New Contactless Method for Non-Destructive Composites Monitoring.

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Abstract: - We report on in-situ studies of the transmission parameter during the polymerization of the polymer containing magnetic microwire inclusions obtained using free space microwave spectroscopy. A substantial change in the transmission parameter in the range of 4-7 GHz is observed during the matrix polymerization. The observed dependencies are discussed considering the heating and matrix shrinkage during the polymerization process of the thermoset matrix on magnetic properties of glass-coated microwires. The observed experimental data can be used in numerous applications as a new method of non-destructive and contactless composites monitoring using glass-coated amorphous microwire inclusions with magnetic properties sensitive to applied stress and temperature.

Key-Words: - magnetic wire, amorphous materials, non-destructive control, magnetic softness, free space microwave spectroscopy, applied stress, shrinkage

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1 Introduction

An unusual combination of physical properties such as high magnetic permeability, giant magnetoimpedance, GMI, effect, magnetic bistability, Matteucci or Widemann effects,) and excellent mechanical properties (plasticity, flexibility, high tensile yield strength) and increased corrosion resistance makes amorphous wires suitable for numerous technological applications [1,2]. In addition, the fabrication technique of amorphous wires, essentially consisting of rapid quenching from the melt, is rather fast and inexpensive. Therefore, the aforementioned magnetic properties can be achieved without any sophisticated post-processing [1,3].

New applications of amorphous materials require new functionalities, such as size reduction, increased corrosion resistance or biocompatibility [1]. Accordingly, low dimensional amorphous materials prepared using melt quenching have been intensively studied during last years [1-3]. Glass-coated microwires with diameters, *d*, ($0.5\leq d \leq 100$ μm) fabricated by the Taylor-Ulitovsky method are recognized among the most promising modern functional magnetic materials [3-5]. Such microwires with amorphous structure and appropriate chemical composition usually present excellent soft magnetic properties. Additionally, the amorphous thin (usually below 10 μm) glass-coating is insulating, biocompatible and flexible. These features improve corrosion resistance and mechanical properties, and hence substantially extend the range of potential applications of such microwires. The magnetic properties of such microwires are rather sensitive to external stimuli, such as applied stress or temperature [3]. One of the promising applications of such microwires is the use of such microwires as inclusions in composite materials. Recently developed free space microwave spectroscopy of such composites with wire inclusions is suitable for contactless monitoring of smart composites [4].

This paper presents our latest results on in/situ evaluation of the stresses and heating arising during the matrix polymerization in fiber reinforced composites, FRCs, containing inclusions of amorphous Co-rich microwires.

2 Materials and Experimental techniques

We have studied the glass-coated $Co_{65.4}Fe_{3.8}Ni_1B_{13.8}Si_{13}Mo_{1.35}C_{1.65}$ with metallic nucleus diameter, d=18.8 μm, and total diameter, D=22.2 μm with vanishing magnetostriction coefficients, λ_s. The studied microwires have been manufactured using the Taylor-Ulitovsky technique, as previously described in details [3,5,6]. The composition of studied Co-rich microwires is selected considering vanishing λ_s ($\lambda_s \approx -10^{-7}$) [7].

The induction method was used to measure axial hysteresis loops. The experimental setup was designed to characterize thin soft magnetic microwires with reduced diameters [8]. The magnetization is represented as normalized magnetization, M/M_0. The GMI ratio, $\Delta Z/Z$, was obtained from the dependence of the sample impedance, Z, on magnetic field, H. Z-values were evaluated using a vector network analyzer from the reflection coefficient, S_{11}, as previously described [9].

The $\Delta Z/Z$ is obtained from [8-10]:

$$\Delta Z/Z=[Z(H)-Z(H_{max})]/Z(H_{max})\cdot 100 \quad (1)$$

being H_{max} –the maximum DC magnetic field, applied by the solenoid.

Studied microwires present good magnetic softness with coercivity, H_c, of about 10 A/m, high giant magnetoimpedance effect (maximum $\Delta Z/Z$-ratio above 100%) (see Fig.1a,b). Such hysteresis loops were measured under effect of tensile applied stresses and during the resin polymerization. A shrinkage of about 8.2 % occurs during the resin polymerization. However, apart of the matrix shrinkage considerable heating up to 80 ºC takes place.

