

## Novel Sensing Technique for Non-destructive Composites Monitoring

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### Abstract

We observed evolution of the transmission and reflection parameters of the composites containing magnetic microwire inclusions during the composites matrix polymerization. A remarkable change of the reflection and transmission in the range of 4-7 GHz upon the matrix polymerization is observed. 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.

### 1. Introduction

Amorphous magnetic materials can present an unusual combination of excellent magnetic properties (e.g. high magnetic permeability, giant magnetoimpedance, GMI, effect, magnetic bistability, Matteucci and Widemann effects, ) and superior mechanical properties (plasticity, flexibility) making them suitable for numerous industrial applications [1]-[2]. Furthermore the preparation method involving rapid melt quenching is quite fast and cheap and above mentioned magnetic softness can be realized without any complex post-processing treatments [1].

The development of novel applications of amorphous materials requires new functionalities, i.e. reduced dimensions, enhanced corrosion resistance or biocompatibility [1]. Glass-coated microwires prepared using the Taylor-Ulitovsky method fit to most of aforementioned expectation: such magnetic microwires have micro-nanometric diameters (typically 0.5-50  $\mu\text{m}$ ) covered by thin, insulating, biocompatible and flexible glass-coating [1] and can present excellent magnetic softness or magnetic bistability [1],[3].

These features of glass-coated microwires allow development of new exciting applications in various magnetic sensors [1]-[3], as well as in smart composites with tunable magnetic permittivity [2]-[3]. A few years ago the stress dependence of the GMI effect is proposed for the mechanical stresses monitoring in fiber reinforced composites (FRC) containing microwires inclusions [2]-[4]. A novel sensing technique involving free space microwave

spectroscopy utilizing ferromagnetic microwire inclusions presenting the high frequency impedance is quite sensitive to tensile stress and magnetic field [4].

In this work we provide our recent results on study of the stresses arising during the polymerization of the matrix in FRCs on permittivity of the FRC with embedded microwire inclusions.

### 2. Materials and methods

We used glass-coated  $\text{Fe}_{3.8}\text{Co}_{65.4}\text{Ni}_{13.8}\text{Si}_{13}\text{Mo}_{1.35}\text{C}_{1.65}$  (metallic nucleus diameter,  $d=18.8 \mu\text{m}$ , total diameter,  $D=22.2 \mu\text{m}$ ,  $\rho =d/D= 0.88$ ) microwires with low negative magnetostriction coefficients,  $\lambda_s$ , prepared by Taylor-Ulitovsky technique described elsewhere [2]. The temperature during the polymerization process has been measured by a standard thermocouple.

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).

We used the free space measurement system previously described in details in [4]. The reflection ( $R$ ) and transmission ( $T$ ) coefficients were measure in free-space. The experimental set-up consists of a pair of broadband horn antennas (1-17 GHz) and a vector network analyzer. The composite was placed in 20 x 20  $\text{cm}^2$  window to avoid the edge effects. This window limits the applicable frequency range in 4-17 GHz. More detailed description of the free space systems is given in our previous publications [4]. The composites with ordered glass coated amorphous wires embedded in the thermoset matrix polymerization were prepared (Fig.1).

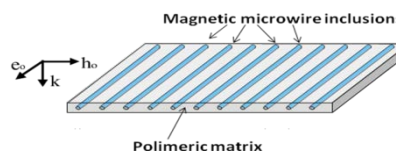


Figure 1 Sketch of a FRC with embedded microwires .

The polymerizing matrix provides external stimuli for the microwire inclusions (Fig. 1), which affects the magnetic properties and the GMI effect of microwires.

### 3. Results and discussion

During the polymerization process of the resin, volume shrinkage of about 8.2 % occurs and solid cured resin is obtained. The mechanical properties of the cured resin are the following: tensile strength of 76 MPa, tensile modulus of 2.9 GPa, and tensile elongation of 8-10%. However, apart of the matrix shrinkage considerable heating takes place. Therefore, in order to understand the processes during the polymerization of the composite that can affect the microwires we have measured the evaluation of temperature using a thermocouple. Obtained temperature changes during the polymerization represented at temperature,  $T$ , versus time,  $t$ , are shown in Fig.2.

