Ferromagnetic microwires enabled multifunctional composite materials

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Abstract

The last two decades have witnessed increasing international interest in ferromagnetic microwires research. Recent attention has turned to the development of innovative materials and composites derived from these microwires, such as microwire polymer composites. Through incorporating an extremely small concentration of microwires (10^{-2} vol.%), the resultant composite exhibits a multitude of functionalities which are desirable for a range of technological applications. This article aims to provide a comprehensive review of current microwire composites research, from processing to structural and property evaluations with a focus on the multi-functionalities presented in these microwire composites. Starting with an introduction to multifunctional composites and the theories pertinent to the multiple functionalities of microwire composites, a detailed description of fabrication methods of microwire composites is given with a comparison of different processing techniques. Two fundamental effects, namely, giant magnetoimpedance (GMI) and giant stress-impedance (GSI) of microwire composites, are discussed in relation to monolithic microwires. Microwave tunable properties in the presence of a dc magnetic field, stress or temperature field are presented and analysed in depth. The ferromagnetic wire composites have also been shown to possess metamaterial characteristics and microwave absorption capability. A detailed discussion of the influence of composite architecture, such as local properties of microwires and topology of wire arrangements, on the performance of resultant composites, provides useful insights for an effective design of smart composites.
for specific engineering applications, such as structural health monitoring, stress sensing, invisible cloaking, microwave absorption and biomedical applications.

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1. Introduction

Materials we are dealing with today can be classified into structural materials and functional materials in terms of their functionalities. Conventionally, composite materials are designed to be used as
lightweight structural materials alternative to their metallic counterparts for structural functions only. In another aspect, functional materials have found a range of domestic and engineering applications with significant impacts. Naturally, an integration of these two categories of functionalities is much aspired for yielding the so-called multifunctional composites as against to the unifunctional ones, in that a wealth of applications can be explored ranging from aircraft wings to drugs dispensing [1]. A multifunctional composite must essentially meet the criteria of good mechanical performance and another one or more value-added physical or chemical functionalities, such as good electrical or thermal conductivity and peculiar electromagnetic behaviours.

To materialise the multifunctional concept, adding functional fillers is considered as one of, if not the only, the most effective routes. To name but a few examples, one of the most intensive research subjects is nanocomposites incorporating carbon nanotubes or nanoclays inside the polymer matrix to obtain favourable thermal and electrical conductivity for some specific applications such as de-icing, electromagnetic interference shielding and sensing applications [2,3]. Sufficient carbon black is capable of turning an insulating polymer into a conductive one when it is added into the polymer in a proper manner [4]. The bioactive fillers can make a dull matrix biologically sensible in vivo and greatly extend the scope of tissue engineering applications [5].

In the past two decades, the ferromagnetic microwires have been intensively studied due to their particular magnetic properties which are of strong application interest [6–10]. Consisting of a metallic core and glass coat, these microwires are primarily of CoFeSiB in composition (often doped with transition metal elements e.g., Mo, Cr, Nb), thereby giving very good ferromagnetic properties. They are structurally amorphous thanks to the existence of metalloid elements and the fast-cooling process, but magnetically anisotropic in part due to their specific domain structure varying with their composition and in part due to their geometry, such as microscale diameter and large aspect ratio. The most important properties of these wires are the so-called giant magnetoimpedance (GMI) and stress-impedance (SI) effects, by virtue of which they can be used as functional fillers to enable their resultant composites to have particular properties such as field/stress tunable properties, microwave absorption functionality whilst retaining the mechanical performance of the composite.

Among the extensive literature on the multifunctional composite topic, there have been some excellent monographs published: the book edited by Xanthos [11] gives a panorama of functional fillers and their composites and the latest review paper by Gibson [1] surveyed different classes of multifunctional composites for hot topics such as self-healing composites and energy harvesting composites. Interested readers are referred to these works and the references therein. In these publications, however, there is no mention of the microwires composites. Although Makhnovskiy and Panina [12] contributed an outstanding book chapter on ferromagnetic microwires-based composites, it is focused on the theoretical treatment of microwave tunable properties. In this context, it would be of significant scientific interest to have a dedicated monograph on the microwire composites to summarise the on-going research on their fabrication, characterisation and perspective applications from an engineering perspective. The aim of this work is exactly to fill this gap by presenting a systematic review of microwire composites from their fabrication to the characterisation of the structural and electromagnetic functionalities of the composites. With this understanding of the structure–property relationship, they can be best exploited to meet a range of specific applications.

The rest of the review is organised as follows: Section 2 introduces the fundamentals of multifunctional composite from fundamental concept to expected properties. The fundamentals and theories of GMI/GSI, microwave tunable properties, metamaterial behaviour and microwave absorption capacity are elucidated. Section 3 details the fabrication techniques of microwires and their composites. The GMI/GSI effects and mechanical properties of microwire composites are elaborated in Section 4. Section 5 is devoted to the study of microwave tunable properties including magnetic field tunable properties, stress tunable properties and temperature tunable properties. The high frequency behaviours of microwires for magnetic and stress sensing are also expounded there. Section 6 is targeted on the singular microwave absorption capacity of the microwire composite. Corresponding to the properties of microwires and their composites, potential applications are outlined in Section 7. The main conclusions of the whole review are summarised in Section 8 together with an outlook.
2. Fundamentals of multifunctional composite materials

2.1. What is a “multifunctional composite”?

A multifunctional composite conventionally refers to a composite material that, beyond the primary structural function, possesses other functionalities as well achieved by constituent components in an optimised structure [13,14]. Therefore, two points are underscored in this definition: (i) the composite must have multiple functions; and (ii) they are enabled by the constitutive materials. Such a logic may lead one to a picture of a complicated composite structure consisting of any specific materials according to the recipe of intended functionalities without regard to the compatibility of these materials. However, the architecture design of the composite will be a huge issue to tackle and it would also be difficult for one to predict the properties of the resultant composite from those of the constitutive materials insofar as the physical and chemical interactions between them are concerned. The brilliant idea of multifunctionalities is then tarnished by the high manufacture and maintenance cost. It appears to make this kind of composite remain at the conceptual level or far from ideal, as demonstrated by a wide spectrum of so-called multifunctional composites in the literature, inasmuch as they merely show a plus functionality at the expense, more often than not, of the mechanical performance. Strictly speaking, they are closer to multi-functional structures or systems rather than materials. To address these issues, the following should be realised: (i) an omnipotent functional filler is essential in that it will ensure the achievement of multifunctionalities and a relatively simple composite architecture. It does not necessarily mean all the functionalities must be exceptional. But the versatility so obtained warrants no increase of cost, weight and complexity [15]; and (ii) a homogeneous material is a great priority for structural integrity and implementation, which can be approached in two ways: chemical intimacy between the fillers and matrix and extremely low loading of fillers that permits physical perturbations only. It is therefore reasonable to consider these standards as implicit behind the term of multifunctionalities, based on which the concept of truly multifunctional composites is initiated. To approach this concept, a couple of aspects are of major concern: (i) the functional fillers as described above; and (ii) the topological arrangements of these fillers. From the vantage point of a bottom-up method, a perfect crystal (at absolutely zero degree and no impurities) is invoked with all atoms static at their own sites presenting superb properties. If such a nanostructure is upgraded to a higher hierarchy at the mesoscale, analogously, one will end up with a composite material with periodical arrangements of static fillers. Thus, it is logical to infer that the composite property can be precisely predicted, when it is subjected to an energy wave, via the interactions between the filling elements and the wave, and nearest neighbour-filler interactions provided that the single filler is well understood. In short, in realising the multifunctionalities, the filler answers the question of yes or no and the filler topology answers the question of how good it can be.

