



## Short Communication

# Microwave stress monitoring using Co-rich amorphous microwire assessed by free space measurements

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## ABSTRACT

We provide new experimental results on studies on effect of applied stress on Reflection coefficient ( $S_{22}$  parameter) of Co-rich glass-coated ferromagnetic microwire measured using free space microwave spectroscopy. Studied Co-rich microwire with vanishing magnetostriction coefficient presents high Giant magnetoimpedance, GMI, effect associated with excellent soft magnetic properties. Tensile stress was applied through the mechanical load, attached to the single Co-rich microwire sample inside the anechoic chamber and the  $S_{22}$  parameter was measured at 2.45 GHz using broadband horn antennas and a vector network analyzer. Upon tensile stress (up to 225 MPa) we observed substantial change in the  $S_{22}$  parameter. The change in the  $S_{22}$  parameter correlates with the stress dependence of hysteresis loops. The experimentally discovered stress dependence of the reflection coefficient allows for contactless stresses and damage monitoring of in composites with microwire inclusions.

## 1. Introduction

Studies of amorphous wires have attracted substantial interest owing to their unique combination of physical properties, such as the magnetic bistability, the giant magnetoimpedance (GMI) effect, high mechanical and corrosion properties [1–6]. Aforementioned peculiar magnetic properties are essentially related to cylindrical symmetry of magnetic wires and disordered glassy-like structure. Accordingly, amorphous wires have been proposed for a variety of technical applications including sensorics, structural health monitoring or biomedical applications [4,7–12].

The GMI effect was discovered in 1935 [13] and intensively studied starting from 90-s [3,7–9]. The continued interest in the GMI effect is due to its unusually high magnetic field sensitivity (few hundred percent change in impedance,  $Z$ , under low external magnetic field,  $H$ , observed in amorphous wires). Therefore, the GMI effect is of great interest for the design of high performance and inexpensive magnetic field sensors and magnetometers [7–9,12]. Initially, the GMI effect was observed at MHz frequency band [3,7]. However, a substantial GMI effect at GHz frequencies has recently been reported in magnetic wires with

reduced diameters [14,15]. The origin of the GMI effect at GHz frequencies is discussed in terms of the magnetization rotation, precession of the magnetization vector in a high-frequency field and the ferromagnetic resonance (FMR) [16,17].

The GMI effect is commonly discussed in terms of the GMI ratio,  $\Delta Z/Z$ , defined as [3,4,7]:

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

where  $H_{max}$  – is the maximum applied DC magnetic field (typically below a few kA/m).

As shown elsewhere [4,18],  $\Delta Z/Z(H)$  dependence is linked to magnetic anisotropy of magnetic wires. Thus, a double-peak  $\Delta Z/Z(H)$  dependence is predicted and experimentally observed for magnetic wires with weak transverse magnetic anisotropy, while a decay of  $Z$  from  $H = 0$  is reported for magnetic wires with axial magnetic anisotropy [4,18]. Accordingly, maximum  $\Delta Z/Z$ -values,  $\Delta Z/Z_m$ , can be observed either at  $H = 0$  or at some  $H$ -value,  $H_m$ , linked to magnetic anisotropy field of studied magnetic wire [4,14,15,18].

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As recently demonstrated,  $\Delta Z/Z_m$ -value, exhibits a maximum at some optimum frequency,  $f_o$ , which depends on the wire geometry and on magnetic anisotropy [14,15]. For thinner magnetic wires generally higher  $f_o$ -values are observed: a decrease in the diameter,  $d$ , is associated with the increase in the  $f_o$ -values [14–16]. Accordingly for thin enough magnetic wires (with diameters,  $d \sim 10 \mu\text{m}$ )  $f_o$ -values are of the order of a few hundred MHz (200–600 MHz) [4,14–16], while for thick wires ( $d \sim 120 \mu\text{m}$ ) typically  $f_o \sim 1 \text{ MHz}$  [19,20].

Additionally, wire impedance,  $Z$ , is sensitive not only to magnetic field, but also to applied stress giving rise to stress impedance effect (SI) [21–23]. Finally, it has recently been reported that the GMI effect can be significantly influenced by temperature [24,25].

