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## Role of the Co-based microwires/polymer matrix interface on giant magneto impedance response



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### Diana Estévez, Jiawei Li, Gang Liu, Qikui Man<sup>\*</sup>, Chuntao Chang, Xinmin Wang, Run-Wei Li

Key Laboratory of Magnetic Materials and Devices, Ningbo Institute of Materials Technology & Engineering, Chinese Academy of Sciences, Ningbo, Zhejiang 315201, China Zhejiang Province Key Laboratory of Magnetic Materials and Application Technology, Ningbo Institute of Materials Technology & Engineering, Chinese Academy of Sciences, Ningbo, Zhejiang 315201, China

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#### A B S T R A C T

The interface of Co-based microwires-epoxy composites was modified by applying silane treatment on the surface of the wires and their magneto impedance (MI) response was evaluated. The aim of the surface treatment was to modify the residual stresses that coexist at the microwires/polymer matrix interface and hence the magnetic anisotropy. X-ray Photoelectron Spectroscopy confirmed the covalent attachment of silane molecule onto the wires surface by the presence of Si–O–Si and Fe–O–Si. The MI curve changed from single peak for untreated samples to double peak behavior for treated samples with a significant improvement of MI ratio. Additionally, the magnitude of the anisotropy field increased with the frequency, which may imply a strongly non-uniform stress distribution towards the surface. The MI variation was explained by the change of the surface magnetic anisotropy owing to the modification of the microwires/polymer matrix interface.

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#### 1. Introduction

Ferromagnetic microwires have found wide applications in sensing applications including magnetic guidance systems, biomedical sensors and geomagnetism [\[1\]](#page--1-0). The functionality of these microwires has improved when they have been used as fillers of composites. Since, properties of composites depend primarily on the properties of their fillers, research efforts aiming to understand and develop optimal formulation strategies of these fillers hold a huge importance and potential in developing more efficient ferromagnetic devices. One of the important characteristics of these microwires is the giant magneto impedance (GMI) effect. The GMI refers to the large change in the ac impedance,  $Z = R + jwL$  $(R$  and  $L$  are the resistance and inductance, respectively) of a magnetic conductor with an ac*l* =  $I_0$ exp( $-jwt$ ) when subjected to an applied dc magnetic field  $(H_{dc})$  [\[2\].](#page--1-0)

The GMI effect is dependent on different factors, such as alloy composition, internal stresses, thermal and stress annealing and geometry of the sample. In all the cases, the impedance change is defined by the value and distribution of the magnetic anisotropy. Annealing under mechanical stress or magnetic field has been shown to induce magnetic anisotropies and the consequent modification of the domain structure  $[3,4]$ . A typical example of the domain structure modification caused by changes of internal stresses is the case of removal of the glass-cover layer from amorphous glass-covered wires [\[5\].](#page--1-0) Interestingly, such modification of domain structure, as removal of glass-cover layer, can lead to considerable variation in the magnetic properties of the wire and hence in GMI effect. An enhanced GMI performance was also obtained in multiwire composites through the collective response of the microwires in the presence of an external magnetic field  $[6,7]$ . However, no study on the interfacial properties of these composites has been reported till date. In the composite, in addition to the residual stresses developed in the wire during fabrication, there exist residual stresses at the interface between the wire and polymer matrix. The modification of GMI effect in these composites clearly points to the important coupling between internal and external stresses that coexist in these materials. Therefore, controlling the stress in the polymer-wire interface could be a way to modify GMI response. Equally, the interfacial stress will have important influence on the magnetoelastic features of the wires according to  $K_{me} = 3/2\lambda(\sigma_i + \sigma_{app})$  where  $K_{me}$  is the magnetoelastic energy component,  $\lambda$  is the magnetostriction,  $\sigma_i$  and  $\sigma_{app}$  are the internal stress and applied stress on the wire, respectively  $[8]$ . In this work, we modified the interface between Co-based microwires and epoxy matrix by applying a chemical treatment on the wires using silane.

<sup>⇑</sup> Corresponding author at: Key Laboratory of Magnetic Materials and Devices, Ningbo Institute of Materials Technology and Engineering, Chinese Academy of Sciences, No. 1219 Zhongguan West Road, Zhenhai District, Ningbo 315201, China. Tel./fax: +86 574 87911392.

E-mail address: [Manqk@nimte.ac.cn](mailto:Manqk@nimte.ac.cn) (Q. Man).



Fig. 1. The procedure of the coating reaction of APTS with  $Co_{63}Fe_4B_{22.4}Si_{5.6}Nb_5$  amorphous wires.



Fig. 2. XPS spectra of untreated wires (a) N 1s and (b) Si 2p.