2.2. Fundamentals of multiple functionalities of microwire composites

In general, the multiple functionalities of composites are enabled by the functional fillers. The relevant theory associated with these functionalities should, therefore, be based on the physics pertinent to these fillers. In other words, although the local properties of fillers cannot decide the average composite behaviour, it may set a limitation for what could be achieved by their composites. Another crucial factor is the topological arrangement of these fillers as discussed above. Other factors include the matrix properties, interfacial properties, geometry of composites and so forth. Clearly, the research of multifunctional composites involves multidisciplinary science as against a unique physical phenomenon that can be explained within one theoretical framework. In this sense, rather than try to lay out universal principles for multifunctional composites, our discussion has been restricted to the microwire composites and narrowed down the relevant theories to specific targets. Since the functionalities

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1 Gibson [1] divided multifunctional composites into three types: (i) multiple structural functions, (ii) non-structural functions plus structural functions, (iii) both. This is a classification of multifunctionalities in a broad sense. In view of the recent development and the trend of multifunctional composites, our discussion has been limited to the type (ii), which we deem to be a judicious and strict definition.
of microwire composites stem from the unique properties of microwires and their arrangements, the following discussion will start with the GMI and GSI fundamentals of microwires, which lays the basis for the particular microwave properties of their composites. Subsequently, the fundamentals of microwave interactions with materials and characterisation methods will also be introduced as essential background knowledge. Following this, microwave properties of the composites including field tunable properties, metamaterial property and microwave absorption properties are addressed separately. It needs to be highlighted that these three classes of properties are actually interdependent, as all of them are characterised by the effective permittivity and/or permeability, i.e. the average dielectric/magnetic response of the material to electromagnetic irradiation.

2.2.1. Giant magnetoimpedance and stress-impedance effects

In this section, a brief introduction to GMI will be given, providing essential knowledge for the following discussion on the microwave tunable properties. GMI is defined as the large variation of magnetic impedance that happens in a soft magnetic conductor carrying a driving alternating current (ac) when it is subjected to a static external magnetic field [16]. The GMI effect is illustrated in Fig. 1, which shows the magnetic field dependence of impedance and GMI for an Fe-based ribbon at 500 kHz. In this example, the maximum of GMI arrives at 33% (Fig. 1b).

Quantitatively, the GMI can be expressed as

$$\Delta Z/Z(\%) = 100\% \times \frac{Z(H) - Z(H_{\text{max}})}{Z(H_{\text{max}})}$$

where $Z(H)$ and $Z(H_{\text{max}})$ are the impedance magnitudes of the microwire in the measured external magnetic field and maximum magnetic field to saturate the wire, respectively. The key points for this definition are discussed as follows:

1. Giant magnetoimpedance, as it manifests itself, has to present a large change in the total impedance of more than 100% in quantity [17]. Vázquez [18] reported on a pronounced GMI of more than 600%. In this case, it is quite promising for the application of magnetic sensors.

2. The GMI materials, whether wires, ribbons or films, are soft magnetic materials. The reasons for this are twofold: first, only magnetic materials are sensitive to the application of an external magnetic field; secondly, they are narrowed down to soft magnetic materials appropriate for applications as magnetic sensors and actuators requiring essentially their low coercivity, small hysteresis loss and high initial permeability that ensure an easy magnetisation process. Note that the magnitudes of these properties described above lead to a narrow hysteresis loop.

3. The alternating current plays an important part in the GMI effect. According to $I_{ac} = I_0 e^{j\omega t}$, its amplitude and frequency are significant parameters to influence the GMI as a result of affecting the sign and magnitude of alternative current. Numerous reports on the influence on GMI of these two

---

**Fig. 1.** (a) The impedance ($Z$) and (b) GMI ratio [i.e., $\Delta Z/Z(\%)$] change as a function of external field ($H$) for a Fe$_{71}$Al$_{14}$Si$_{14}$B$_{8.5}$Cu$_{1}$Nb$_{3.5}$ nanocrystalline ribbon. Reprinted with permission from [7], copyright 2008 Elsevier.
agents have been published as regards its mechanism right down to the alteration of samples’ domain structures, which are determined by the magnetic anisotropy associated with the alternative current. It is highlighted that the orientation of the current should be perpendicular to the anisotropy direction for the purpose of producing the best GMI effect. In microwires, for instance, the current and the external magnetic field should be in the same direction along the axis so that the circular field it creates can interact with the magnetic structure of the wire and the external magnetic field, yielding a GMI effect based on the dynamics of magnetisation consequently.

4. The last but not least key point concerns the external magnetic field. Its function is realised by its influence on the magnetisation process of the materials, therefore its orientation should be chosen to make the best of this function with respect to the magnetic structures of the materials. It has been proved that longitudinal giant magnetoimpedance (LGMI) gave rise to the largest magnetoimpedance ratio (MIR) and field sensitivity compared with the transverse GMI (TGMI) and perpendicular GMI (PGMI), for example, in the case of ribbon [19]. In the LGMI and TGMI measurements, the dc magnetic field is applied in the ribbon plane, parallel and perpendicular, respectively, to the probe current. In the PGMI geometry, the dc field is perpendicular to the sample plane and the probe current. Also, the magnitude of the external magnetic field should be of the order of a few Oersteds (Oe)² because the essence of GMI is that it occurs at relative low frequencies, and it is sensitive to a variation of external field of only few Oe, i.e., high field sensitivity, which is desirable for applications of magnetic sensors and transformers.

Now let us dig a bit deeper into the GMI effect in physics terms. The GMI effect has an origin based on the theory of classical electrodynamics, unlike giant magnetoresistance (GMR) that cannot be explained without appealing to the quantum mechanics [20,21]. A theoretical explanation through the deduction originated from the Maxwell equations and Landau-Lifshitz equation is given here.

Basically, magnetoimpedance can be approached by solving Maxwell equations

\[
\text{curl} \mathbf{H} = \mathbf{J}, \quad (2.2a)
\]
\[
\text{curl} \mathbf{J} = -\frac{\mu}{\rho} (\mathbf{H} + \mathbf{M}), \quad (2.2b)
\]

and

\[
\text{div} (\mathbf{H} + \mathbf{M}) = 0, \quad (2.2c)
\]

which can be reduced to

\[
\nabla^2 \mathbf{H} - \frac{\mu_0}{\rho} \mathbf{H} = \frac{\mu_0}{\rho} \mathbf{M} - \text{grad div} \mathbf{M}, \quad (2.3)
\]

where \(\mu_0\) and \(\mu\) is the vacuum permeability and material permeability, respectively; \(\rho\) stands for electrical resistivity. and the Landau–Lifshitz equation simultaneously

\[
\mathbf{M} = \gamma \mathbf{M} \times \mathbf{H}_{\text{eff}} - \frac{\alpha}{M_s} \mathbf{M} \times \mathbf{M} - \frac{1}{\tau} (\mathbf{M}_0 - \mathbf{M}), \quad (2.4)
\]

where \(\gamma\) denotes gyromagnetic ratio, \(M_s\) the saturation magnetisation, \(M_0\) the static magnetisation, \(H_{\text{eff}}\) the effective magnetic field and \(\alpha\) the damping parameter. The impedance can be calculated. But due to the difficulty in solving both equations simultaneously, Eq. (2.4) is often dismissed as far as a linear relationship between the inductance and magnetic field is concerned. In this way, by solving Eq. (2.3), impedance for the wire can be resolved as follows [16,22]:

\[
Z = R_{dc} k J_0/(ka)/2J_1/(ka), \quad (2.5)
\]

In Eq. (2.5), \(R_{dc}\) is the electrical resistance, \(k = (1 + j)/\delta_m\), \(a\) is the radius of the wire, and \(J_0\) and \(J_1\) are the Bessel functions of order 0 and 1, respectively.

² 1Oe = 80 A/m, we try to restrict the use of units to SI system, yet the prevalence of mks units in majority of publications on electromagnetic materials hinders us to do so. As such, both units will be regarded in the present paper. Unfamiliar readers are advised to refer to textbooks on electromagnetism for unit conversion.
The penetration depth is a function of the effective permeability, resistivity and frequency as described in the following equation for wire-shaped ferromagnetic materials [7]:

$$\delta_m = \frac{c}{\sqrt{4\pi^2 f \sigma \mu_{eff}}}.$$  \hspace{1cm} (2.6)

From Eqs. (2.5) and (2.6), a convincing explanation can be reached that a variation of permeability caused by the static external field gives rise to changes in the skin depth and finally in impedance. Obviously, in order to obtain a large MI ratio (MIR), a small resistivity and large permeability are necessary. On the other hand, reduction of resistivity and/or increase of permeability do not necessarily result in an improvement of GMI in that the dominating factor that determines the MIR is circumstantial. That is why strikingly different results and simulation models are often given regarding the relationship between the same two parameters and a reason as well for further research on the GMI effect with respect to exploring new materials and theories [23].