The magnetic wires with a GMI effect at GHz frequencies have been proposed for development of Free Space microwave sensing technique based on tunable effective permittivity at the GHz range in composites with magnetic wire inclusions [26,27]. The starting point for the concept of such smart composites is the tunability of the effective permittivity at the GHz range due to magnetic wire inclusions [28]. Finally, experimentally was demonstrated that the composite media with ferromagnetic wires exhibit microwave scattering strongly dependent on external stimuli (applied magnetic field and applied stress) [29,30].

The remarkable dependences of the effective permittivity, transmission or reflection parameters of such smart composites on external stimuli in the GHz range indicate that such composite with magnetic microwire inclusions can be useful for structural health monitoring and self-sensing applications [31–33]. One of the advantages of this technology is that proposed free space microwave spectroscopy allows remote monitoring of external stimuli, like stress or temperature. Such composites require magnetic wires with enhanced mechanical properties and high corrosion resistance. Therefore, glass-coated amorphous microwires produced by the so-called Taylor-Ulitovsky technique are considered as the most suitable materials for such smart composites development. Such microwires with amorphous structure present superior mechanical properties (high tensile yield, better plasticity and flexibility ...) together with better corrosion resistance owing to the presence of flexible and insulating glass-coating [4,34,35]. On the other hand, properly processed glass-coated microwires can present extremely soft magnetic properties with coercivity,  $H_c$ , about 2 A/m and GMI ratio up to about 700 % [4,36,37]. This combination of excellent soft magnetic properties and mechanical properties of amorphous materials is associated with their glass-like structure, characterized by the absence of crystalline structure and defects typical of crystalline materials (dislocations, grain boundaries, twins etc.) [4,34,35].

Accordingly, recently versatile properties of glass-coated microwires inclusions with high GMI effect stimulated development of novel applications in structural health monitoring including remote stresses or temperature monitoring in aircraft and car industries or civil engineering [38,39]. Certainly, contactless stress monitoring in carbon fiber composite materials for the aircraft industry is vital to the safety of modern aircrafts. The peculiarity of such composites with carbon fibers for aircraft industry is that such carbon fibres are conductive. Therefore, further developments were needed to separate the microwave response of magnetic microwires from that of conductive carbon fibres by using a low frequency modulated magnetic field [38]. For such applications high GMI effect and high stress sensitivity of the microwave response of magnetic microwires are essentially relevant. Recently, substantial optimization of the GMI effect in glass-coated microwires was reported using several types of post-processing including annealing under carefully selected conditions [4,37].

Although there are several reports on tunability of the effective permittivity, transmission or reflection parameters of such composites with magnetic wires inclusion at the GHz range on applied magnetic field, stress or heating [29,33], the influence of applied stress on microwave response of single microwire at GHz frequencies has not been experimentally studied.

Accordingly, in this paper, we present our recent experimental results on in-situ studies of applied stress in microwave response of single Co-rich microwire using free space technique.

## 2. Experimental details and samples

We studied  $\text{Co}_{72}\text{Fe}_4\text{B}_{13}\text{Si}_{11}$  glass-coated amorphous microwires with metallic nucleus diameters,  $d$ , of about 42  $\mu\text{m}$  and a total diameter,  $D$ , of about 49  $\mu\text{m}$  manufactured by the aforementioned Taylor-Ulitovsky method. The chemical composition was selected considering dependence of the magnetostriction coefficient,  $\lambda_s$ , on chemical composition in Co-Fe based amorphous alloys and vanishing  $\lambda_s$ -values ( $\lambda_s \approx -10^{-7}$ ) in Co-rich amorphous alloys [40,41]. Recently a high GMI effect was reported in such microwire [37]. Therefore this microwires is considered as a promising candidate for the contactless stress monitoring in carbon fiber composite materials with magnetic wire inclusions for the aircraft industry aiming safety and contactless monitoring of modern aircrafts [38].

Briefly, the fabrication method (commonly known as Taylor-Ulitovsky method) involves melting a metallic alloy ingot inside a glass (typically Duran or Pyrex) tube using a high-frequency inductor, forming the glass capillary from softened glass, drawing of such capillary filled with the molten metallic alloy and winding of the solidified glass-coated microwires onto a rotating bobbin [4,42,43]. It is worth noting that such fabrication technique was developed in the 60s [42,43]. Recently this technology was substantially updated: the last fabrication facility is provided with a feedback system suitable for controlling more precisely the microwire geometry ( $d$  and  $D$ -values) using a PC [4].

