



# In-situ microwave characterization of ferromagnetic microwires-filled polymer composites: A mini review

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## ARTICLE INFO

### Article history:

Received 16 June 2014

Received in revised form

15 October 2014

Accepted 16 October 2014

### Keywords:

Ferromagnetic microwires

Microwire composites

Microwave behavior

Tunable properties

## ABSTRACT

This review describes the emerging research area and relevant physics of polymer-based composites enabled by amorphous ferromagnetic microwires. Fruitful results ranging from their tunable magnetic field and mechanical stress properties and influences of direct current on their microwave behavior are displayed in addition to the brief analysis on the underlying physics. The multifunctionalities exhibited strongly imply a variety of potential applications such as structural health monitoring and high-performance sensors. This article underlines that the future challenge mainly lies in proper microwire tailoring in expectation of a better microwave performance of microwire composites.

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## 1. Introduction

Recent technological and industrial advances concerning magnetic sensors and devices require and anticipate materials with exceptional soft magnetic properties [1–3]. As a strong candidate, amorphous ferromagnetic microwires have been identified and intensively investigated for decades due to their excellent sensing properties. Their electromagnetic response can be conveniently tailored through designing the geometrical factors such as wire diameter and chemical composition. Moreover, they have been proved to have instant tunability in the millimeter wave frequencies towards external stimuli, e.g., external magnetic fields and mechanical stresses [4,5]. These merits, also evident in the giant magnetoimpedance (GMI) and giant stress-impedance (GSI) effects, provide a number of potential application areas such as microwave absorption, structural health monitoring, etc.[6]. To further enhance their magnetic properties, many efforts have been made in the microwire fabrication stage in addition to the subsequent physical and chemical wire treatments in the context of the needs of high-performance sensing devices [4]. However, this usually implied high costs, rendering such materials less suitable for industrial applications. Moreover, the Achilles heel of free standing microwires is their small dimensions (usually tens of microns) and the fragile nature of their mechanical fracture

behavior. Despite the overall tendency of magnetic device miniaturization, it remains an issue that how we tackle the above problems without compromising their excellent sensing properties.

Most recently, a smart strategy of embedding microwires into polymer-based composites has been devised by our group to multifunctionalize the resultant composites, which enables additional electromagnetic functionalities and preserves the mechanical performance of the polymer matrix at the same time [5–9]. Based on this perspective, fruitful experimental results and their underlying physics pertinent to ferromagnetic microwire composites have been systematically studied associated with other influential roles at play, for instance, the wire volume fraction [10], wire length [11] and polymer matrices [12]. From all these exciting studies, microwire composites are believed to be used in potential applications such as microwave devices and sensors in the future. It is therefore of significant interest to dispose a panorama on the microwire composites to summarize the on-going research on their fabrication, characterization and perspective applications from the standpoint of engineering. The aim of this review is to present some of our recent progress on the fabrication and microwave characterization of microwire composites with or without external field/stress/current.

The remainder of this review paper is organized as follows: we start by considering some fundamental aspects in Section 2 on the techniques for processing of microwires and their composites. Herein, different kinds of polymer composites were used as the matrices to offer mechanical constraint to microwires in order to

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study the particular external stimuli-dominated microwave behavior. Then in Section 3, we capitalize on recent advances on the microwave properties of microwire composites in presence of dc magnetic fields. A crossover field is identified in both Fe-based and Co-based wire composites. In addition, a double peak characteristic in the permittivity spectra of Co-based wire composites is discovered. Section 4 targets the stress effect of microwire composite, where the linearly increased permittivity dispersion is indicated in the Co-based composite while a contrary relation is obtained in its Fe-based counterpart. Section 5 is devoted to the influences of a direct current bias on the magnetoimpedance of polymer composites containing melt-extracted microwires. This current tunability can be exploited in specific engineered multifunctional microwire composites for active microwave devices. We conclude the whole review in Section 6 in addition to some remarks on the outlook.

## 2. Fabrication techniques of microwires and their composites and microwave characterization

Based on conventional techniques of fabricating amorphous alloys, a variety of fabrication techniques have been advanced for microwire processing, among which the modified Taylor–Ulitovskiy method is believed to be the most widely used approach [13]. Wires made by this technique usually have a metallic core with a glass coating closely attached. A detailed review discussing the fabrication and the GMI properties of microwires is listed here for interested readers [4]. In some of our studies, the microwires involved were processed by this technique [14–16]. The main advantages of such a technique are the repeatability of microwire properties in mass-production in a very economical way and the capability of fabricating continuous long pieces of microwire up to 10000 m [3,17].

On the other hand, alternative methods have been reported in the literature [4]. For instance, the melt-extraction (MET) technique has been recently adapted to yield high-quality and high-performance microwires with improved mechanical properties, as compared to other approaches such as in-water rotational spinning and glass-coated melt spinning [18, 19].

