Magnetic Properties and Applications of Glass-coated Ferromagnetic Microwires

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ABSTRACT A remarkable magnetic softness and giant magnetoimpedance (GMI) effect at GHz frequency range havebeen observed in glass-coated microwires subjected to appropriate postprocessing. Co-based microwires present higher GMI effect. Insulating and flexible glass-coating allows use of magnetically soft amorphous glass-coated microwires for stresses or temperature monitoring in smart composites using free space facility. Such composites with magnetic microwire inclusions can present tunable magnetic permittivity. We report on in-situ the evolution of the transmission and reflection parameters of the polymer containing magnetic microwire inclusions during the polymerization process. A remarkable change of the reflection and transmission in the range of 4-7 GHz upon the matrix polymerization is observed. Observed dependencies are discussed in terms of the effect of the temperature and stresses variation on magnetic properties of glass-coated microwires during the thermoset matrix polymerization. Obtained results are considered as a base for novel sensing technique allowing non-destructive and non-contact monitoring of the composites utilizing ferromagnetic glass-coated microwire inclusions with magnetic properties sensitive to tensile stress and temperature.

INDEX TERMS Magnetic microwires, magnetic softness, internal stresses, non-destructive control.

I. INTRODUCTION

Rapid melt quenching allows preparation of amorphous magnetic materials with an unusual combination of excellent soft magnetic properties together with good mechanical properties. This combination of properties makes them suitable for numerous industrial applications [1-6]. Magnetic softness of amorphous alloys is originated by the absence of the magnetocrystalline anisotropy and defects (dislocations, grain boundaries…) typical for crystalline magnets [1-3, 6-8]. Furthermore, the fabrication method involving rapid melt quenching is rather fast and cheap and above-mentioned magnetic softness can be realized without any complex post-processing treatments [3-5].

The development of novel applications of amorphous materials requires new functionalities, i.e. reduced dimensions, enhanced corrosion resistance or biocompatibility [6,8]. Therefore, great attention has been paid to development of alternative fabrication methods allowing preparation of amorphous materials at micro-nano scale involving melt quenching [6-8]

Glass-coated microwires prepared by the Taylor-Ulitovsky technique fit to most of aforementioned expectations: such magnetic microwires have micro-nanometric diameters (between 0.5 and 100 μm) covered by thin, insulating, biocompatible and flexible glass-coating and can present excellent magnetic softness or magnetic bistability [7-11].

These features of glass-coated microwires allow development of new exciting applications in various magnetic sensors, as well as in smart composites with tunable magnetic permittivity [12-19]. One more advantage of glass-coated microwires is their excellent mechanical properties [5].

Recently, the stress dependence of hysteresis loops and GMI effect are proposed for the mechanical stresses monitoring in fiber reinforced composites (FRC) containing microwires inclusions or using magnetoelastic sensors based on stress dependence of various magnetic properties [18, 20,21].

One of the common problems in the composite materials is the monitoring of the matrix polymerization as well as stresses monitoring. Usually the polymerization process monitoring is performed by different sensors like the pressure transducers and dielectric sensors [22]. However, employed sensors are not wireless [22]. Another proposed solution for non-destructive FRC monitoring is use of the piezoelectric fibers with diameters of 10 to 100 μm [23]. However, this solution requires plates to supply an electrical field, occupying a significant amount of space.

Among the promising solutions, addressing the problem of non-destructive FRC monitoring is a new sensing method involving free space microwave spectroscopy using inclusions of ferromagnetic microwire presenting the high frequency impedance quite sensitive to applied stress and magnetic field [17,18,24]. The aforementioned glass-coated microwires with metallic nucleus diameters of 0.2 - 100 μm presenting excellent mechanical and corrosive properties, are perfectly suited for the requirements of this technique, making it suitable for remote stresses and temperature monitoring in FRCs [17,18,24,25]. Recently several successful attempts for non-destructive FRC monitoring have been reported [26].

Magnetic softness of glass-coated microwires is substantially affected by the chemical composition of metallic nucleus: basically better magnetic softness and higher GMI effect are reported for Co-rich microwires with vanishing magnetostriction [8].