Axial hysteresis loops were measured by the fluxmetric method using a specially designed setup for studying soft magnetic microwires of reduced diameter. In this method, the electromotive force,  $\varepsilon$ , is induced in the pick-up coil with number of turns,  $N$ , due to a change in the magnetic flux,  $\phi$ , when the magnetization reversal of a sample with magnetization  $M$  occurs [44].

This  $\varepsilon$  is given as:

$$\varepsilon = -N \frac{d\phi}{dt} \quad (2)$$

To avoid the error related with precise evaluation of the magnetic nucleus diameter we represented the hysteresis loops as the normalized magnetization  $M/M_o$  (where  $M_o$  is the magnetic moment of the samples at maximum magnetic field amplitude,  $H_o$ ) versus  $H$ .

The GMI ratio,  $\Delta Z/Z$ , was defined using eq. (1) from experimentally measured reflection coefficient  $S_{11}$  using a vector network analyzer using the expression:

$$Z = Z_o \frac{(1 + S_{11})}{(1 - S_{11})} \quad (3)$$

where  $Z_o = 50 \text{ Ohm}$  is the characteristic impedance of the coaxial line.

Use of the specially designed sample holder with coaxial line connections allows to measure  $Z(H)$  dependencies up to GHz frequencies [15].

The morphology of the samples has been evaluated using Carl Zeiss - Axio Scope A1 microscope. As can be observed from Fig. 1a, the studied microwire presents perfectly cylindrical geometry of the metallic nucleus and rather uniform glass-coating. The amorphous state studied sample has been confirmed by a broad halo in the X-ray spectra obtained using X-ray diffraction (see Fig. 1b).

For wireless measurements of microwire response under stress at 2.45 GHz, we used the free space measurement system, consisting of two broadband horn antennas fixed to the anechoic chamber and a vector network analyzer, VNA, previously used for the composites characterization at free space (see Fig. 2a) [29,33,38]. We measured the scattering,  $S_{22}$ , parameter. The indexes 1 and 2 of the  $S$ -parameters defines

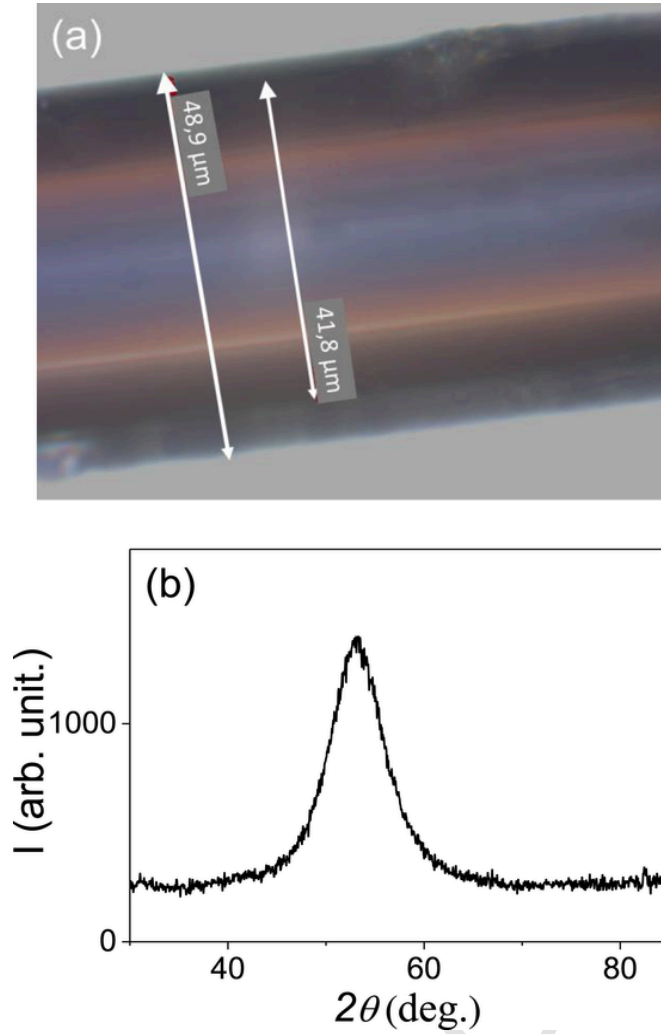


Fig. 1. Image obtained using optical microscope (a) and XRD pattern (b) of studied sample.

the ports to the VNA and also the excitation and detection direction of the electromagnetic wave to the studied sample. As recently proposed [38], the AC modulating magnetic field was applied parallel to the ferromagnetic microwires to modulate the impedance in the microwire. In the case of composites with carbon fibers use of such modulating magnetic field allows to distinguish the signal originated from magnetic microwires [38]. Similarly to our recent studies, we use a modulation magnetic field frequency,  $f$ , of 80 Hz [38].

