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Comparative Study on Magnetic Properties and Microstructure of As-prepared and Alternating Current Joule Annealed Wires

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Abstract

X-ray diffraction (XRD), high-resolution transmission electron microscopy (HRTEM), magnetic measurement including impedance measurement were used for investigating the microstructure and magnetic properties of as-prepared and alternating current Joule annealed (ACJA) Co-rich amorphous microwires for potential sensor applications. Experimental results indicated that as-cast and ACJA wires both were amorphous characteristic, while ACJA wire has an enhanced local ordering degree of atom arrangement. There was a transform of magnetic properties after ACJA treatment, namely increasing coercivity, maximum magnetic permeability and saturation magnetization, resulting from the coactions of magnetic anisotropy and magnetic moment exchange coupling. Moreover, ACJA treatment can drastically improve the GMI property of melt-extracted wires. At 5MHz, the maximum GMI ratio $[\Delta Z/Z_0]_{\max}$ of ACJA wire increases to 205.93%, which is nearly 4.1 times of 50.62% for as-cast wire, and the field response sensitivity ζ_{\max} of ACJA wire increases to 463.70%/Oe by more than 2 times of 212.15%/Oe for as-cast wire. From sensor application perspective, the sensor applied frequency range (SAFR) of ACJA wire is 3MHz-7MHz (the better working frequency is at 5MHz). It can therefore be concluded that the ACJA wire (60mA, 480s, 50Hz) has better GMI and magnetic properties, is more suitable for potential magnetic sensor applications working at low-frequency and relatively high-working-magnetic field.

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1. Introduction

Much interest has been paid to the excellent giant magnetoimpedance (GMI) amorphous wires owing to their potential GMI sensor applications specially to detect weak magnetic field^[1]. Melt-extracted microwire exhibits higher solidification rate and more unique super-soft magnetic properties in comparison with in-rotating water spinning and glass-coated melt spinning^[2]. Importantly, there is an important trend of the selection for more sensitive GMI materials: searching novel magnetic microwires with enhanced GMI and magnetic properties. Generally, this purpose can be achieved through some proper and effective post-processing treatments for as-prepared microwires, e.g. Joule annealing^[3].

X.Z. Zhou et al.^[4] investigated that the effect of AC Joule-heating annealing (including annealing current strength & waveform, annealing duration time, cooling conditions and the geometry of samples) of melt-spun $\text{Co}_{68.25}\text{Fe}_{4.5}\text{Si}_{12.25}\text{B}_{15}$ amorphous ribbon on GMI effect extensively and it can influence the easy direction magnetization, magnetic anisotropy and domain structure. It is greatly improved the GMI ratio $[\Delta Z/Z_0]_{\max}$ of about 180% at 900kHz for special annealing conditions (annealing 30 min with applied AC current of 2.8×10^7 A/m²). K.D. Sossmeier et al.^[5] compared the magnetic properties of CoFeSiB glass-covered amorphous microwires treated by AC (100Hz or 500Hz) Joule heating, with an increase of applied stress, both GMI ratios are achieved to 100%, and the behavior of the GMI curves exhibits a tendency from single peak to double peak, the feature of AC500 sample is related to the modifications of the anisotropy directions induced by the applied stress and the relative changes in the transverse permeability associated with the circumferentially magnetized shell. On the other hand, the magnetic properties are closely related to microstructures yielded by ACJA from structure-property perspective. However, there also lacks the comparative study on magnetic properties and microstructure of as-cast and ACJA microwires, so it is worthwhile to conduct this investigation.

This paper aims to comparatively investigate the magnetic properties and microstructure of as-cast and ACJA microwires, and identify some wires with better GMI properties for possible sensor applications.

2. Experimental details

In this experiment, mother alloy ingot with the nominal composition of $\text{Co}_{69.25}\text{Fe}_{4.25}\text{B}_{10.5}\text{Si}_{13}\text{Nb}_3$ (in at. %) was fabricated by arc-melting in pure argon and copper mould casting methods. Subsequently, the top-end of club-shaped alloy in BN crucible was melted by induction coil in melt-extraction facility. Then, the microwires were extracted by the edge of a high speed rotating copper wheel in purified argon atmosphere. Therefore, the microwires with diameter around 35 μm were selected and post-processed by Joule annealing. AC Joule annealing (ACJA) was conducted by passing an AC current (the average amplitude: $I_m=60\text{mA}$, annealing duration time: $t=480\text{s}$ and AC frequency: $f=50\text{Hz}$, air cooling) supplied by a mini-type self-design ACJA device through the amorphous Co-rich microwire.