Accordingly, in this paper, we present our last results on studies of magnetic properties of glass-coated Co-rich microwires and on in-situ polymerization process monitoring of the FRC with Co-rich glass-coated microwires inclusions.

II. EXPERIMENTAL DETAILS AND SAMPLES

We studied glass-coated $Fe_{3.8}Co_{65.4}Ni_{1}B_{13.8}Si_{13}Mo_{1.35}C_{1.65}$ (metallic nucleus diameter, d=18.8 μm, total diameter, D=22.2 μm, ρ =d/D= 0.88) microwires with vanishing magnetostriction coefficients, λ_s, produced by Taylor-Ulitovsky method described elsewhere [7,8,11,12].

Hysteresis loops of studied microwires have been measured using fluxmetric method previously described in details elsewhere [27]. We represent the hysteresis loops as the dependence of normalized magnetization, M/M_0 (where M is the magnetic moment at given magnetic field and M_0 is the magnetic moment of the sample at the maximum magnetic field amplitude) versus magnetic field, H. The homogeneous axial magnetic field is produced by long solenoid (about 1 cm in diameter and 12 cm in length). All the measurements were performed at low AC magnetic field frequencies (100 Hz). The hysteresis loops measurements of individual microwires upon applied tensile stress and of the polymerizing matrix upon polymerization was a tool to demonstrate soft magnetic character of studied samples and to check what kind of stresses appear during the matrix polymerization.

The sample impedance, Z, in extended frequency range has been evaluated using the micro-strip sample holder from the reflection coefficient, S_{11}, obtained using Vector Network Analyzer (VNA), as previously described [28]. Such micro-strip holder with sample has been placed inside a long solenoid producing homogeneous magnetic field, H. The GMI ratio, ΔZ/Z, is obtained from $Z(H)$ dependence as:

$$\Delta Z/Z = [Z(H) - Z(H_{max})]/Z(H_{max}), \qquad (1)$$

where H and H_{max} are given and maximum applied fields respectively.

As expected from previously published papers on Co-rich microwires, as-prepared $Fe_{3.8}Co_{65.4}Ni_{1}B_{13.8}Si_{13}Mo_{1.35}C_{1.65}$ microwires present good magnetic softness (coercivity of about 10 A/m), high GMI effect (GMI ratio about 100%) (see Fig.1) and low negative magnetostriction coefficients, λ_s, of about -01x10^{-6} [29].

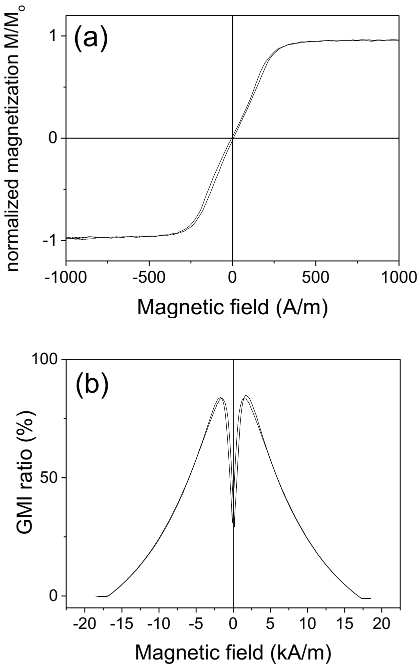

FIGURE 1. Hysteresis loop (a) and ΔZ/Z(H) dependence measured at 300 MHz of $Fe_{3.8}Co_{65.4}Ni_{1}B_{13.8}Si_{13}Mo_{1.35}C_{1.65}$mcirowire.

For the composite matrix we used a vinylester resin (DERAKANE 8084) resin, accelerated with Cobalt Octoate (0,3 pph) and catalyzed with Methyl Ethyl Ketona (MEK 60%, 1,5 pph). DERAKANE 8084 epoxy vinyl ester resin is an elastomer modified resin, designed to provide increased adhesive strength, superior abrasion resistance and enhanced mechanical stress, while providing greater toughness and elongation.

The liquid resin exhibits the following properties: 1.02 g/ml density (25°C), the dynamic viscosity ≈360 MPa (25°C) and about 40% styrene content. Detailed technical information of resin is provided in its technical data sheet (Document 1820 V5 F2, Language ES “draft”, © 2017 Ashland Inc.).