During the hysteresis loop and free space measurements, a tensile stress was applied to the sample by means of a mechanical load hanged to one end of studied microwire (see Fig. 2b). The applied stresses value,  $\sigma$ , acting on the metallic nucleus has been evaluated considering different Young's moduli of the metallic alloy and the glass,  $E_1$  and  $E_2$  respectively, as was described earlier [33]:

$$\sigma = \frac{K \cdot P}{K \cdot S_m + S_{gl}}, \quad (4)$$

where  $k = E_2/E_1$ ,  $P$  - the applied mechanical load, and  $S_m$  and  $S_{gl}$  are the cross sections of the metallic nucleus and glass coating respectively.

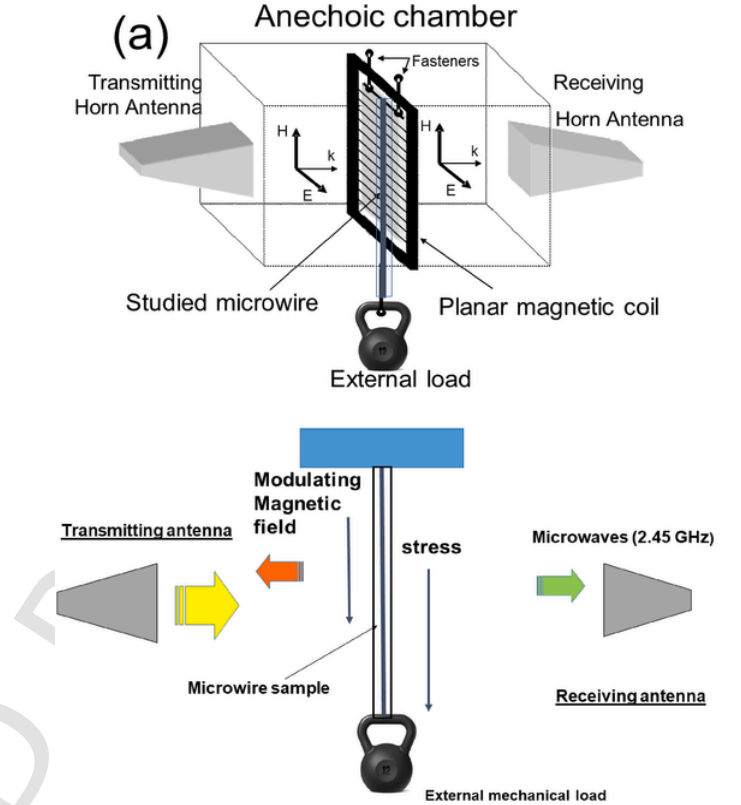


Fig. 2. Sketch of the free-space setup (a) and scheme of the experimental measurements at 2.45 GHz under applied stress (b).

### 3. Experimental results and discussion

The hysteresis loop of studied sample is provided in Fig. 3a. From Fig. 3a, rather soft magnetic properties of studied microwire with coercivity,  $H_c$ , about 21 A/m and magnetic anisotropy field,  $H_k$ , about 130 A/m are evidenced. Such magnetic properties are typical for Co-rich amorphous microwires with low negative  $\lambda_s$ -values.

As shown in Fig. 3b, the  $\Delta Z/Z(H)$  dependencies measured at difference frequencies,  $f$ , have a double-peak shapes with a maximum  $\Delta Z/Z$ -value,  $\Delta Z/Z_m$ , at certain magnetic field,  $H_m$ . Such double-peak  $\Delta Z/Z(H)$  dependencies are typical for magnetic wires with circumferential magnetic anisotropy [18]. Such features of the  $\Delta Z/Z(H)$  dependencies correlate with the hysteresis loop, particularly with low  $H_c$  and  $H_k$ -values. A rather high  $\Delta Z/Z_m$ -value up to  $\Delta Z/Z_m \sim 550\%$  is observed in studied sample.  $\Delta Z/Z_m$ -values are affected by  $f$ , being the highest at  $f \sim 300$  MHz. The  $\Delta Z/Z_m(f)$  dependencies obtained from  $\Delta Z/Z(H)$  dependencies measured in the frequency range up to 1 GHz are shown in Fig. 3c.