The as-cast and ACJA microwires were examined by X-ray diffraction with CuK_α radiation (XRD, Rigaku D/max- γB), high-resolution transmission electron microscopy (HRTEM, JEM 2010F). The magnetic properties were performed by vibrating sample magnetometer (VSM) with a maximum applied field of 0.2 T. The impedance measurements were performed using Aligent 4294A precision impedance analyzer at frequencies 40Hz-110MHz. And sample length for impedance measurement is about 18mm. GMI ratio, $\Delta Z/Z_0$, is defined as:

$$\frac{\Delta Z}{Z_0} (\%) = \left[\frac{Z(H_{ex}) - Z(H_0)}{Z(H_0)} \right] \times 100\% \quad (1)$$

and magnetic field response sensitivity, ζ , is expressed as:

$$\xi = \frac{d \left[\frac{\Delta Z}{Z_0} (\%) \right]}{dH_{ex}} \quad (2)$$

where $Z(H_{ex})$ is the impedance under external field, H_{ex} applied by a long solenoid is below 4.25Oe. $Z(H_0)$ is the initial impedance at 0Oe. All measurements were performed at room temperature (25 °C).

3. Results and discussion

Fig.1 (a) shows X-ray diffraction patterns of as-cast and ACJA wires. Both XRD patterns consist of one broad diffused maximum, which indicate that the microstructure of wires entirely consists of amorphous phase or structure. Fig.1 (b) displays the HRTEM morphology and fast Fourier transformation (FFT) pattern images of as-cast and ACJA microwires. The surface HRTEM morphology of as-cast and ACJA wires is quite smooth, uniform and no obvious formation of nanocrystalline with large size. Consistent with the XRD results, both the FFT patterns of wires only consist of halo rings, exhibiting mainly amorphous characteristic. In deed, there is an obvious difference of local ordering degree between as-cast and ACJA microwires, and the ACJA wire exhibits an enhanced degree of local ordering of atomic arrangement by the statistical calculation of auto-correlation function (ACF) technique conducted by DigitalMicrograph software (not shown). It means ACJA annealing treatment can induce atomic diffusion with releasing internal residual stress and the formation of regularly arranged atomic micro-regions (RAAMs) in the amorphous wires and consequently increases the regularity of atomic arrangement under the action of thermal activation and AC magnetic field energy^[6]. Therefore, the ACJA wire is more likely to possess better soft magnetic properties than as-cast wire.

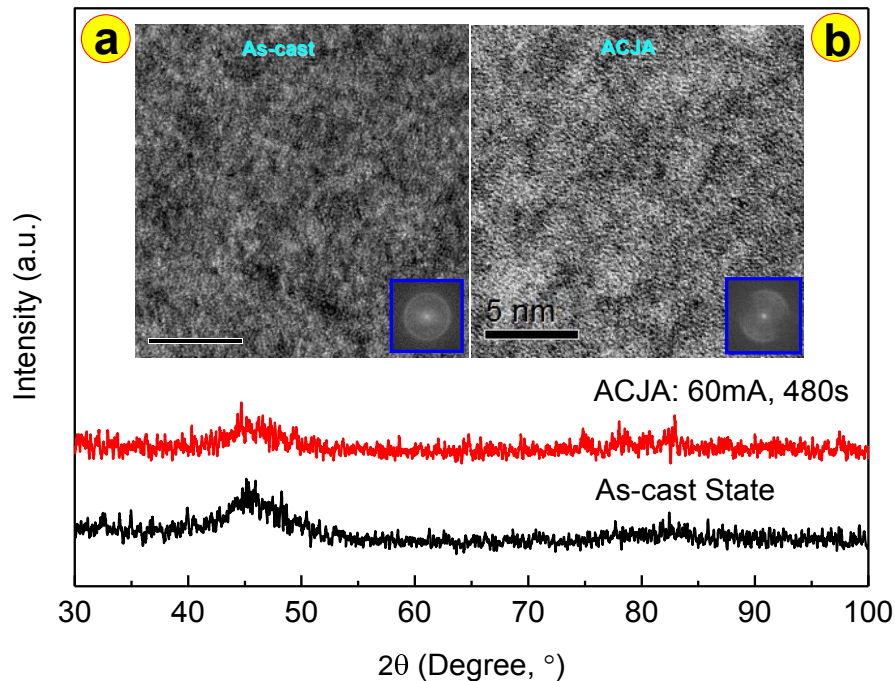


Fig. 1. (a) X-ray diffraction patterns; (b) HRTEM images (including FFT patterns) of as-cast and ACJA Co-rich microwires.