Research on the mechanism of GMI is also done from another perspective, whereby the real part and imaginary part of complex impedance are investigated separately according to the mathematical expression of complex impedance $Z = R + Xi$. It has been found that the extent of influence on the resistance (real part) [24] and the reactance (imaginary part) [25] varies with the frequency, but the influences on both do exist and amount to the influence on the total impedance. It is worth highlighting that Yelon et al. [26–29] have successfully established the connection between GMI and FMR at high microwave frequency and argued that they are rigorously equivalent, which proves to be instrumental for design of microwire arrays in order to manipulate the so-called GMI resonance [30] or FMR in the microwave frequency range of specific application interest [30]. For more detailed discussion on the GMI theory, one can refer to previous reviews and the references therein [7,31].

2.2.2. Microwave interactions with materials

Before proceeding directly to the electromagnetic properties of composites, one has to understand the basics of microwave interactions with different kinds of materials. This section provides the preparing knowledge for one to understand the non-linear field effect and specific dispersion properties as well as the reflection and transmission features of the microwire composites.

The responses of materials to the incident microwave, in general, are characterised by two fundamental parameters, namely, (relative) complex permittivity $\varepsilon = \varepsilon' - j\varepsilon''$ and relative complex magnetic
permeability $\mu = \mu' - j\mu''$; Permittivity is to measure how much resistance is encountered when an electric field is formed in a vacuum. It represents the ability of a material to polarise or 'permit' in the electric field. Likewise, the permeability is to measure the ability of a material to magnetise or 'permeate' in the magnetic field. 'Relative' refers to the normalisation by the vacuum permittivity $(8.85 \times 10^{-12} \text{ F/m})$ or permeability $4\pi \times 10^{-7} \text{ H/m}$. $\varepsilon''$ and $\mu''$ represent the dielectric and magnetic losses, respectively. They are close to zero at zero frequency or infinite frequency. The root reason for the emergence of dielectric/magnetic loss is the lag of response of materials to the external field.

$$\varepsilon'' = \varepsilon''_p + \frac{\sigma_{dc}}{\varepsilon_0 \omega},$$

(2.7)

where $\varepsilon''_p$ denotes polarisation or relaxation loss, $\sigma_{dc}$ is dc conductivity. The dielectric loss is mainly contributed by Ohmic (conduction) loss and/or polarisation (relaxation loss). It follows that, for conductors, higher conductivity tends to result in a larger energy dissipation. On the other hand, larger conductivity indicates a stronger skin effect according to Eq. (2.6) and the matter would be more reflective. Magnetic loss mainly results from the ferromagnetic resonance at high frequency, among other mechanisms such as hysteresis loss, eddy current loss and the magnetic after-effect for low frequency. They can to some extent indicate the microwave absorption capacity of a material, however, to get accurate information, they should be considered together with the loss tangent (or loss factor), which are defined as $\tan\delta_\varepsilon = \varepsilon''/\varepsilon'$ (dielectric loss tangent) and $\tan\delta_M = \mu''/\mu'$ (magnetic loss tangent).

When the electromagnetic wave is incident on a material, the microwave energy is dissipated as heat through the interactions of electromagnetic force fields with the material’s molecular and electronic structure, i.e., through damping forces on the polarised atoms and molecules and through finite conductivity. Based on the complex Poynting vector theorem, the absorbed power of the EM flowing into a volume $V$ through a closed surface $S$ is given as [32]:

$$P_{\text{diss}} = \text{Re} \left\{ \frac{1}{2} \oint_S (\mathbf{E} \times \mathbf{H}^*) \cdot dS \right\} = \frac{\omega}{4} \iiint_V \varepsilon_0 \varepsilon'' |\mathbf{E}|^2 + \mu_0 \mu'' |\mathbf{H}|^2 \, dV. \quad (2.8)$$

The characterisation of electromagnetic parameters at gigahertz frequency can be measured through different means. Here we introduce two methods that are dedicated to study the external field effects on the microwave properties.
2.2.2.1. Free space measurement system. Investigation of the microwave tunable properties of composites was carried out in the free space using the standard calibration technique named Through-Reflection-Line (TRL) as a well-received testing method for dielectric materials and any non-coaxial measurements of S-parameters [33–37]. The schematic graph of the measuring system employed for our experiments is shown in Fig. 2 [38]. To neutralise the influences of the noises on the scattering, the walls of the compact anechoic chamber are made of plywood and covered on the inside by a microwave absorber and a Network Analyser with the time domain option is employed [34,39,40]. Antennas are connected to the ports of a HP8720ES Spectrum Network Analyser through RG402 cables with the Subminiature Version A (SMA) connectors (see Fig. 3) [41]. The detailed features of antennas are as follows: (1) length: 887 mm; (2) aperture: 351 × 265 mm; (3) frequency range: 0.85–17.44 GHz; (4) standing-wave ratio (SWR) < 1.7; (5) effective area > 150 mm² in the range of 0.85–15 GHz.

![Free space measurement system](image)

**Fig. 4.** The construction of a planar coil for laboratory investigations. The composite sample is placed in between two coil layers. The coil becomes “invisible” for a plane-polarised wave with the electrical vector directed transversely to the coil turns, as shown in Fig. 2. To provide the uniform magnetic field inside the coil, the distance between the coil layers must be equal to the interturn distance d, and these layers must be shifted transversely across one another over steps of d/2. Reprinted with permission from [366].

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![Photographs of instrumentation for microwave measurements](image)

**Fig. 5.** Photographs of instrumentation for microwave measurements. Reprinted with permission from [277], copyright 2005 IOP.
The distance between antennas is controllable with the mobile front walls of the chamber where the antennas are fastened to meet the requirement of preliminary TRL calibration. The frame’s function is to guarantee the uniform heating or magnetic field along the sample surface when the microwave response depends on the external stimuli including field, stress and temperature. A current bus or a planar coil was used for the same reason and also makes the composites easily tunable by a weak magnetic field. It is highlighted that the parallel current wires must be oriented perpendicularly to the vector of the electrical field in the plane-polarised accidental electromagnetic wave in order to allow for nothing but the interaction between the composites and electromagnetic wave. Note that the design of the switch makes the most of the analyser, between the non-contact microwave test in the anechoic chamber and the contact test on magnetic wires in the measuring cell (Fig. 3) [38,41].

If a very high magnetic field is required, the planar coil is preferred to the current bus because all turns in a coil are connected in series, so passing the total current. The construction of the frame out of planar coil is shown in Fig. 4. The sample is placed between the two coil layers.

The complex permittivity can be computed from the \( S \)-parameters collected from the measurement. \( S \)-parameters \( S_{11} \) and \( S_{21} \) can be expressed via reflection coefficient \( \Gamma \) and transmission coefficient \( T \) as [33]:

\[
S_{11} = \frac{\Gamma(1 - T^2)}{1 - T^2 \Gamma^2},
\]

(2.9a)

and

\[
S_{21} = \frac{T(1 - \Gamma^2)}{1 - T^2 \Gamma^2}.
\]

(2.9b)

\( \Gamma \) and \( T \) can then be calculated by:

\[
\Gamma = \frac{(Z_{\text{in}} - 1)}{(Z_{\text{in}} + 1)},
\]

(2.10a)

and

\[
Z_{\text{in}} = (1/\sqrt{\varepsilon^*})T = e^{-\gamma d}, \quad \gamma = (2\pi/\lambda_0)(\varepsilon^*)^{1/2},
\]

(2.10b)

where \( \lambda_0 \) is the wavelength in the free space and \( d \) is the thickness of the sample. The complex permittivity is given by:

\[
\varepsilon^* = \frac{\gamma}{\gamma_0} \left( 1 - \frac{1}{1 + \Gamma} \right).
\]

(2.11)