As described above, studied sample has been placed inside an anechoic chamber between the transmitting and receiving horn antennas. The applied stress was produced by a mechanical load, attached to the sample. Such mechanical load with the weight up to 250 g produced tensile stress,  $\sigma$ , up to 225 MPa, evaluated using eq. (4). While low frequency AC magnetic field (80 Hz) was applied by a planar coil placed inside the anechoic chamber. Using the VNA we measured the  $S_{22}$  parameters at 2.45 GHz at different  $\sigma$ -values.

The same experimental scheme was recently used to distinguish the microwave signals from magnetic microwires from that originated by conductive carbon fibers in carbon fibre composites with embedded magnetic microwires [38].

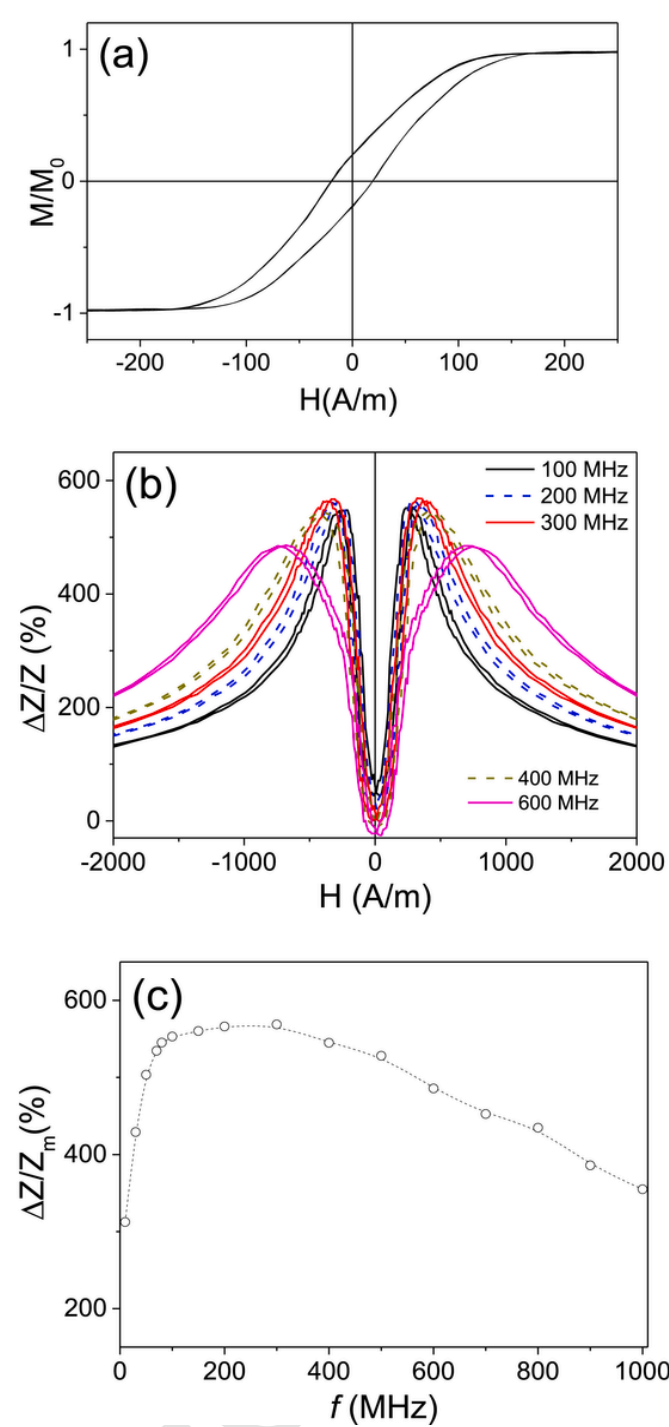


Fig. 3. Hysteresis loops (a),  $\Delta Z/Z(H)$  (b) and  $\Delta Z/Z_m(f)$  (c) dependencies of studied sample.