Silane coupling agents are silicon-based chemicals that contain two types of reactivity – inorganic and organic – in the same molecule. A typical general structure is  $(RO)_3$ SiCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>–X where RO is a hydrolysable group, such as methoxy, ethoxy or acetoxy, and X is an organofunctional group, such as amino, metacryloxy and epoxy. The organofunctional groups are chosen for reactivity or compatibility with the polymer while the hydrolysable groups interact with inorganic substrates through hydrogen bonding to surface hydroxyl groups [\[9\]](#page--1-0). The surfaces of the untreated and treated wires were characterized with X-ray Photoelectron Spectroscopy (XPS) and the GMI properties of the composites were evaluated.

#### 2. Experiment

#### 2.1. Sample preparation

 $Co_{63}Fe_{4}B_{22.4}Si_{5.6}Nb_{5}$  amorphous wires with average diameter of 100 µm were prepared by arc melting type melt extraction method. They were cleaned and degreased in ethanol and distilled water before being exposed to a base/acid sequence to enhance the concentration of surface hydroxides (10 min. sonication in 1 M NaOH, 5 min. sonication conc.  $H_2SO_4$ ). The -OH on the surface may react with the silane molecule as the process shown in Fig. 1. The cleaned wires were then immersed for 30 s in a 2% solution of 3-Aminopropyltriethoxysilane (APTS) in acetone, rinsed free of excess materials with acetone and allowed to dry at room temperature for at least 2 h before use.

#### 2.2. Surface characterization of untreated and treated wires

XPS spectra were obtained using a Kratos AXIS ULTRADLD instrument with a monochromic Al K $\alpha$  X-ray source (hv = 1486.6 eV). Spectral responses were subjected to standard curve fitting procedures in order to determine the chemical state of the elements. All peak binding energies presented were corrected for charging against the C 1s binding energy that was taken to be 284.8 eV. The binding energies associated with the surface species were identified using the NIST XPS database [\[10\]](#page--1-0).

#### 2.3. Composites preparation

An epoxy–amine mixture was cast into a metallic mold in which a single  $Co_{62}$  $Fe_4B_{22.4}Si_{5.6}Nb_5$  wire was located in grooves with 0.5 mm in diameter. The composite was then cured at room temperature. The resultant composite sample was a cylinder with diameter of 5 mm and about 25 mm in length.

#### 2.4. GMI measurement

The GMI of the wire-composites was measured with a precision impedance analyzer (HP4294A). The external magnetic fields were applied parallel to the wire axis by changing the DC current in the range of 0.005–2.5 A through a Helmholtz coil. This adjustment produced a calibrated field up to the maximum/minimum values  $H_{\text{max}}$  = ±50 Oe. The percentage change of magneto-impedance (i.e. GMI ratio) is defined as:  $\Delta Z/Z(\%)$  = 100%  $\times$  [Z(H<sub>ext</sub>) – Z(H<sub>max</sub>)]/Z(H<sub>max</sub>). Where Z(H<sub>ext</sub>) and Z(H<sub>max</sub>) are the impedance values of the sample under an external and maximum magnetic field respectively.

#### 3. Results and discussions

To examine the chemical change before and after silane application, curve fitting was performed for N 1s and Si 2p spectra and the functionalities associated with each element were elucidated. High-resolution XPS spectra of nitrogen and silicon for wires without any chemical modification (untreated wires) are shown in Fig 2 with the corresponding band assignments and peak areas shown in Table 1. In the case of N1s (Fig. 2(a)), the spectra did not show any representative signal confirming that nitrogen was not present on the surface of the untreated wires. The Si 2p spectrum in Fig. 2 (b) shows a Si photopeak at 99 eV which corresponds to elemental Si  $(Si^0)$ .

The high-resolution spectra of nitrogen and silicon for silane treated wires are shown in [Fig. 3](#page--1-0) and the corresponding assignments and peak areas are given in [Table 2](#page--1-0). Compared with untreated wires, two N 1s signals at 399 eV and 404 eV were detected due to amine  $NH<sub>2</sub>$  groups and positively charged ammonium groups (NH<sup>+3</sup>), respectively  $[11]$ . These data are in accordance with the chemical structure of APTS and confirm the

#### Table 1

Peak assignments for the N 1s and Si 2p XPS spectra for untreated wires. FWHM stands for the full width at half maximum.

XPS spectra (UW)	Peak position (eV) Assignment Area (%)			<b>FWHM</b>
N <sub>1s</sub> Si 2p	- 99	Si <sup>0</sup>	100	$\overline{\phantom{0}}$ 1.53

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