The composites with ordered glass coated amorphous wires embedded in the thermoset matrix polymerization have been prepared (Fig.2a).

For wireless measurements we used the free space measurement setup consisting of two broadband horn antennas (1-17 GHz) fixed at the anechoic chamber and a vector network analyzer, previously employed for the characterization of the composites with magnetic wire inclusions (see Fig.2b) [18,26]. Such setup, consisting in fixed horn antennas fixed in anechoic chamber allows allows to characterize the composite of 20 x 20 cm^2 with fixed incidence and fixed polarization placed inside the window (see Fig.2b). The other, completely different, setup with fixed horn antennas allows measuring the composites of 20 x 20 cm2 with fixed incidence and fixed polarization of placed inside the window.

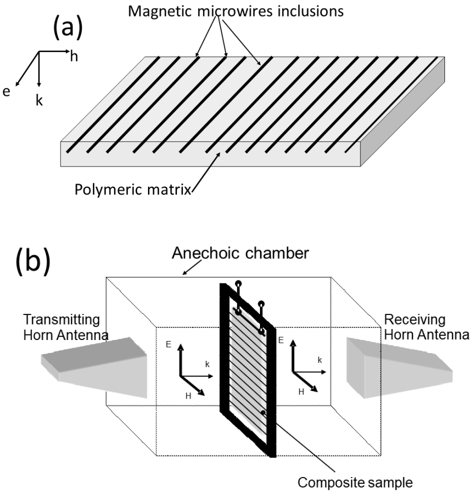

FIGURE 2. Sketch of a FRC with embedded microwires and (a) and of the free-space setup (b).

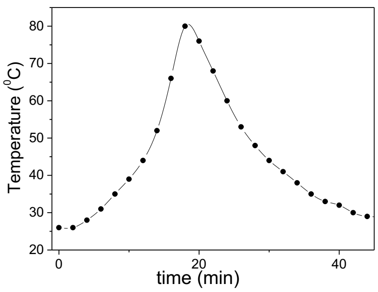

FIGURE 3 Evolution of temperature upon the matricx polymerization

During the matrix polymerization, various processes take place, which affect the magnetic properties and the GMI effect of microwire inclusions (Fig. 1a).

In order to understand the processes during the polymerization of the composite that can affect the microwires we have measured the temperature using a conventional thermocouple. Obtained temperature changes during the polymerization presented at temperature, T, versus time, t, are shown in Fig.3.

As can be observed from the Fig.3, the matrix polymerization produces a heating of the composite up to 80 ^{o}C. On the other hand, when a resin polymerizes, it shrinks, since the solid obtained will have a smaller volume than the monomer from which it started.

During the polymerization process of the resin, two processes occur simultaneously: volume shrinkage of about 8.2 % and resin heating (up to approximately 80 ^{o}C). The cured resin presents the following mechanical properties: a tensile strength of 76 MPa, a tensile modulus of 2.9 GPa, and a tensile elongation of 8-10%.

The value of applied stresses within the metallic nucleus, σ_m, has been evaluated as previously described elsewhere [27]:

$$\sigma_m = \quad K.P/KS_m+S_{gl}, \qquad (1)$$

where $k = E_2/E_1$, where E_1 and E_2 are the Young's moduli at room temperature for the metallic alloy and the glass respectively, P is the applied mechanical load, and S_m and $S_{gl}$are the cross sections of the metallic nucleus and glass coating respectively.

III. EXPERIMENTAL RESULTS AND DISCUSSION

As described above, we performed in-situ experiments on glass-coated microwires embedded in a polymerized composite placed inside the anechoic chamber during the resin polymerization. We measured the transmission parameter, *T*, and reflection parameter, *R*, of the polymerizing matrix containing the $Fe_{3.8}Co_{65.4}Ni_1B_{13.8}Si_{13}Mo_{1.35}C_{1.65}$ microwires inclusions using the free space setup. As shown in Fig.4, significant variation in the *T* parameter is observed in the frequency range, f, of 4-7 GHz during thermoset matrix polymerization. A non-monotonic variation of the *T*-parameter is observed (Fig.4a). Additionally, some changes in the R- parameter are also observed over a wide frequency range (Fig.4b).