In Fig. 4a the dependence of  $S_{22}$  parameters measured at 2.45 GHz on frequency,  $f$ , is shown. In the case of using one single microwire, the reflected by a single microwire signal is very low, it is below the noise level in time domain. To detect such a low-intensity reflected wave ( $S_{22}$  or  $S_{11}$  parameter) from a microwire and separated it from parasitic background, we applied a low-frequency modulating magnetic field, acquired the reflection waveform at fixed frequency (2.45 GHz) and applied Fast Fourier Transform (FFT) to obtain the frequency spectrum. As observed, the  $S_{22}$  parameters present a maximums at  $f = 80n$

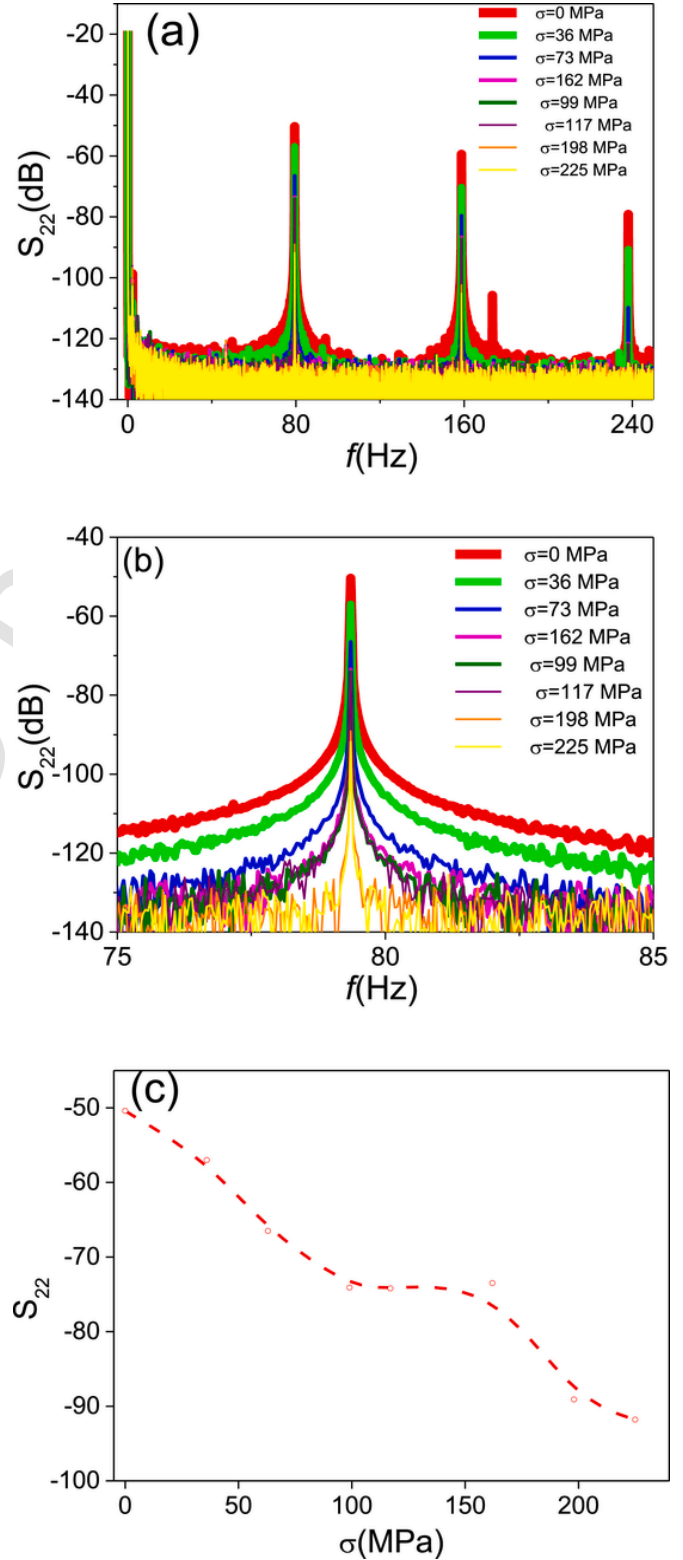


Fig. 4. Frequency Spectrum of  $S_{22}$  module (a), zoom (b) and effect of applied stress on amplitude of the 1st harmonic (fundamental, 80 Hz) of  $S_{22}$  at different  $\sigma$ -values (c).

( $n = 1,2,3 \dots$ ), reflecting effect of modulating low frequency field at  $f = 80$  Hz and 1,2,3,  $n$  harmonics.

