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Journal of Alloys and Compounds 429 (2007) 357–364

Journal of ALLOYS AND COMPOUNDS

www.elsevier.com/locate/jallcom

Influence of process parameters and alloy composition on structural, magnetic and electrical characteristics of Ni–Fe permalloys

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Received 2 July 2006; received in revised form 27 October 2006; accepted 1 November 2006
Available online 19 December 2006

Abstract

Structural, magnetic and electrical studies have been carried out in three Ni–Fe based permalloys (82.13, 79.90 and 47.01% Ni) under hydrogen atmosphere by varying process parameters such as annealing temperature, cooling rate and holding time. These materials have been characterized for various magnetic properties such as remanence, coercivity, peak permeability and core loss under changed annealing profile conditions and correlated the results with XRD and microstructural observations. We found that grain diameter and ordering parameter have significant effect on the magnetic properties of the alloys. 82.13% Ni alloy is found to show better magnetic properties over the other two alloys as a function of different process parameters. We attribute it to relatively larger grain diameter and short-range order (SRO) developed in 82.13% Ni alloy over other two alloys. The alloys were tested for watch movement and audio head applications. We found that the battery life of the watch movement improved by 38% using 79.90% Ni alloy over 47.01% Ni alloy. Also, found that audio head performance is better with 82.13% Ni alloy as compared to 79.90% Ni alloy.

Keywords: Annealing; Magnetic properties; Microstructure; Electro-mechanical characteristics; Playback characteristics

1. Introduction

The Ni–Fe alloys are based on the face-centered cubic (FCC) portion of Ni–Fe system and contain about 40–90% Ni, up to a few percent each of other alloying elements like Cu, Mo, Cr, Co, Mn and balance is Fe [1–3]. Their remarkable magnetic properties have made them the subject of intensive study over the last about half a century. Three ranges of nickel content are used as soft magnetic alloys: 36% Ni for maximum resistivity, 50% Ni for maximum saturation magnetization and 80% Ni for optimum initial and maximum permeabilities [4,5]. From the above alloys, the 50 and 80% Ni alloys are most widely used in rotor and stator laminations, stepping motors, shieldings, relay parts, audio head, etc.

Watch is an electro-mechanical device, which contains some mechanical, electrical and magnetic components. The heart of the movement is a bipolar stepper motor that converts electrical energy to magnetic energy and then to mechanical motion [6,7]. The stepper motor consists of rotor, a stator and a coil. The rotor is formed by a permanent magnet whereas core and stator are made up of Ni–Fe permalloy.

Similarly, the audio recording head comprises of three Ni–Fe permalloy components, each has its own valuable contribution in audio recording. These are case, core and shield plate, where core act as a media for the magnetic flux to travel in, case is for blocking out the noise and protection of the magnetic circuit and resin and shield plate is meant for prevention of cross talking between different channels [8,9].

The magnetic properties of the permalloys not only depend on the heat treatment under hydrogen atmosphere [2,3,10–14] but also depend on the values of the anisotropy and magnetostriction constants. Both the parameters further depend on the degree of short-range order (SRO) developed in the materials [15]. Optimum magnetic properties are obtained when a critical degree of SRO is developed in these alloys, which allow both the anisotropy energy and magnetostriction constant to be simultaneously reduced to small values. Various degrees of order can

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0925-8388/$ – see front matter
doi:10.1016/j.jallcom.2006.11.048

be obtained by controlled cooling of the specimens at different rates in the temperature range between about 500 and 400 °C. The structure sensitive magnetic properties such as coercivity, permeability and hysteresis loop shape of the Ni–Fe alloys also depend on the microstructure. These are governed by magnetization process of domain nucleation, domain wall motion and domain rotation. These processes in turn are governed by the intrinsic properties such as saturation magnetization and magnetic anisotropy energy as well as microstructural details such as inclusion content and grain size. From domain theory, rotational processes leads to general expression [16]

$$\mu_i \propto \frac{M_s^2}{K_{eff}} \tag{1}$$

where μ_i is the initial permeability, M_s the saturation magnetization and K_{eff} is an effective anisotropy constant covering all sources of anisotropy energy. A domain wall motion model taking grain size into effect has the expression [17]

$$\mu_i \propto \frac{M_s^2 d}{(AK)^{1/2}} \tag{2}$$

where A is exchange constant, K the anisotropy constant and d is the grain diameter. Hence, the maximization of initial permeability requires maximization of M_s, d and minimization of K. The coercive field strength H_c determined by the grain boundaries is a linear function of the reciprocal grain diameter [11]