Similarly to the T and R parameters, changes in the frequency dependencies of the T and R-phases are observed during the polymerization (see Fig.5).

As in the case of the T-parameter, a non-monotonic change in the T-phase is observed (see Fig.4a). Similarly, the R-parameter exhibits a monotonic change in the R-phase upon polymerization (see Fig.4b).

Although generally the distribution of the internal stresses arising during polymerization is generally non-homogeneous, the matrix generally shrinks during cooling [30]. Therefore, we can assume that the matrix shrinkage produces compressive stresses in magnetic nucleus of glass-coated microwires.

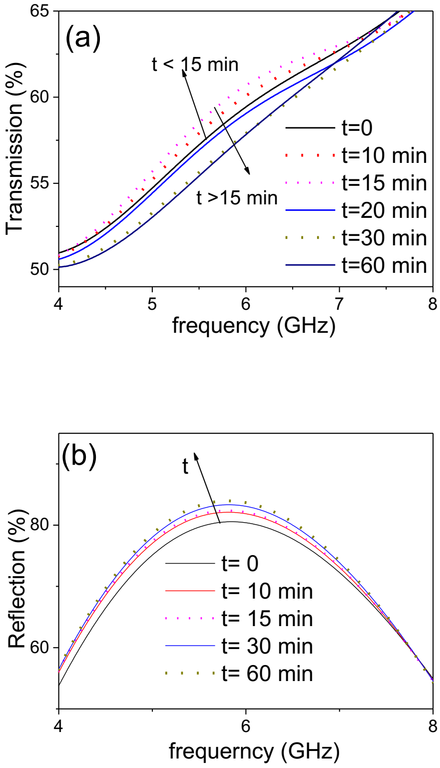

FIGURE 4. The Transmission, T (a) and reflection, R (b) parameters measured using free-space system during the

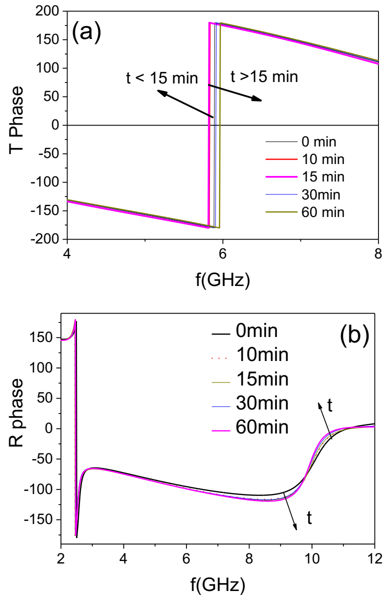

FIGURE 5. The T-phase (a) and R-phase (b) measured during the composite polymerization

Accordingly, the observed evolution of electromagnetic properties must be attributed to the combination of heating and mechanical stresses arising during the matrix polymerization.

The observed *T(f)* dependencies with are non-monotonic (see Fig.4a): there is an initial increase in T up to *t*=15 min (at *f* ≈4-7 GHz), followed by a decrease in T for *t*>15 min. Such evolution of *T*-parameter can be therefore associated to the heating and consequent cooling of the FRC.

In order to evaluate the effect of the matrix polymerization, we provided the additional experiments. Previously was demonstrated that the hysteresis loops of amorphous microwires are rather sensitive to applied stress [31,32]. Therefore, we have measured the hysteresis loops of studied samples under applied tensile stress (see Fig.6a). The hysteresis loops of studied Co-rich microwire maintain their linear shape under applied stress; however, a significant and nearly linear increase in the magnetic anisotropy field, H_k, upon tensile stress can be observed (see Fig.6a,b). On the other hand, the change of the hysteresis loops of the microwires embedded in the thermoset resin during its polymerization exhibits a different behavior: For a short polymerization time, *t*, the hysteresis loops maintain the inclined character (similar to the behavior of the individual wires under tensile stress) (see Fig.6c). However, as *t* increase, the hysteresis loops become almost rectangular. This behavior is the opposite to the effect of tensile stress shown in Fig.6a. This difference can also be appreciated in the $H_k(t)$ dependence shown in Fig.6d.

Therefore, one can assume that such behavior at large *t* must be attributed the matrix shrinkage, i.e. the effect of compressive stresses on magnetic properties of studied microwires.