Upon application of stress a substantial decay of the  $S_{22}$  parameter measured at  $f = 2,45$  GHz is observed (see Fig. 4a and b). The depen-

dence of  $S_{22}$  on  $\sigma$ , evaluated from Fig. 4b, is shown in Fig. 4c. From Fig. 4b and c, it is evident when the applied stress changes from 0 to 225 MPa,  $S_{22}$  changes on 40 dB (100 times) from  $-50$  to  $-90$  dB.

The observed substantial  $S_{22}(\sigma)$  dependence can be explained considering the change in magnetic permeability upon applied stress. To evaluate such effect we have measured the effect of applied stress on hysteresis loops of studied samples. Effect of applied tensile stress on hysteresis loop of studied microwire is provided in Fig. 5. As can be appreciated from Fig. 5, the hysteresis loops shape of the studied microwire retains its inclined shape. However, the slope of the linear region of the hysteresis loops decreases with increasing applied tensile stresses.

Stress dependence of magnetic permeability of magnetic materials has been extensively discussed since long time mostly using Preisach or Jiles-Atherton (JA) models considering stress dependence of hysteresis loops and magnetostriction coefficient [48–52]. Hysteresis loops and, in particular, magnetic permeability are influenced by a number of factors such as microstructural changes, applied and internal stresses, dislocation density, fatigue and creep damage, etc. [49–53]. In these models the domain wall motion, and domain rotation have been considered to explain stress dependence of magnetic properties [49–53]. However, despite the fact that at elevated frequencies domain walls are damped, a substantial GMI effect is observed up to GHz frequencies [14–16,38]. The origin of the GMI effect at elevated frequencies is attributed to precession of the magnetization vector in a high-frequency field and the ferromagnetic resonance (FMR) [16,17,51]. Considering the saturation magnetization,  $\mu_0 M_s$ , of studied microwire of about 0.8 T [1], we can evaluate the stress dependence of magnetic susceptibility,  $\chi$  (from relation  $\chi = \mu_0 M_s / H$ ) (see Fig. 5 inset). As can be appreciated from Fig. 5,  $\chi$  monotonically decrease upon applied tensile stress,  $\sigma$ .

The observed influence of the applied stress on  $S_{22}$  parameter must be linked to the dependence of microwave scattering of a single microwire on its magnetic permeability, also associated with the GMI effect [29,30,45–47]. Particularly, it was demonstrated that the microwave scattered intensity produced by a single microwire can be affected by the magnetic permeability [46]. The origin of the peaks on  $S_{22}$  ( $f$ ) dependencies observed at 80, 160 or 240 Hz must be attributed to the influence of applied modulating field on the permeability. While, the observed  $S_{22}(\sigma)$  dependence must be related to the dependence of magnetic susceptibility,  $\chi$ , on applied stress shown in the inset of Fig. 5.

On the other hand, the dependence of the hysteresis loops of Co-rich microwires with vanishing negative  $\lambda_s$  -values on both applied and internal stresses (modified by changing the thickness of the glass-coating)

has previously studied experimentally [4,48]. A change in the hysteresis loops consisting in increase in the magnetic anisotropy field,  $H_k$ , upon tensile or internal stress increasing was reported. A change in hysteresis loops has been reported, consisting of an increase in the magnetic anisotropy field,  $H_k$ , with an increase in applied tensile stress,  $\sigma$ , or internal stress,  $\sigma_i$ . In the case of the studied microwire we observed the same tendency (see Fig. 5). The origin of such  $H_k(\sigma)$  and  $H_k(\sigma_i)$  dependencies was recently discussed in terms of relationship between the  $\lambda_s$  and  $H_k$  [48]. Such relationship between the  $\lambda_s$  and  $H_k$  is given as [49]:

$$\lambda_s = \mu_0 M_s (H_k / 3\sigma) \quad (5)$$

where  $\mu_0 M_s$  is the saturation magnetization.

On the other hand, the  $\lambda_s$  -values of amorphous materials with low and vanishing  $\lambda_s$  -values is affected by the applied stresses,  $\sigma$ , [49]. Such  $\lambda_s(\sigma)$  dependence is described as [48,49]:

$$\lambda_{s,\sigma} = \lambda_{s,0} - B\sigma \quad (6)$$

being  $\lambda_{s,\sigma}$  -the magnetostriction coefficient under stress,  $\lambda_{s,0}$  -the zero-stress magnetostriction constant and  $B$  is a positive coefficient of order  $10^{-10}$  MPa. Accordingly, the  $\lambda_s(\sigma)$  dependence originates a further  $\lambda_s$  decrease in Co-rich microwires and, hence, an increase in  $H_k$  with  $\sigma$  increasing (as predicted from eq. (5)) [48].