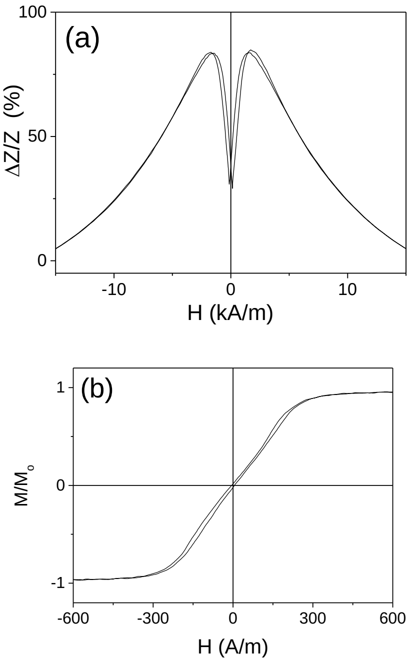

Fig. 1. $\Delta Z/Z(H)$ dependence measured at 300 MHz (a) and hysteresis loop (b) of studied microwire.

The free space measurements have been performed using the free space microwave spectroscopy. The experimental setup consists a vector network analyzer and two broadband horn antennas fixed at the anechoic chamber [11].

3 Experimental Results and Discussion

In order to evaluate the effect of the matrix shrinkage, we measured the influence of applied tensile stress on the hysteresis loops of studied samples and the effect of the resin polymerization on hysteresis loops (see Fig. 2). The hysteresis loops maintain their linear shape under applied tensile stress. While, during the polymerization of the resin, a different behavior of the in hysteresis loops of the microwires embedded in the resin was observed. Thus, at the beginning of polymerization (low polymerization time, t) the hysteresis loops retain the linear shape. This behavior of the hysteresis loops is quite similar to the behavior of the individual wires under effect of tensile stress (see Fig.2b). However, with t increasing, the hysteresis loops take an almost rectangular shape. This behavior is the opposite to the stress effect observed in Fig. 2a for individual microwire. Therefore, we must assume the matrix shrinkage effect that create the compressive stresses and effect of heating affecting the hysteresis loops.

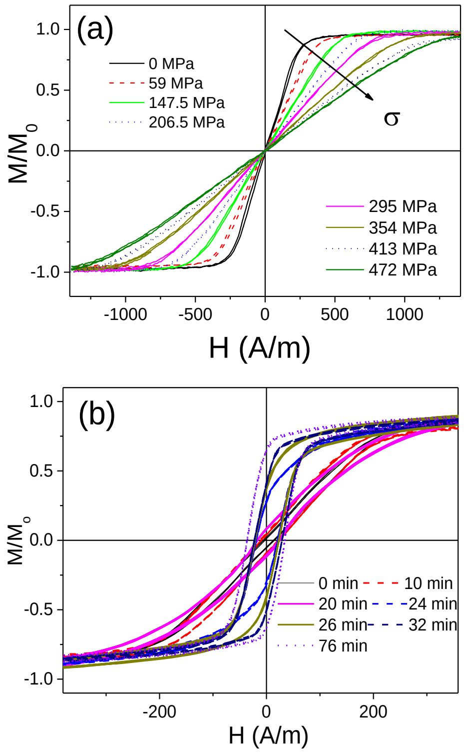

Fig. 2. Effect of applied stress on hysteresis loops of studied microwires (a), and evolution of the hysteresis loops of the microwires embedded in the thermoset resin during its polymerization (b).

Using the free space setup, we measured the transmission, T, parameter of the thermoset matrix with studied Co-rich glass-coated microwires embedded during the polymerization. As shown in Fig. 3, substantial change in the T-parameter at frequencies, *f*, between 4 and 7 GHz during thermoset matrix polymerization is observed. The observed changes in the *T*-parameter during polymerization are non-monotonic (Fig. 3).

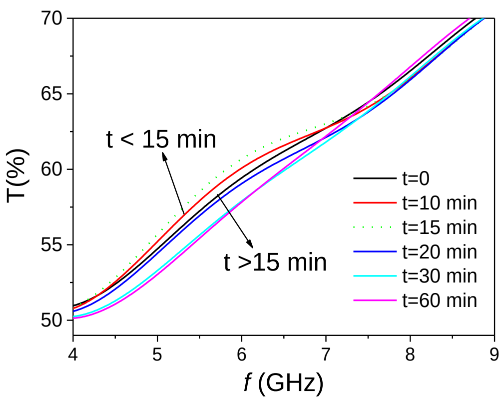

Fig. 3. The Transmission, T, parameter measured using free-space system during the composite polymerization.