$$H_c \approx \frac{3\gamma_w}{J_s d} \tag{3}$$

where J_s is the saturation polarization, γ_w the wall energy, which is proportional to $\sqrt{K_1}$ and K_1 is the first order magneto-crystalline anisotropy constant. The magneto-crystalline anisotropy K_1, depends not only on alloy composition but also on the degree of long and short-range order of Ni_3Fe, which is stable below 600 °C. The grain diameter has also great effect on magnetic losses [18–21]. When the grain size diameter d increases, the hysteresis loss decreases in proportion to $1/d$. The total core loss is minimized at an optimum grain diameter. This optimum grain size is obtained after the final annealing.

This paper describes the influence of various important process parameters such as annealing temperature, cooling rate and holding time under hydrogen atmosphere on the magnetic properties of the three different permalloy materials. The phase analysis, microstructural characterization and magnetic measurements are carried out by XRD, optical microscope and B-H analyser, respectively. All indigenously developed materials were tested for watch and audio head applications. The electro-mechanical characteristics of watch movement and playback characteristics of audio head have been studied to compare the performance of the device developed through the proposed route with the existing one.

2. Experimental procedure

The three Ni–Fe permalloys of 82.13% Ni (sample A), 79.90% Ni (sample B) and 47.01% Ni (sample C) have been investigated for the present study. The detail composition of the samples was determined by wet chemical analysis and atomic absorption spectrometer (GBC, Model 932 AA) and is given in Table 1. The samples in the form of rings having outer and inner diameter of 10 and 6 mm, respectively, were punched from strip by the use of a specially designed punch and die. These samples were annealed in a purified dry H_2 atmosphere with a dew point of about −60 °C under different process parameters like variable holding time, cooling rate and annealing temperature. The different annealing profiles performed on the samples are given in Table 2.

Table 1
Chemical compositions of the Ni–Fe permalloys

Element	Sample A	Sample B	Sample C
Ni	82.13	79.90	47.01
Fe	12.38	14.47	49.50
Mo	4.90	5.00	–
Mn	0.41	0.43	0.38
C	0.01	0.02	0.05
Si	0.10	Traces	0.63
Cr	0.10	–	–
Co	0.05	–	0.03
Cu	0.05	–	–

The crystal structure of the annealed samples was analysed by X-ray diffractometer (Rigaku, D-max IIIC). The diffractometer comprised of a copper target, a curved single crystal monocromator of graphite and NaI scintillation counter as a detector. The strip sample used for analysis was of the size (19 mm × 17 mm) held in 0.5 mm deep cavity of a sample holder with the help of binder. Diffraction pattern was recorded at a scanning speed of 3°/min. The sample data was matched with the standard database compiled in software PCPDFWIN provided by the International Centre for Diffraction Data—ICDD (formerly known as JCPDS, The Joint Committee on Powder Diffraction Standards) [22,23]. The microstructures of the annealed samples were developed using marble's reagent (50 ml HCl, 10 g $CuSO_4$, 50 ml H_2O) and optical microscope (Nikon, Japan). Several micrographs were taken in order to precisely evaluate the microstructure of each sample with the help of image analysis system (Sis, Germany). The average grain size of the samples was evaluated using linear intercept method [24]. Eq. (4) represents the linear intercept method, where d is the diameter of the grain, L the length of the line, N the number of intersections and M is the image's magnification.

$$d = \frac{L}{NM} \tag{4}$$

The toroid shape samples were prepared by stacking rings and using 20 numbers of primary and secondary turns. The ac magnetic properties were studied using B-H analyser (AMH-401, Walker Scientific, USA) under different processing parameters.

The annealed samples of core and stator were tested in the watch movement. The current consumption of the movement was measured at 1.5 V by increasing the resistance of the coil core from 2.30 to 3.50 kΩ and simultaneously the torque of the movement was measured using torque meter (Witchi, China). The standard battery SR 626 having battery life of 24 mAh was used in the movement for the measurement of current consumption. The formula used for calculating the battery life is [25]:

$$\text{Battery life of movement (h)} = \frac{\text{Standard life of battery}}{\text{Current consumption of the movement}}$$

Table 2
Annealing profiles performed on samples

Annealing temperature (°C)	Holding time (h)	Cooling rate through the ordering range, 700–300 °C, (°C/min)
1100, 1120, 1140, 1150, 1160, 1180	2	2.5
1150	1, 2, 3	2.5
1150	2	2.0, 2.5, 3.0, 5.0, 7.0