Fig. 2 illustrates the longitudinal magnetic hysteresis loops of as-prepared and ACJA microwires. The shapes of hysteresis loops are nearly rectangles, which exhibit excellent soft magnetic characteristics, and their magnetization is easy to achieve the saturation state at relatively low external magnetic field. There was a variation of magnetic properties in both microwires, including that the coercivity H_c slightly increases from 0.2459Oe of as-cast wire to 0.5437Oe of ACJA wire, the saturation magnetization M_s drastically increases from 53.849emu/g of as-cast wire to 65.026emu/g of ACJA wire, and the maximum magnetic permeability μ_{max} rapidly increases from 0.7352 of as-cast wire to 1.1801 of ACJA wire etc. In addition, the electrical resistivity ρ_m increases from 117.06 $\mu\Omega\cdot\text{cm}$ of as-cast wire to 148.39 $\mu\Omega\cdot\text{cm}$ of ACJA wire. These modified magnetic properties of ACJA wire can be accounted for by the change of local ordering degree of atomic arrangement, i.e. RAAMs, further to achieve quick rotational magnetization and generate the stable circular magnetic domain distribution, which lead to an increase of circumferential magnetic anisotropy and an increase of magnetic moment exchange coupling by the action of external magnetic field H_{ex} .

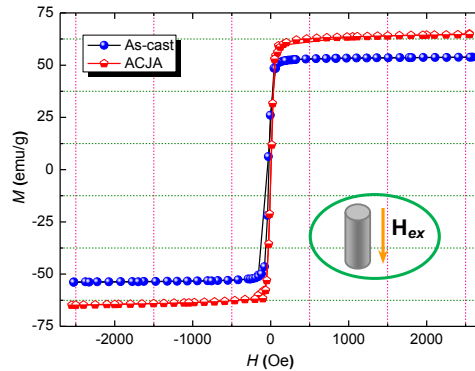


Fig. 2. Longitudinal hysteresis loops (M - H) of as-cast and ACJA microwires. The uni-directional arrow indicates the direction of external magnetic field H_{ex} .

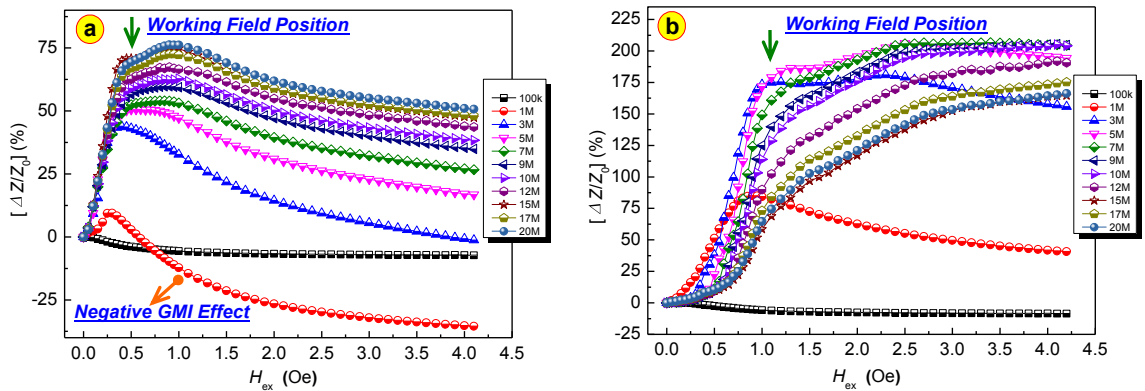


Fig. 3. (a) GMI ratio $\Delta Z/Z_0$ dependence of external magnetic field H_{ex} (0–4.25 Oe) of as-cast wire; (b) GMI ratio $\Delta Z/Z_0$ dependence of H_{ex} of ACJA (60mA, 480s, 50Hz) wire. The orange uni-directional arrow in (a) indicates the negative GMI effect.

Fig. 3 reveals the dependence of GMI ratio $\Delta Z/Z_0$ on external magnetic field H_{ex} (0–4.25Oe) of as-cast and ACJA (annealing at 60mA for 480s at 50Hz) microwires. Form above mentioned GMI profiles of wires, all curves increases rapidly at first (0–1.0Oe) then decreases monotonically or keeps small variation with an