The observed  $S_{22}(\sigma)$  dependence at microwave frequencies is potentially suitable for exciting applications in structural integrity and health monitoring related to wireless stresses monitoring and integrity in civil engineering, aircraft, automobile and construction industries. The possibility of wireless stresses and damage monitoring at reasonable cost is critical for the fundamental structural health monitoring and the control of the manufacturing and construction processes [54].

The obtained results show a high sensitivity of the reflection coefficient  $S_{22}$  to the applied stresses up to 0.2 dB/MPa in the applied stresses range from 0 to 200 MPa.

#### 4. Conclusions

We experimentally demonstrated the influence of applied stress on Reflection coefficient ( $S_{22}$  parameter) of Co-rich glass-coated ferromagnetic microwire measured using free space microwave spectroscopy. Studied Co-rich microwire with vanishing magnetostriction coefficient presents high Giant magnetoimpedance, GMI, effect associated with excellent soft magnetic properties. Tensile stress was applied through the mechanical load, attached to the single Co-rich microwire sample inside the anechoic chamber and the  $S_{22}$  parameter was measured at 2.45 GHz. Upon tensile stress (up to 225 MPa) we observed substantial change in the  $S_{22}$  parameter on 40 dB (100 times): from  $-50$  to  $-90$  dB. This effect correlated with the influence of applied stress on hysteresis loops. We experimentally observed an increase in the magnetic anisotropy field and a decrease in the magnetic susceptibility upon applied tensile stress in studied microwires. The experimentally discovered remarkable dependence of the reflection coefficient on applied stress opens up possibilities for contactless monitoring of stresses and damages in smart composites through magnetic microwires inclusions. Observed dependencies are discussed in terms of stress dependence of the magnetic susceptibility and stress dependence of the magnetostriction coefficient.

#### Uncited References

[53].

#### CRedit authorship contribution statement

**Valentina Zhukova:** Visualization, Validation, Resources, Methodology, Investigation, Funding acquisition, Formal analysis,

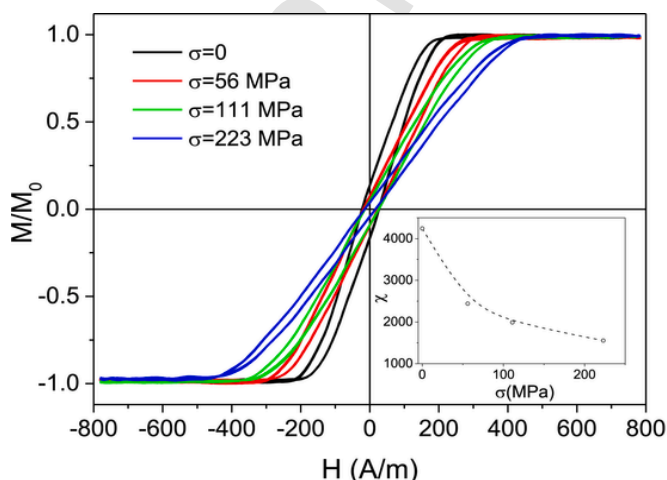


Fig. 5. Effect of applied stress on hysteresis loops of studied microwires. The influence of applied stress on magnetic susceptibility is shown in the inset.

Data curation. **Mihail Ipatov:** Writing – review & editing, Visualization, Validation, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Arcady Zhukov:** Writing – review & editing, Writing – original draft, Visualization, Supervision, Project administration, Investigation, Funding acquisition, Formal analysis, Conceptualization.

#### Data availability statement

The data that support the findings of this study are available on request from the corresponding author, AZ.

#### Declaration of generative AI in scientific writing

The authors declare that they have not used any type of generative artificial intelligence for the writing of this manuscript, nor for the creation of images, graphics, tables, or their corresponding captions.

#### Declaration of interests

Dear Editor, The authors of the manuscript “*Microwave stress monitoring using Co-rich amorphous microwire assessed by free space measurements*” by V. Zhukova, M.Ipatov and A. Zhukov declare no competing financial or/and non-financial interests.

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