2.2.3. Field and stress tunable properties

The microwave tunable properties of a microwire composite, by nature, are the response of effective permittivity to the electromagnetic wave through the surface impedance. Therefore, to explain this phenomenon, one needs to understand the basics of wave interactions with the materials and the effective medium approaches to characterise heterogeneous composites.
2.2.3.1. Effective permittivity. The effective permittivity should be treated differently in two types of composites. In composites containing short wires, the Lorentz model proves to be effective, whereas the Drude model is applicable to composites containing long wires. The Lorentz model is first considered to be applicable to all insulator materials. Along the inclusion length the current with a linear density \( j(x) \) is induced by the local electrical field \( \varepsilon_{loc} \exp(-i\omega t) \). Using the continuity equation and integration by parts with boundary conditions the electric dipole moment \( D \) can be calculated:

\[
D = i \omega \int_{l/2}^{l/2} j(x) \, dx \Rightarrow \alpha = \frac{D}{\varepsilon_{loc}},
\]

where \( l \) is the length of the wire and \( V \) is the inclusion volume. And within the frame of this approach the dielectric polarisability \( \alpha \) of the inclusion can also be calculated [42]:

\[
\alpha(\omega) = \sum_n \left( \frac{A_n}{(\omega_{res,n}^2 - \omega^2) - i\Gamma_n \omega} \right),
\]

where \( \omega_{res,n} \) is the angular resonance frequency, \( A_n \) are amplitude constants and \( \Gamma_n \) are the dumping parameters. \( A_i \), whereof contributes most to the polarisability corresponding to the lowest frequency. \( \Gamma_n \) is considerably influenced by the resistive magnetic losses [43] and it presents a strong dependence on the external magnetic field or stress in the vicinity of an antenna resonance in certain conditions. An experimental proof of this equation has been provided in Refs. [44,45].

The bulk polarisation of the composites can be expressed as:

\[
\mathbf{P} = (\varepsilon_{loc}) \mathbf{p} \alpha = \varepsilon_0 \varepsilon_{eff},
\]

where \( \varepsilon_{loc} \) is the averaged local field, \( \mathbf{p} \) is the volume concentration of the inclusions, \( \varepsilon_0 \) is the external electric field and \( \varepsilon_{eff} \) is the effective bulk susceptibility. When \( \mathbf{p} \ll \mathbf{p}_e \), it follows that \( \varepsilon_{loc} \approx \varepsilon_0 \) [46]. Thus the effective permittivity can be obtained by:

\[
\varepsilon_{eff} \approx \varepsilon + 4\pi\varepsilon\mathbf{p}_e(\mathbf{r}),
\]

where \( \varepsilon \) is the permittivity of matrix, \( \langle \alpha \rangle \) is the averaged polarisability of an individual inclusion. Further calculations involve the GMI effect and surface impedance of wires, which are presented in the following section.

For continuous-wire composites, charge distribution along the wire axis is absent and hence no current or dipole response exists. It can then be treated as a medium with diluted plasma according to Pendry et al. [47]. Thus, the dispersion of effective permittivity for this kind of composite is characterised by the plasma frequency expressed as:

\[
\omega_p^2 = \frac{2\pi\varepsilon^2}{b^2 \ln(b/a)},
\]

where \( b \) is the wire period. In this context, the deduction of effective permittivity can be approached by solving the Maxwell equations in a homogenisation procedure as the wire parameters have no influence on the permittivity. For a nonmagnetic wire composite, the effective permittivity can be given by [48]:

\[
\varepsilon_{eff} = \varepsilon - \frac{2\varepsilon_c F_1(k_c a)}{(ak_c)^2 F_1(k_c a) \ln(L/a) - 1},
\]

\[
F_1 = J_1(x)/J_0(x), \quad \mathbf{p}_e = \pi a^2/L^2, \quad \varepsilon_c = 4\pi\varepsilon\sigma/\omega, \quad k_c = 4\pi i\omega/\sigma^2,
\]

where \( \mathbf{p}_e \) is the wire volume concentration, \( \varepsilon_c \) is the dielectric permittivity of the conductor, \( \sigma \) is the wire conductivity, \( k_c \) is the wave number, and \( J_{0,1} \) are Bessel functions. At microwave frequency, there is a very strong skin effect, i.e., \( ak_c \sim a/\delta \gg 1 \). Thus Eq. (2.17) can be reduced to Eq. (2.16), justifying the application of the model for continuous-wire composites. As with short-wire composites, impedance should be calculated first.
2.2.3.2. Impedance tensor. The surface impedance is a parameter to characterise the voltage response in the wire system, as described in the GMI phenomenon. To calculate the impedance tensor in the wire, the electromagnetic conditions about the wire should be fully understood. Fig. 6 shows the current $e_x$ along the wire axis direction inducing the circular field $h_\phi$, of which the tangential component $h_{x\phi}$ plus the external field induce the electric field $e_\phi$. In this case, the response to the electromagnetic field from the wire via the impedance tensor with the boundary conditions can be written as [49]:

$$
E_t = \zeta(n \times H_t),
$$

(2.18)

where $n$ is the unit normal vector directed inside the wire, $E_t$ and $H_t$ are the tangential vectors of the total electric and magnetic fields at the wire surface, including both scattered and external fields. Adopting the typical simplified approach for antenna problems, Eq. (2.18) can be written in polar coordinates $(x, \phi)$ [12]:

$$
E_x = \zeta_{xx} \overline{H}_\phi - \zeta_{x\phi} \overline{H}_x,
$$

$$
E_\phi = \zeta_{\phi\phi} \overline{H}_\phi - \zeta_{x\phi} \overline{H}_x.
$$

(2.19)

2.2.3.3. Stress and field dependence of impedance and permittivity. For short-wire composites, it follows Eq. (2.15) that the averaged polarisability needs to be worked out. When the interactions between the wires are reasonably neglected, it can be derived as [50]:

$$
\langle \chi \rangle = \frac{1}{2\pi \ln(l/a)(ka)^2} \left( \frac{2}{kl} \tan(kl/2) - 1 \right),
$$

$$
k = \omega \sqrt{\varepsilon} \left( 1 + \frac{ic \zeta_{xx}}{\omega \zeta \ln(l/a)} \right)^{1/2}.
$$

(2.20)

It should be noted that the equations are established in the frame of the composites in question with a moderate skin effect, under which the radiation loss is overshadowed by the magnetic and resistive losses.

It can be seen from Eqs. (2.15) and (2.20) that the permittivity depends on the surface impedance in this case. Due to the GMI effect as previously analysed, the dependence of permittivity through impedance on the external field is well established according to Eqs. (2.5), (2.6), and (2.23).

In the case of long-wire composites, Eq. (2.17) was extended to approach to the case of magnetic wires. By substituting the impedance formula, Eq. (2.17) is transformed to [51]:

$$
\varepsilon_{\text{eff}} = \varepsilon - p_\rho \frac{w^2}{2} \left( 1 + i \frac{\zeta_{xx}}{\omega \zeta \ln(l/a)} \right).
$$

(2.21)

Thus, the effective permittivity for continuous-wire composites is dependent on the wire surface impedance via the plasma frequency.

Fig. 6. Schematic diagram of the magnetic configuration in a wire. Reprinted with permission from [12], copyright 2005 Nova Science.
Due to the amorphous structure of the microwires, their anisotropy consists of no shape anisotropy but the magnetoanisotropy coupled by the internal and/or external stress and magnetostriction. The influence of internal stress and applied stress on GMI has been reported theoretically in Refs. [52,53]. Regarding the glass-coated microwires, the following equations are held:

\[
K = K_0 - 1.5\lambda(\sigma_{zz} - \sigma_{\phi\phi} + \sigma_{\text{applied}})
\]

and

\[
H_k = 2K/\mu_0M_s,
\]

where \(K\) and \(H_k\) are the anisotropy constant and field, respectively, \(\sigma_{zz}, \sigma_{\phi\phi}\) and \(\sigma_{\text{applied}}\) are the axial, azimuthal stress and applied stress, respectively, \(M_s\) is the saturation magnetisation and \(\mu_0\) the permeability in vacuum. Also the effective permeability depends on the ratio of \(H_k\) to \(M_s\), which is determined by \(K\), expressed as [54]:

\[
\mu_{\text{eff}} = \frac{M_s \sin^2(\theta + \theta_e)}{H_k [h \sin^2(\theta + \theta_e) + \cos(2\theta)]},
\]

where the anisotropy field \(H_k = 2K/M_s\) and \(h = H_{ex}/H_k\). \(\theta\) is between the anisotropy angle \(\theta_e\) and \(\pi/2\). Besides, the static magnetisation angle also changes with the static magnetisation angle, as indicated in the equation for the magnetostatic energy \(U_m\) based on the equivalent uniaxial anisotropy [55]:

\[
U_m = -|\tilde{K}| \cos^2(\alpha - \theta) - M_0H_{ex}\cos\theta,
\]

\[
|\tilde{K}| = \frac{K + (3/2)\lambda\sigma_a}{\cos(2\alpha)}
\]

and

\[
\tilde{\alpha} = \frac{1}{2} \tan^{-1} \frac{3|\lambda\sigma_1|}{|K + (3/2)\lambda\sigma_a|},
\]

where \(\alpha\) is the anisotropy angle which takes different values according to the relationship between the anisotropy constant \(K\) and the product of magnetostriction constant \(\lambda\) multiplying the axial stress \(\sigma_a\) [50].