increase of the DC magnetic fields at the selected frequencies. The maxima GMI ratio $[\Delta Z/Z_0]_{\max}$ values of as-cast wire are 9.44%, 43.92%, 50.62%, 54.03%, 60.72%, 74.99% and 76.24% at 1MHz, 3MHz, 5MHz, 7MHz, 10MHz, 15MHz, 20MHz, respectively, as shown in Fig. 3(a). In particular, the $\Delta Z/Z_0$ curve at 1MHz of as-cast wire displays a negative GMI effect with increasing field as a result of inhomogeneous distribution of the locally critical field of magnetization rotation. So, it is hard to achieve the complete rotational magnetization under relatively high external magnetic field, even the inductance X of impedance Z appears to an obvious variation at some relatively low frequency. Generally, the resistance R of Z is not changed at different frequencies, Z decreases with X decreasing, then it gives birth to negative GMI effect. The $[\Delta Z/Z_0]_{\max}$ values of ACJA wire are 85.01%, 180.99%, 205.93%, 206.61%, 205.02%, 164.61% and 166.18% at 1MHz, 3MHz, 5MHz, 7MHz, 10MHz, 15MHz, 20MHz, respectively, as seen in Fig. 3(b).

Fig. 4 shows the magnetic field sensitivity ζ_{\max} (%/Oe) and its corresponding GMI peak position H_p (namely the equivalent anisotropy field H_k) of ACJA microwire at selected frequencies ranging from 100kHz to 20MHz. At 5MHz, ACJA wire has larger $[\Delta Z/Z_0]_{\max}$ of 205.93%, maximum field sensitivity ζ_{\max} of 463.70%/Oe and relatively larger $H_p=0.75$ Oe comparing with as-cast of GMI effect (H_p corresponds to the working magnetic field range of sensor, as shown in Fig.3), which are nearly 4.1 times of 50.62% and more than 2 times of 212.15%/Oe and $H_p=0.60$ Oe for as-cast wire respectively. At 10MHz, ACJA wire also has relatively higher $[\Delta Z/Z_0]_{\max}$ of 205.02%, ζ_{\max} of 317.74%/Oe and $H_p=0.90$ Oe comparing with as-cast of GMI effect, which are nearly 3.3 times of 62.52%, nearly 1.8 times of 176.30%/Oe and $H_p=0.85$ Oe for as-cast wire respectively. Notably, from sensor application perspective, namely larger $[\Delta Z/Z_0]_{\max}$, ζ_{\max} and relatively higher H_p at relatively low frequency is propitious to improve the resolution and accuracy of GMI sensor, according to values of $[\Delta Z/Z_0]_{\max}$ and ζ_{\max} of ACJA wire, the sensor applied frequency ranges from 3MHz to 7MHz, and the better applied or working frequency is at 5MHz. In conclusion, the ACJA (60mA, 480s, 50Hz) wire with enhanced GMI property is therefore more suitable for use as the magnetic sensitive materials working at low-frequency (3MHz-7MHz) and relatively high-magnetic field (0-0.9Oe) of high-resolution GMI sensor.

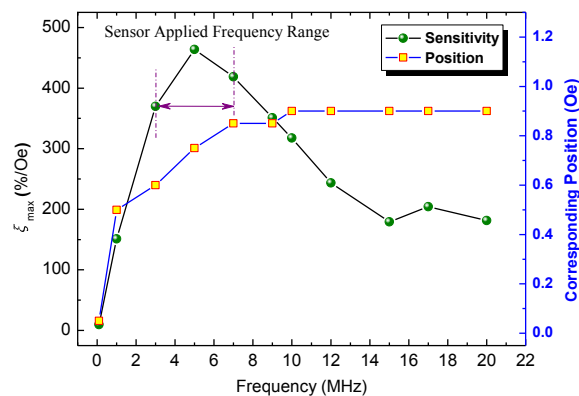


Fig. 4. Frequency dependence of field response sensitivity ζ_{\max} (%/Oe) and its corresponding GMI peak position H_p at selected frequencies of ACJA microwire. The bi-directional arrow indicates the sensor applied frequency range (SAFR) of 3MHz-7MHz.

4. Conclusions

It can be seen from above results that the ACJA treatment has remarkable effect on GMI and magnetic properties. In comparison with as-cast wire, the ACJA (annealing at 60mA for 480s at 50Hz, air cooling) wire with better magnetic properties containing slightly larger coercivity, higher magnetic permeability and

saturation magnetization, even including enhanced GMI effect, exhibiting a maxima GMI ratio $[\Delta Z/Z_0]_{\max}$ of 205.93% and the maximum field response sensitivity ζ_{\max} of 463.70%/Oe by more than 4.1 times and 2 times of as-cast wire at 5MHz, and the GMI peak position tends to be 0.75Oe and 0.90Oe at 5MHz and 10MHz respectively, is a promising candidate sensitive material working at 3MHz-7MHz to potential high-performance GMI sensor applications.

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