Observed changes of electromagnetic properties must be attributed to two main phenomena arising during the resin polymerization: heating and matrix shrinkage.

Eventually the same technique can be used in many industries, such as civil engineering , car or aircraft industries for real life contactless and non-destructive stresses and temperature monitoring,

4 Conclusion

We studied in-situ the effect of matrix polymerization on the transmission parameters evolution in composites with inclusions of ferromagnetic microwires. We observed a significant change in the parameter T (at frequencies between 4 and 7 GHz) during polymerization of the composite using the free space microwave spectroscopy. The observed experimental results are attributed to the shrinkage of the matrix and heating during polymerization. Such changes correlate with the matrix polymerization on the hysteresis loops of magnetic microwires.

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References:

[1] Mohri, K.; Uchiyama, T.; Shen, L. P.; Cai, C. M.; Panina, L. V., Amorphous wire and CMOS IC-based sensitive micro-magnetic sensors (MI sensor and SI sensor) for intelligent measurements and controls, *J. Magn. Magn. Mater*., Vol. 249, 2002, pp. 351-356.

[2] Zhukov, A.; Vázquez, M.; Velázquez, J.; Garcia, C.; Valenzuela, R.; Ponomarev, B., Frequency dependence of coercivity in rapidly quenched amorphous materials, *J. Mat. Sci. Eng. A* Vol. 226-228, 1997, pp. 753-756

[3] Zhukov, A.; Corte-Leon, P.; Gonzalez-Legarreta, L.; Ipatov, M.; Blanco, J. M.; Gonzalez, A.; Zhukova, V., Advanced functional magnetic microwires for technological applications, *J. Phys. D: Appl. Phys*., Vol.55, 2022, pp. 253003.

[4] Zhukova, V.; Ipatov, M.; Corte-Leon, P.; Gonzalez, A.; García- Gómez, A.; Vallejo, F. J.;

Olaskoaga, P.; Garcia-Etxabe, R.; Malm, J.; Johansson, C.; Zhukov, A., Free Space Microwave Sensing of Carbon Fiber Composites With Ferromagnetic Microwire Inclusions, *IEEE Sens. Lett*. Vol. 8(1), 2024, pp. 2500104.

[5] Baranov, S. A.; Larin, V. S.; Torcunov, A. V., Technology, Preparation and Properties of the Cast Glass-Coated Magnetic Microwires, *Crystals* , Vol. 7, 2017, pp. 136.

[6] Chiriac, H.; Lupu, N.; Stoian, G.; Ababei, G.; Corodeanu, S.; Óvári, T-A., Ultrathin Nanocrystalline Magnetic Wires, *Crystals*, 7, 2017, pp. 48.

[7] Churyukanova, M.; Semenkova, V.; Kaloshkin, S.; Shuvaeva, E.; Gudoshnikov, S.; Zhukova, V.; Shchetinin, I.; Zhukov, A., Magnetostriction investigation of soft magnetic microwires, *Phys. Status Solidi A* , Vol. 213, 2016, pp. 363–367.

[8] Gonzalez-Legarreta, L.; Corte-Leon, P.; Zhukova, V.; Ipatov, M.; Blanco, J. M.; Gonzalez, J.; Zhukov, A., Optimization of magnetic properties and GMI effect of Thin Co-rich Microwires for GMI Microsensors, *Sensors*, Vol. 20, 2020, pp. 1558.

[9] Zhukov, A.; Ipatov, M.; Corte-Leon, P.; Blanco, J. M.; González-Legarreta, L.; Zhukova, V., Routes for Optimization of Giant Magnetoimpedance Effect in Magnetic Microwires, *IEEE Instrumentation & Measurement Magazine*, Vol. 23(1), 2020, pp. 56-63.

[10] Panina, L. V.; Mohri, K., Magneto-impedance effect in amorphous wires, *Appl. Phys. Lett.*, Vol. 65, 1994, pp. 1189-1191.

[11] Makhnovskiy, D.; Zhukov, A.; Zhukova, V.; Gonzalez, J., Tunable and self-sensing microwave composite materials incorporating ferromagnetic microwires, *Advances in Science and Technology*, Vol. 54, 2008, pp 201-210.

Contribution of Individual Authors to the Creation of a Scientific Article (Ghostwriting Policy)

The authors equally contributed in the present research, at all stages from the formulation of the problem to the final findings and solution.

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Conflict of Interest

The authors have no conflicts of interest to declare that are relevant to the content of this article.