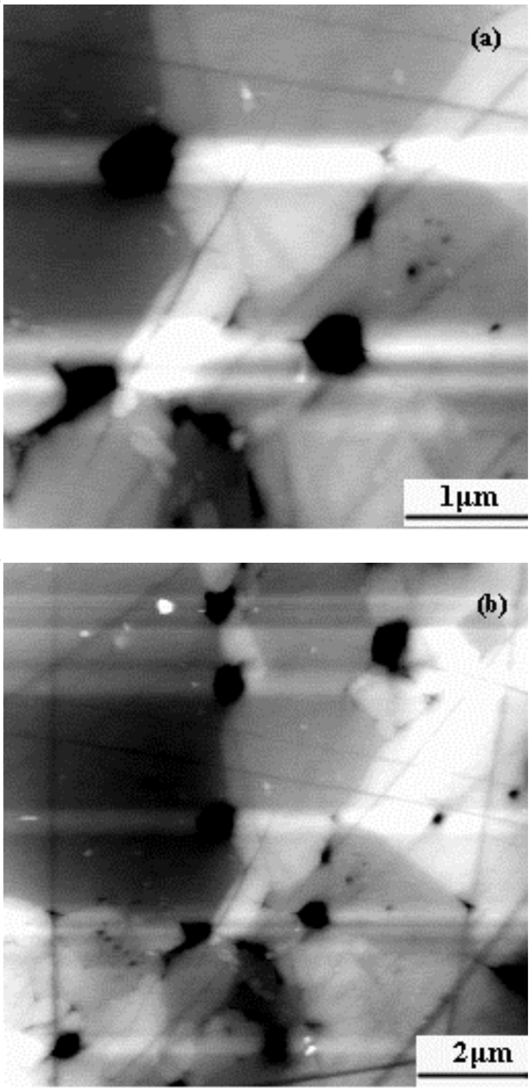

Fig. 4. Typical AFM images of 12 wt % (Ni-22Fe)/Al_2O_3 composite (a) image size 5 μm × 5 μm; (b) image size 10 μm × 10 μm

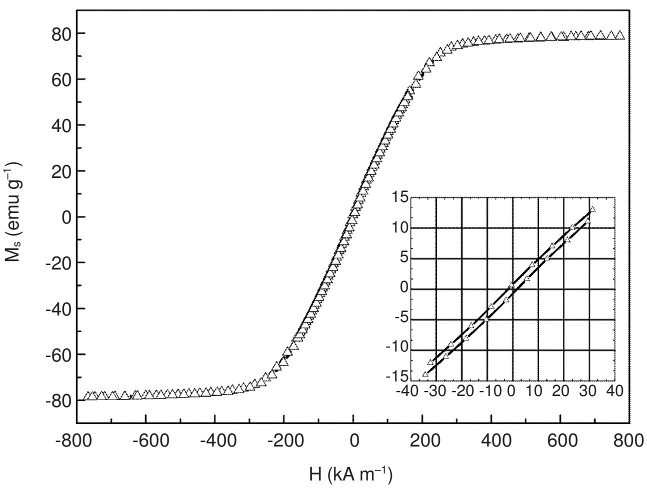

Fig. 5. Hysteresis loop of 12 wt % (Ni-22Fe)/Al_2O_3 composite at room temperature (insert shows enlarge of the plot near the origin)

that the specimens exhibit ferromagnetism. It can be seen that the magnetization of sample reached saturation at the external field of *ca.* 400 kA m^{-1}. Saturation magnetization Ms is around 78.1 emu/g and coercivity is 22.41Oe at room temperature. The displayed ferromagnetism should originate from incorporated magnetic Ni-22Fe alloy particles. Compared with the magnetic properties of coarse-grained permalloy[5], the coercivity of γ-Ni-22Fe/Al_2O_3 composites is much higher than conventional Ni-Fe alloy (usually several oersteds), while its saturation magnetization Ms becomes lower.

It is well known that the magnetic properties of nanostructural magnetic materials depend on its chemical composition and grain size[12]. The increase of coercivity of Ni-22Fe/Al_2O_3 composites may be attributed to the decreased particle size and isolated dispersion in ceramic-matrix. And residual stress are also responsible for the magnetic properties of Ni-22Fe/Al_2O_3 composites[4].

Conclusion

In summary, nanocrystalline γ-Ni-28Fe alloy was successfully synthesized by using mechano-chemical process. Ultrafine grained γ-Ni-22Fe/Al_2O_3 composites were fabricated with using hot-pressing sintering of nanometer Ni-22Fe alloy/Al_2O_3 mixed powder. Sub-micrometer-sized matrix Ni-22Fe particles are dispersed homogeneously at the Al_2O_3 matrix. Ferromagnetic γ-Ni-22Fe alloy dispersion led Ni-22Fe/Al_2O_3 composites to display ferromagnetism with the decrease of saturation magnetization and the increase of coercivity. The Ni-22Fe/Al_2O_3 composites with magnetic performance would have potential functional applications.

ACKNOWLEDGEMENTS

This work was supported by the Natural Science Foundation of the High Education School of Anhui Province, China (No: KJ2010A095).

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