The heating influence on the hysteresis loops can be understood by considering the origin of the internal stresses in glass-coated microwires. As discussed elsewhere, the main source of internal stresses in glass-coated microwires is the rapid solidification of the metallic alloy surrounded by the glass-coating, which has rather different thermal expansion coefficients [33-36]. Accordingly, as the difference in thermal expansion coefficients between the metal alloy and the glass coating decreases upon heating, internal stresses decrease. Recently such effect of heating on the hysteresis loop of Co-rich microwires with vanishing magnetostriction coefficient has been experimentally confirmed [37]. Similarly to our results, the transformation of the hysteresis loop from linear to rectangular was observed upon heating [37].

In contrast to the non-monotonic change in temperature during polymerization, we assume that the compressive stresses due to matrix shrinkage change monotonously. Therefore, we assume that the observed changes in Figs 4 and 5 are a result of both heating and matrix shrinkage during the polymerization.

Consequently, we have demonstrated that the evolution of the transmission and reflection parameters of composites with magnetic microwire inclusions using free space technique can be utilized to monitoring the polymerization composite. Moreover, this method proves to be useful for remote stress or temperature monitoring during polymerization, as well as for real-time non-destructive monitoring of stresses and temperature in composites during their operational use.

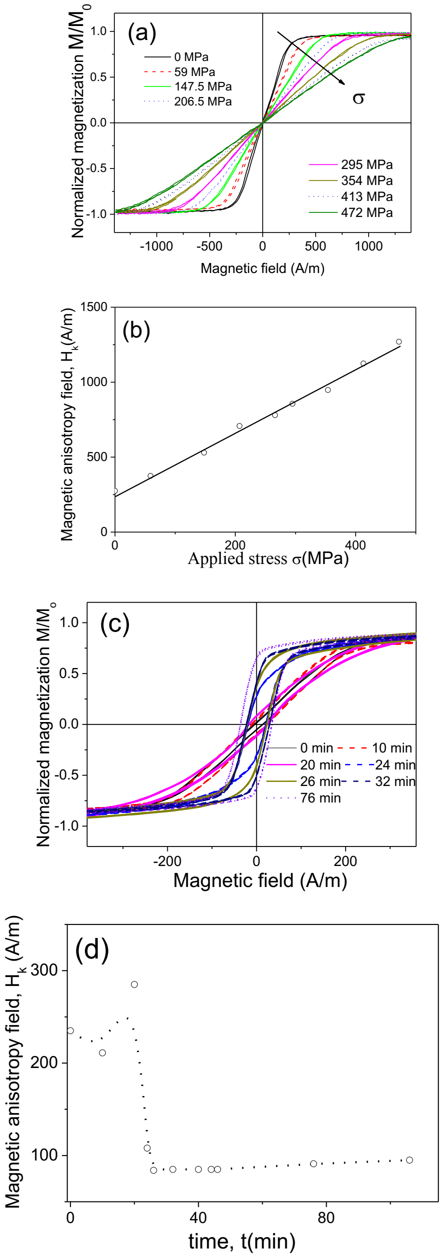

FIGURE 6. Effect of applied stress on hysteresis loops of studied microwires (a), $H_k(\sigma)$ dependence (b), evolution of the hysteresis loops of the microwires embedded in the thermoset resin during its polymerization (c) and Hk(t) dependence (d).

IV. CONCLUSION

We have carried out in-situ studies to investigate the the impact of matrix polymerization on the evolution of the transmission and reflection parameters in composites containing glass-coated microwires inclusions, using the free space technique.

Significant and non-monotonic changes in the T-parameter (within the 4-7 GHz) and *R*-parameter were observed during the polymerization of the composite. These experimental results have been correlated to the heating observed during the matrix polymerization and the matrix shrinkage that occurs as a result. The influence of heating and compressive stresses caused by matrix shrinkage on the hysteresis loops of glass-coated microwires was discussed.

These experimental findings are crucial for the development of a new sensing technique that enables non-destructive and non-contact monitoring of the composites with tunable magnetic permittivity sensitive to applied stress and temperature utilizing ferromagnetic glass-coated microwire inclusions with magnetic properties that are sensitive to applied stress and temperature variations.

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