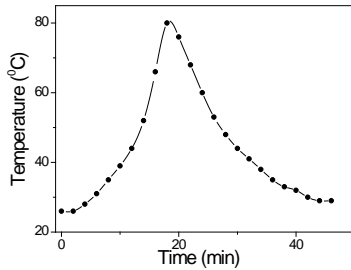


Figure 2. Evolution of temperature upon the polymerization.

As can be observed from the Fig.2, the matrix polymerization produces a heating of the composite up to 80 °C. As described above, we measured the transmission,  $T$ , and reflection,  $R$ , parameters of the composite containing Co-rich microwires ( $\text{Fe}_{3.8}\text{Co}_{65.4}\text{Ni}_1\text{B}_{13.8}\text{Si}_{13}\text{Mo}_{1.35}\text{C}_{1.65}$ ) using the free space system.

As can be appreciated from Fig.3, considerable variation of the  $T$ -parameter is observed in the range frequency,  $f$ , of 4-7 GHz upon thermoset matrix polymerization (Fig.3). A non-monotonic variation of  $T$ -parameter upon polymerization is observed (Fig.3a). Additionally, some changes of  $R$ -parameter are also observed in a wide  $f$ -range (Fig.3b).

Observed changes of electromagnetic properties can be related to two main phenomena arising during the composite matrix polymerization: heating and mechanical stresses. As we mentioned above, apart of the matrix heating, the polymerization is accompanied by change of density and shrinkage. Therefore we can assume that the matrix shrinkage produces compressive stresses in magnetic nucleus of glass-coated microwires. Observed  $T(f)$  dependencies with are non-monotonic: some increase of  $T$  observed up to  $t=15$  min (at  $f \approx 4-7$  GHz) followed by  $T$  decrease at  $t > 15$  min. Such evolution of  $T$ -parameter can be therefore associated to the heating and consequent cooling of the FRC.

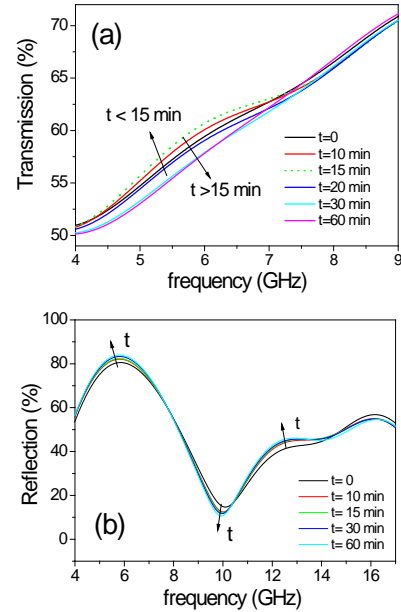


Figure 3. The Transmission,  $T$  (a) and reflection,  $R$  (b) parameters measured using free-space system during the composite polymerization.

### 4. Conclusions

We propose a novel sensing technique for non-destructive composites monitoring utilizing ferromagnetic microwire inclusions with magnetic properties sensitive to tensile stress and temperature. We have studied in-situ the impact of matrix polymerization on the evolution of the  $T$ - and  $R$ - parameters of the composites with microwire inclusions. We observed considerable variation of the  $T$ -parameter (in the range of 4-7 GHz) and  $R$ -parameter upon composite polymerization. Observed dependencies are discussed considering the matrix shrinkage during the polymerization and heating during the matrix polymerization and their influence on magnetic properties of glass-coated microwires.

### References

- [1] A. Zhukov, M. Ipatov and V. Zhukova, *Advances in Giant Magnetoimpedance of Materials*, Handbook of Magnetic Materials, ed. K.H.J. Buschow, Vol. 24, 2015, pp. 139-236 (chapter 2).
- [2] K. Mohri, T. Uchiyama, L. P. Shen, C. M. Cai, L. V. Panina, Amorphous wire and CMOS IC-based sensitive micro-magnetic sensors (MI sensor and SI sensor) for intelligent measurements and controls, *J. Magn. Magn. Mater.* 249: 351-356, 2002.
- [3] M.H. Phan, and H.X. Peng, Giant magnetoimpedance materials: fundamentals and applications, *Prog. Mater. Sci.*, **53**, 323-420 (2008).
- [4] D. Makhnovskiy, A. Zhukov, V. Zhukova, J. Gonzalez, Tunable and self-sensing microwave composite materials incorporating ferromagnetic microwires, *Advances in Science and Technology*, 54 (2008) pp. 201-210