The tunable properties can be characterised by the free-space measurement. From what has been discussed above the dependencies of effective permittivity through magnetoimpedance on the field and stress are well established. A note is in order here. Such metal-dielectric composites incorporating wire-shaped inclusions have been treated theoretically and experimentally for decades [56]. Most recently, researchers have found that it is possible to obtain a negative permittivity and/or permeability at certain frequencies for this kind of composite and recognised its importance [57]. This brings about the next important functionality of the wire-composite, i.e., metamaterial properties, which will be discussed in the following section.

2.2.4. Metamaterial properties

2.2.4.1. Fundamentals of metamaterials. Metamaterials are one of the most appealing subjects in materials and physics nowadays (see, e.g., [57–62]). Any research, if connected to metamaterials, appears to be absolutely fascinating and cutting-edge. To begin with, the basic question is elucidated as to what a metamaterial is.

A metamaterial is by definition an artificially engineered material that gains its properties from its structure rather than its constituents. The resultant properties are not encountered in naturally occurring materials, such as negative refractive index and negative stiffness [63]. First of all, a metamaterial must be non-existent in nature, which accounts for the origin of its name as meta means ‘beyond’ or ‘of a higher kind’ in Greek [64]. It is an extension to the conventional materials in terms of material behaviours. Second, the unique properties of metamaterials are not derived from their constituent materials but from their structure, which distinguishes them from conventional composite materials. Typically yet not essentially, a metamaterial boasts an ordered structure, realised by a periodic arrangement of the functional units, as schematically depicted in Fig. 7. Another implicit regulation
is that the physical dimensions of unit (scale) and neighbouring distance (periodicity) must be smaller than the incident wavelength so that homogeneity can be ensured without which a material cannot be recognised as ‘material’ [63]. The collective responses (effective responses) of all units to the external field (stimuli) give the macroscopic properties of a metamaterial. By manipulating the scale and periodicity of these units one can tailor the properties of a metamaterial. The effective medium theory finds great use herein to study the behaviour of metamaterials as reviewed in Ref. [65], which in turn can be instrumental to metamaterial design and engineering for specific applications. In this sense, metamaterials can also be categorised into smart materials and multifunctional composites.

In this review, the metamaterials are restricted to electromagnetic metamaterials of our major interest. The metamaterial engineered by functional units as basic building blocks can be analogised to the conventional materials out of atoms. Likewise, Maxwell equations can be transformed from microscopic to macroscopic form, making it possible to describe the electromagnetic response of a metamaterial via both an effective permittivity \( \varepsilon(\omega) \) and permeability \( \mu(\omega) \). At the sub-wavelength scale, these two parameters can be manipulated independently thanks to the decoupling of the magnetic and electric field. It follows that some intriguing properties can be achieved for some appropriate

**Fig. 7.** Generic sketch of a volumetric metamaterial synthesised by embedding various inclusions in a host medium.

**Fig. 8.** Negative refraction. (a) An empty glass. (b) A glass filled with an ordinary medium with positive refractive index, such as water; the straw inside the glass is refracted. (c) The water is replaced by a negatively refracting medium. Reprinted with permission from [367], copyright 2006 OSA.
For instance, magnetic responses can be realised in metamaterials consisting of mere non-magnetic constituents [66]. At certain frequencies, a negative refractive index material (NIM) can be obtained with both $\varepsilon(\omega)$ and $\mu(\omega)$ being negative ($n = -\sqrt{|\mu/\varepsilon|}$), as depicted in Fig. 8. The exploitation of NIMs opens up new prospects of manipulating light and produces revolutionary impacts on present-day optical technologies.

2.2.4.2. Classification of and approaches to metamaterials. Since $\varepsilon(\omega)$ and $\mu(\omega)$ can be controlled independently, it is possible to obtain the unusual media with either only negative $\varepsilon(\omega)$ (ENG) or only negative $\mu(\omega)$ (MNG), and both of them negative (DNG) as against the conventional media with both parameters positive. Such a classification according to the sign of $\varepsilon(\omega)$ and $\mu(\omega)$ is shown in Fig. 9 and each class is detailed in the below.

![Classification of materials in the $\varepsilon\mu$ plane in terms of their signs. Reprinted with the permission from [59], copyright John Wiley & sons.](image)

Fig. 9. Arrays of SRR structure (a) and nanorods (b). Reprinted with the permission from [57], copyright 2005 OSA.

![Arrays of SRR structure (a) and nanorods (b). Reprinted with the permission from [57], copyright 2005 OSA.](image)
ENG Many plasmas exhibit this characteristic below plasma frequencies according to the Drude model. Veselago [67] initially proposed gaseous and solid plasmas. Decades later it was found that noble metallic wires (e.g., silver, gold) behave in this manner in the infrared (IR) and visible frequency domains [57]. This is theoretically proposed first by Rotman and Pendry [47,68]; Smith et al. [61] and Shelby et al. [60] realised the idea using thin wires as the scattering elements. Composites containing conductive sticks have also been intensively investigated to realise negative permittivity by Lagarkov et al. [44,46] and Makhnovskiy and Panina [12].

MNG Typical materials of this kind are gyrotropic materials. The composite media with thin wires enabling a resonance feature are also capable of giving negative values of effective permeability near the resonance frequency.

DNG Also known as Veselago medium named after its discoverer [67], left hand material and NIM. It was realised by Smith and Shelby et al. [61] via split ring resonators (SRRs), as shown in Fig. 10a. It consists of two planar concentric conductive rings, each with a gap. Shalaev et al. [57] also successfully fabricated the NIMs using arrays of gold nanorods or thin wires as illustrated in Fig. 10a.

2.2.4.3. Applications of metamaterials. The reason why metamaterials become a focus of intense study is primarily that they afford a range of novel applications. Perfect lenses proposed by Pendry [58] are one of the most exciting applications. A lossless slab (Fig. 11) with a refractive index \( n = -1 \) projects an image of the object placed into the near field with subwavelength precision. This has profound impacts on biomedical imaging and subwavelength photolithography [69].

Metamaterials are a basis for building a practical cloaking device. The possibility of a working invisibility cloak was demonstrated in Ref. [70]. The cloak deflects microwave beams so they flow around a “hidden” object inside with little distortion, making it appear almost as if nothing were there at all. Such a device typically involves surrounding the object to be cloaked with a shell which affects the passage of light near it. Using FE simulations, Cai et al. [71] designed an optical cloaking device which deploys an array of metallic wires projecting from a central spoke that would render an object within the cloak invisible for red light.

Most recently, with a flooding study of metamaterials, more potentials are explored for industrial applications. Sato et al. [72] reviewed the applications of metamaterials in automobiles as summarised in Fig. 12. Melik et al. [73] proposed a metamaterial-based stress sensor with exceptional resolution. Metamaterials are also applied to improve the performance of antennas as reported, e.g., by

![Fig. 11. A superlens capable of high-resolution imaging. Reprinted with the permission from [368], copyright 2007 Nature Publishing Group.](image-url)
Alici and Özbay [74]. The microwave-absorbing capacity of a conventional shielding material is enhanced by the metamaterial coating recently reported by Zou et al. [75], implying the potential of metamaterials in electromagnetic interference shielding (EMI) applications. EMI will be discussed in the next section.

Overall, there are still many unknown applications yet to be tapped into for metamaterials. In the present work, arrays of magnetic microwires are employed to obtain negative effective permittivity. Plus, the antiferromagnetic resonance features of ferromagnetic microwires suggest that a negative permeability is also obtainable. In this sense, the microwire-composite could demonstrate the metamaterial functionalities, for which corresponding applications are anticipated.