To aim at the microwire composites, different polymer materials should be considered to serve the particular investigation purposes as the candidate matrix material. Elastomers usually have much lower Young's modulus ( $\sim 2$  MPa) compared to other polymers and hence would develop appropriate strain when subjected to even smaller external force. Regarding the experimental details, one piece of transparent silicone rubber sheet was used as the matrix material with parallelly arranged continuous microwires laid on, followed by bonding with another rubber piece using silicone glue to yield the resultant wire composites (Fig. 1) [20,21]. Apart from the elastomers, epoxy is believed to be the most extensively studied material for all categories of composites and coatings for engineering applications due to its high mechanical properties. Thus epoxy can also be considered as the matrix material for the wire incorporation.

The room temperature S-parameter measurement was carried out in a modified frequency-domain spectroscopy. The  $S_{11}$  and  $S_{21}$  parameters of an asymmetric microstrip transmission line containing the sample were measured by a vector network analyzer (Agilent, model H8753ES) with SOLT (short, open, load, and thru) calibration in the frequency band of 0.3–6 GHz under the quasi-TEM transverse electromagnetic mode [22]. The electromagnetic measurements were conducted with the wave vector perpendicular to the sample width direction. Using the Nicolson–Ross procedure for the transformation of the load impedance by a transmission line,  $\epsilon = \epsilon' - j\epsilon''$  is determined by the transmission

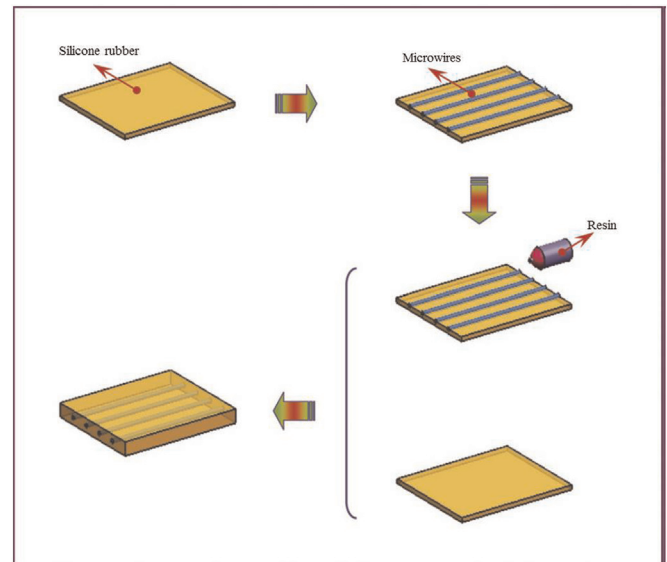


Fig. 1. Illustration of silicone rubber composites containing continuous microwires. (reprinted with permission from Ref. [5], copyright 2013 Elsevier).

$S_{21}$  and reflection  $S_{11}$  parameters [22]. The measurement of  $\epsilon$  under external dc fields from 0 to 1000 Oe was performed by placing the microstrip line in between the poles of an electromagnet. [22] Our procedure for measuring  $\epsilon$  of soft materials under application of a uniaxial stress along the sample longitudinal direction is reminiscent of the innovative technique described in Ref. [23]. To study the current effect, a dc current was applied by connecting a current source to wires. To obtain accurate measurements of  $\epsilon$ , it is particularly important to account for the residual air-gap between the sample and the line walls [23].

## 3. Magnetic field tunable properties

It is established that Fe-based and Co-based wires have significantly different domain structures and hence rather different static and dynamic electromagnetic performances. As such, the field sensitivity of Co-based wires can be easily saturated at much lower magnetic fields. By looking into the field dependence of epoxy composites containing  $\text{Fe}_{4.84}\text{Co}_{56.51}\text{B}_{14.16}\text{Si}_{11.41}\text{Cr}_{13.08}$  glass coated microwires, a larger magnetic field tunability (typically 150%) of the effective permittivity for the samples with microwire content of 0.026 vol% is observed, together with an independent relation between field tunability and wire volume fraction [10]. This shifts our later investigations to the effect of wire length.

Remarkably, similarities in the field dependence seen in the transmission and reflection coefficients for all samples investigated indicate that there is a crossover field at 300 Oe, i.e., the resonance frequency shifts to lower frequencies with fields increasing to 300 Oe then gradually shifts to higher frequencies when fields are larger than 300 Oe (Fig. 2). We have elucidated that this crossover field is due to the competition between GMI effect and ferromagnetic resonance (FMR) of the microwires. At lower fields, the GMI effect dominates the dipole resonance behavior resulting in the compromise of  $\epsilon$  and reflection coefficients with increasing field due to the improved surface impedance [10]. On the other hand, the FMR prevails at higher magnetic bias, therefore inducing enhanced eddy current and skin effect because of the drastically mitigated skin depth, eventually contributing to the frequency increase of resonance peak (Fig. 2(e)–(f)).

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