2.2.5. Electromagnetic interference shielding and microwave absorption

2.2.5.1. EMI shielding. Electromagnetic interference (EMI) by definition is the process by which disruptive electromagnetic energy is transmitted from one electronic device to another via radiated or conducted paths or both. Suppression is the process of reducing or eliminating EMI energy. It may include shielding and filtering [76]. EMI problems have impacted almost all electrical and electronic systems from daily life to military activity, and to space exploration, for the following major reasons: (i) the proliferation of electronic equipment and various devices in both industrial and domestic environments affords a good chance for EMI; (ii) miniaturisation of components in electronic systems and systems themselves physically increases the potential of EMI; and (iii) new digital technology turns out to be in favour of EMI [76].

EMI has been found not only detrimental to electric appliances but also harmful to human health as it may contribute to serious diseases such as cancer [77]. Effective shielding materials are therefore desired to minimise the influence of EMI. To locate effective shielding materials, one has to understand foremost the EMI theory, or, what constitutes EMI and what material properties are desirable to arrest EMI.

EMI theory lies within the framework of electromagnetic wave theory. An electromagnetic wave consists of two components, namely, a magnetic field $\vec{H}$ and an electric field $\vec{E}$. They are normal to each other and the prorogation direction of the wave is normal to the plane containing them. The ratio of $\vec{E}$ to $\vec{H}$ is termed as impedance. EMI shielding can be divided into the near field region and the far field region according to whether the distance between the radiation source and the shield is smaller than $\lambda/2\pi$ or not. It is the far field region that the EM wave theory is applicable, whilst the electric and magnetic dipoles are responsible for the EMI occurring in the near field region [78].

EMI capacity is parametrised by the shield effectiveness (SE), which is defined as the ratio of the residual energy to impinging energy. The EMI attenuation afforded by a shield relies on three mechanisms as depicted in Fig. 13. The first is the reflection of the wave from the shield ($R$); the second is the absorption of the wave when it passes through the shield ($A$); and the third originates from the
multiple reflections of the wave at various interfaces within the shield (M). The total SE can be given in the form [79]:

$$SE = -10 \log \frac{P_{\text{out}}}{P_{\text{in}}} = SE_R + SE_A + SE_M,$$

where $P_{\text{in}}$ and $P_{\text{out}}$ are the incident power and transmitted power, respectively. The reflection shielding effectiveness, $SE_R$, can be approximated as $SE_R = -10\log(1 - R)$. Likewise, the multiple reflection shielding effectiveness, $SE_M$ can be expressed as $SE_M = -10\log(1 - M)$. Thus, the absorption shielding effectiveness, $SE_A$, can be approximated as $SE_A = -10\log[T/(1 - R - M)]$, where $T$ represents transmission.

To suppress the increasing EMI, it is necessary to pursue new materials with enhanced EMI shielding capacity. Due to the skin effect (see Eq. (2.6)), high frequency EM radiation only occurs in the surface layer of an electrical conductor. Since a smaller skin depth results in larger attenuation, lower resistivity and higher permeability are required for achieving a better shielding performance. In this context, a wide range of candidate materials have been suggested for EMI shielding applications. A most typical material among them is the polymer composite containing metallic fillers such as steel, nickel or silver in the form of powder fibre or flake [80–83]. It was reported that the shielding effectiveness (SE) exceeded 30 dB for steel fibre reinforced composites with a filler loading of 6 wt.% and a thickness of 1.5 mm at 1–2 GHz [82]. An SE of more than 50 dB was also reported for the nickel powder-filled composites with a filler loading of 40 wt.% and a thickness of 2.85 mm [80]. While these fillers benefit the shielding properties of the resultant composites, the large density, physical rigidity and low corrosion resistance of the metallic phase are disadvantageous. In addition, a relatively high loading of these fillers is required for achieving low resistivity and high SE. Recently, carbon fibre [84–87] and carbon nanotubes (CNT) [88–95] have been shown to be useful for EMI shielding applications. In the case of carbon fibres, it would be ideal if they could yield large SE as these materials are widely used in the aerospace and automobile industry. However, when compared with metallic fillers, the higher resistivity of carbon fibres imposes a stringent requirement of a larger loading of carbon fibres in order to achieve the same shielding effectiveness, thus raising the manufacture cost. While some efforts have been made to improve the conductivity of the fibres by coating them with a thin metallic layer, the coating itself could be easily damaged during the coating processing [84]. For the case of the carbon nanotubes-based composites, Li et al. [88] reported an SE of more than 25 dB for MWCNT/polyacrylate composites with 10 wt.% CNTs and a 1.5 mm thickness at X-band (8–12 GHz). Park et al. [89] found that a 15 μm thin CNT bucky paper containing more than 50 wt.% CNTs reached the SE of 22 dB at 2–18 GHz. By catalysing the CNTs with magnetic particles such as Fe, Co, Ni and their compounds, the SE of the CNTs-based composites was significantly improved due to the increased absorption as a result of the introduction of magnetic permeability [90–95]. However, the difficulty in dispersing nano-sized fillers and high fabrication costs hinder the CNTs-based composites from commercial
applications. Some conductive polymers have also been developed recently for EMI shielding applications, but their poor mechanical properties make them undesirable for such applications [96–99].

2.2.5.2. Microwave absorption. Absorption, as mentioned above, occurs when the microwave interacts with the materials. The absorption or attenuation originates from the dielectric loss (polarisation and conduction loss) and the magnetic loss (ferromagnetic resonance, eddy current loss, hysteresis loss, etc.). The key to realise high absorption consists in not only a large loss factor but matching permittivity and permeability (impedance match) according to the transmission line theory [100]. For a single layer composite, the reflection loss (RL) is given by [100,104,105]:

\[
RL = 20 \log \left| \frac{\mu \tan \left( j \frac{2 \pi f d}{c} \sqrt{\mu \varepsilon} \right)}{\varepsilon \tan \left( j \frac{2 \pi f d}{c} \sqrt{\mu \varepsilon} \right) + 1} \right|, \tag{2.26}
\]

where \( \mu \) and \( \varepsilon \) are the relative permittivity and permeability of the absorbing material, respectively.

\[
\alpha = \Re(\gamma) = \Re \left( \frac{j \omega \sqrt{\mu \varepsilon}}{c} \right) = \frac{\omega}{\sqrt{2c}} \sqrt{\mu'\varepsilon'' - \mu'\varepsilon' + \sqrt{\left(\mu^2 + \mu''^2\right)\left(\varepsilon^2 + \varepsilon''^2\right)}}. \tag{2.27}
\]

Here one can see that the attenuation constant is dependent on complex magnetic permeability. To obtain large attenuation constant at any frequency, it requires large \( \mu'' \), \( \varepsilon'' \), \( \tan \delta_\varepsilon \), and \( \tan \delta_\mu \). However, this condition needs to be tempered with Eq. (2.26), whereby a matching condition must be satisfied in order to obtain minimum reflection loss, expressed as [107–109]

\[
\sqrt{\frac{\mu}{\varepsilon}} \tan \left( j \frac{2 \pi f d}{c} \sqrt{\mu \varepsilon} \right) = 1. \tag{2.28}
\]

Detailed calculation reveals that the absorption bandwidth and maximum absorption are actually two offset parameters. For a dielectric absorber, the usual case is that larger \( \varepsilon'' \) gives rise to larger loss tangents and an optimised \( \varepsilon'' \) and associated loss tangent can be expected to achieve the maximum absorption condition (too small or too large \( \varepsilon'' \) will not be in favour of absorption). For a magnetic absorber, the matching frequency can be regarded as the ferromagnetic resonance frequency, which can be given as

\[
f = \frac{r}{2\pi} H_k, \tag{2.29}
\]

where \( r = 2.8 \text{ MHz/Oe} \) is the gyromagnetic ratio. The anisotropy field \( H_k \) is given by [110]

\[
H_k = \frac{2|K_1|}{\mu_0 M_s}, \tag{2.30}
\]

where \( K_1 \) is the anisotropy constant and \( M_s \) is the saturation magnetisation. A larger saturation magnetisation or smaller anisotropy field will redshift the resonance frequency, which also means an improved absorption bandwidth, since there exists a trade-off between resonance frequency and absorption bandwidth [111].

---

3 The aim of this section is to provides a basic understanding of MA theory relevant to microwire composite. More detailed discussion on the case of multilayer absorber and the design principle can be found in other review papers [101] or books [100,102,103].
Since the implementation of carbon black and Al flakes as radar absorbing material in the airborne and seaborne vehicles during WWII [112], a plethora of absorbing materials have been developed and commercialised including dielectric absorbing materials such as carbon black [104,113,114], carbon nanotubes [115–117], SiC fibres [118] and nanowires [119], short carbon fibres [120–122], and other non-magnetic conducting particles; and magnetic absorbing materials such as M-type hexaferrites [123,124], magnetic nanoparticles [106,125] and magnetic particles filled nanotubes [90,91,126]. Although the nanomaterials have attracted much attention as promising absorbing materials, the most widely used absorbing materials are still magnetic ferrites [127–129], carbon black and carbonyl iron powder [130–132]. In comparison with the magnetic powders, the ferromagnetic microwires are advantageous in their unique shape anisotropy that aids wave attenuation, a capability to overcome the limitation of Snoek’s law, and superior tailorability of electromagnetic properties through wire geometry and concentration [133,134]. In this context, in spite of their relatively large diameter, the ferromagnetic microwires with superior electromagnetic properties and mechanical properties are investigated as structural absorbing materials in quite a few studies, which will be discussed in Section 6.2.

3. Techniques for processing microwires and their composites

3.1. Amorphous metallic wires

Based on the conventional techniques for fabricating amorphous alloys, a variety of fabrication techniques have been advanced for microwires fabrication, including melt spinning [135–139], in-rotating water spinning [8,140–145], electrodeposition [146–151] and the most popular technique, the Taylor–Ulitovskiy method [8,152–159]. For details of these processing techniques, one can refer to the previous reviews on GMI [7,31] and microwires [6,9,160,161]. Here we just discuss the melt-extraction technique that is absent in previous reviews.

Melt-extraction (MET) was first applied to prepare metallic fibres as long as four decades ago [162]. It was further developed to find wide use in the fabrication of amorphous wires, ceramic fibres such as calcium aluminate [163] and high resistivity alloy wires such as MgZnCa [164]. Recently, it is increasingly used in the fabrication of magnetic microwires [165–169]. The basic principle of MET is to apply a high-speed wheel with a sharp edge to contact the molten alloy surface and then to rapidly extract

![Fig. 14. Schematic of melt-extraction facility.](image)
and cool a molten layer to be wires, as schematically shown in Fig. 14. There are three main advantages of this technique: (i) MET gives a higher solidification rate of $10^5$–$10^6$ K/s than any other method, which is in favour of the form of amorphous phase. (ii) The wires produced by this method possess extraordinary mechanical properties due to the quality faultless surface and circular geometry [164,170]. (iii) The soft-magnetic properties of the materials are significantly improved by fabricating into microwires via MET; this is believed to be attributed to the considerable quenched-in stress [167]. The main drawback of this method is that it has loose control on the diameter of the produced wires. The typical diameter range for the magnetic wires is about 30–60 μm [165–167,171] depending on the processing parameters. Apparently, it is not suitable for preparing ultra thin magnetic microwires.

3.2. Microwire composites

Loosely defined, microwire composites can be any form of material consisting of wires and a matrix material, while in engineering parlance, microwire composites conforming to the above definition may not be able to serve general structural purpose and hence cannot be categorised as composite materials. In the section, the scope will be extended to any form of microwire composite with a particular note of its application range. The emphasis is placed on the microwire composites that meet the quest for both functional and structural use.

3.2.1. Microwires-epoxy

Epoxy is believed to be the most extensively used as the matrix material for all kind of composites and coatings for engineering applications. Zhang et al. [172] prepared a microwire composite coating on the surface of aluminium plate using polyamine dissolved by alcohol. The resultant composite and microwires arrangement are shown in Fig. 15. Liu et al. [173] also used epoxy to cast the toroidal samples for microwave characterisation. Starostenko and Rozanov [30] fabricated the composite mat by coprecipitation of glass fibre and wire pieces in a dilute solution of polystyrene. It should be noted that the wire pieces are used in these works are in the range of 5–10 mm. If the wires are too long, it would be challenging to realise a good dispersion of wire pieces and receive the 2D plane-isotropic composite. If the wires are too short, the demagnetising effect would be too strong and ruin the overall electromagnetic properties of the microwire composite.

3.2.2. Microwires-elastomers

Compared to epoxy, elastomers usually have much smaller Young's modulus (2 MPa) and hence will appropriate strain when subject to even a relatively small force. Thus rubber-based composites are suitable for characterising their responses to the stress.

Qin and co-workers [174,175] used two pieces of transparent silicone rubber sheets to prepare the planar composites with periodically arrange continuous wires. The procedure schematically shown in Fig. 16 is as follows:
(1) 500 mm long microwires were laid out in a periodical manner with a fixed wire spacing on the white sheet.

(2) Around 80 g thoroughly stirred and vacuumed liquid silicone rubber/hardener mixture were uniformly cast on the surface as the adhesion layer, which was followed by covering the transparent layer right on the top. An aluminium plate and heavy weights were then placed on the top of the preform to assist the curing process in the ambient air.

Fig. 16. Schematic of the preparation of continuous-wire composites based on silicone rubber for free-space measurements; the final dimensions of the resultant composite are $520 \times 500 \times 1.5$ mm.

Fig. 17. Schematic structure of toroidal composites: (a) Isotropic samples; (b) anisotropic sample. Reprinted with permission from [171], copyright 2007 Nonferrous Metals Society of China.
(3) After 24 h when the resin was cured, four surface-roughed glass-fibre tabs of 10 mm × 500 mm with two drilled holes of 6.35 mm were attached on both sides normal to the longitudinal direction of the sheet. The holes were designed for load bearing.

(4) The sample was then sent to an oven to cure the resin used for gluing the tabs at 70 °C for 1 h. As a final step, the holes in the tabs were further pierced through the sample and strings were passed through the holes as well for fastening the weights in the study of stress tunable effect.

For the case of short-wire composites, Marin et al. [176] dispersed 40 g 1 mm Fe-rich microwires into the silicon resin to receive a composite sheet for microwave absorption application. Di et al. [171] and Zhang et al. [172] used rubber dissolved by acetone to prepare the composite of the toroidal ring shape, in that it can fit the sample holder specific for the measurement by a vector network analyser. The wire arrangement can be made either regular or random as shown in Fig. 17, which determines if the composite is isotropic or anisotropic. Yet this kind of composite (inner diameter of 3 mm, outer diameter of 7 mm and height of 3.5 mm) is very small and has limited application.

3.2.3. Microwire E-glass prepregs

E-glass prepregs have been widely used in the industry and are themselves excellent structural composites. Naturally, using it as a matrix to make microwire composite secures the structural function even with a very small amount of microwires.

Ref. [177] detailed the preparation of short-wire composites and continuous-wire composites with E-glass prepregs as matrices. For example, the preparation work of the short-wire composite was done in the following steps:

(1) Five centimetre wire-pieces were laid out at zero degree along the glass-fibre direction between the two layers of prepregs with in-plane size of 50 cm × 50 cm (the size may vary to fit different measurements). The wire spacing was controlled at fixed values of the order of centimetres (comparable to the wavelength at gigahertz range) in perpendicular and parallel directions as shown in Fig. 18a. Note that the wire length and spacing are selected within the resolution range of the microwave measurements at gigahertz.

(2) Another two layers were laid up on the top and bottom of the wire-embedded layers in the same direction, giving a layup of four prepreg layers containing short wires.

(3) After bagging the composite on an aluminium plate with air sucked out to the required vacuum of 94.6–104.7 kPa, the material was cured in an autoclave. The curing conditions are as follows: the temperature was raised at a rate of 2 °C/min to 127 °C and kept for 80 min before cooling down naturally to room temperature. At a rate of 69 kPa/min the pressure was increased to 206.7 kPa (30 psi) and kept at this level for 30 s and then 690 kPa for 600 min before decreasing at a rate of the 20.7 kPa/min.

A typical cross-sectional view of prepreg based wire composite is shown in Fig. 18b. On the exterior surface, one can see several ridges of different colours from other regions (indicated by arrows), which is attributed to a non-uniform distribution of the resin and glass-fibre in the prepregs [28], as revealed in the scanning electron microscope image of the cross-section (Fig. 19b). It
is the microwire, which is slightly larger than the glass-fibres in diameter, that results in the non-uniform distribution of the resin in the region close to it. However, such influence from the microwires is limited to the near-wire region only and is comparable to the inherent defects in the prepregs. Besides, since the wire-composite is intended to contain a very small number of wires that are separated in a spacing of a few millimeters to a few centimetres, which is three to four orders of magnitude higher than the diameter of the microwires, the disruption of microwires to the composite integrity is minimal.

4. Basic magnetic and mechanical properties of microwire composites

4.1. Magnetic properties of composites

Due to the inclusion of magnetic fillers, the polymer composite becomes magnetic, i.e. responsive to the external static or dynamic magnetic field. Although most studies are devoted to the dynamic response of these kinds of heterogeneous composite media [178], i.e., complex permeability, it is worthwhile exploring the static magnetic properties of microwire composites for two reasons: (i) the composite with wire arrays could be of some application interest in the magnetic sensing field.
as quite a few studies are devoted to the microwire arrays [179–184]. (ii) AC permeability is associated with the static magnetic properties such as saturation magnetisation and the anisotropy field according to the modified model based on Snoek’s law proposed by Acher and co-workers [111,185]

\[ \int_0^F \mu'(f) f \, df \leq \frac{\pi}{6} \tau (\gamma 4\pi M_s)^2, \quad (4.1) \]

where \( \mu \) is the imaginary part of complex permeability, \( f \) is the frequency, \( \tau \) denotes the volume fraction of magnetic fillers, \( \gamma \) is the gyromagnetic factor with a value of 2.8 MHz/Oe, \( M_s \) is the saturation magnetisation. In this connection, this section addresses some of the static magnetic properties of microwire composites.

Phan et al. [186] prepared E-glass prepreg-based composites with bundles of microwires and measured their magnetic properties were measured in comparison with those of the single wires. Fig. 20 shows the M–H curves of the single microwire and composite. It is interesting to note that, for both longitudinal and transverse measurements, the coercivity of the as-prepared composite is much smaller than that of the single microwire. This indicates better soft magnetic properties for the microwire composite than single wires. It is also shown that the effective anisotropy field for bundles of microwire is strongly increased as compared to the single wire [171]. Such changes of magnetostatic and magnetoelastic characteristics are due to the long range dipolar interactions [184,187] between neighbouring wires as well as the interfacial stress between microwires and matrix. The dipolar interactions have a strong impact on the magnetic properties in a similar way to classical spins interacting throughout long-range interactions, resulting in the changes in the hysteresis loop. Equally, interfacial stress of the order of hundreds of MPa [175,188], resulting from the different coefficient of thermal expansion between the microwire and polymer matrix, will have significant effect on the magnetoelastic features of the wires according to \( K_{me} = 3/2 \lambda (\sigma_i + \sigma_{app}) \), where \( K_{me} \) is the magnetoelastic energy component, \( \lambda \) is the magnetostriction constant, \( \sigma_i \) and \( \sigma_{app} \) are the internal stress and applied stress on the wire, respectively [189,190]. In the case of Fe-based wire of a length greater than critical length, due to the dipolar interactions, the coercivity and anisotropy field is supposed to be increased with number of wires. On the other hand, the coercivity is decreased with increasing stress and anisotropy presents an opposite trend [188,189,191]. The combination of these two mechanisms results in the observed contrast between the microwire composites and monolithic wire. The presented magnetic properties indicate that the microwire composites are promising for magnetic sensing applications [8].

Due to the involvement of polymer, magnetic filler and multi-interfaces, the temperature plays an important role in regulating the magnetic behaviour of composites. The glass transition temperature \( (T_g) \) of the composite samples is evaluated for one to gain a better understanding of their magnetic properties (see Fig. 21). Phan et al. found that, by annealing in the vicinity of the glass transition temperature, there exists a critical temperature of 177 °C. Before the temperature reaches it, the coercivity decreases while afterwards the opposite trend is shown. This is contrary to the conventional case that the coercivity should decrease with the stress relaxation due to the thermal annealing below crystalization temperature [192]. Clearly, such a complex annealing temperature dependence of coercivity involves multiple mechanisms at different interfaces, viz, the structural relaxation in the wire, the stress relief in the polymer–wire interface. The competitive and interdependent relationship between these two mechanism vary sensitively with the temperature. At low temperature relative to glass transition temperature, the second mechanism prevails and could result in a rearrangement of microwires, i.e, regulation of the composite mesostructure. Thus the coercivity could be increased due to the enhanced interaction between the wires [193]. Subsequently, the decrease of coercivity should result from the stress relief contributed by both glass-transition and wire structural relaxation. The gradual decrease of remnant magnetisation can be accounted for by the reduction of the volume of the axially magnetised area as an effect of stress relief due to the positive magnetostriction constant [189,191].

### 4.2. GMI effect

As with wires, their composite also presents a significant GMI effect. It has been shown by Phan et al. [186] that, at a given frequency of 10 MHz, the GMI ratio and its field sensitivity reached the
highest values of 450% and 45%/Oe for those composites containing four wires, while the composite containing only one wire gave 14% and 1%/Oe, respectively (see Fig. 22). The wire addition effect on the GMI is believed to be attributed to the reduction of resistivity as one of the causes. Another cause is the combination of the high circumferential permeability of the microwire and a multiwire-parallel configuration is likely to cause a magnetic flux closure and hence resulted in an extremely high effective permeability [193,194]. It is worth noting both GMI and anisotropy field are enhanced, which is highly desirable for realising high-performance sensing entailing large sensitivity and broad working range. In addition, compared to single wire, the existence of polymer matrix could accommodate a versatile and judiciously designed multi-wire system, which provides more possibility to extend its applications. However, the GMI presented in Ref. [186] is very weak, with suitable optimisation of single wires via advanced annealing techniques such as stress-joule-annealing [195] and recently reported multi-angle annealing techniques [196], the GMI of wire composite could be further improved.

Qin et al. [197] also demonstrated the pronounced improvement of maximum MI effect with increasing wire inclusions, attributed to the effective response of all the wires. The microwire composite is

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Fig. 21. Magnetic hysteresis loops of as-prepared composite annealed at $T_a = 375\, \text{K}$, 405 K and 435 K. The glass transition temperature is 449.5 K. Reproduced with permission from [186], copyright 2007 AIAA.

Fig. 22. Frequency dependence of GMI profiles with varying wire number $n = 1, 2, 3, 4$ mm. Reproduced with permission from [369], copyright 2007 Elsevier.
shown to yield a larger GMI at higher frequencies but with minor increase of energy loss, which is desirable for sensing applications. The multi-wires in the parallel manner constitute an increase of the total cross sectional area of the wires, resulting in a stronger skin effect when considering the ratio of radius and penetration depth. Consequently the GMI effect is improved. In addition, if the wire is simply dealt with as a single domain substance, the interactions between wires in the multi-wire composites induce a magnetic closure to make the whole structure more stable, albeit the total internal energy is increased [187]. These results differ significantly from those reported by Garcia et al. [198], who demonstrated a multi-wire system exhibiting a reduced GMI effect with the number of wires, which is explained by the authors as an effect of shielding between wires. This difference should be ascribed to the functions of the matrix and the curing process for our composite media. The wire-sample underwent annealing in the curing stage and the domain structure were significantly modified. It is suggested that, the confinement of the matrix also helps maintain the modified configuration, giving rise to the peculiarity of the GMI behaviour. Of note is that the involvement of polymer matrix opens up new routes to modulate GMI performance, although limited study has been reported thus far. A particularly important aspect is interface. The homogeneities of interface would be critical to the GMI characteristics, it is therefore necessary to study the detailed interfacial conditions. Surface treatment using chemical agents such as silane or nanotubes [199] could also be considered to enhance both interfacial bonding and GMI properties. Another aspect of practical interest [200] is to develop superior bonding technology to accommodate multiple wires so that a high stability of GMI signal output can be ensured.

It is well established that GMI effect of microwires associated with the magnetic properties is strongly influenced by the length of wires [180, 201–204]. The argument holds true for the microwire composite system. It has been revealed that the GMI ratio increased from 15.5% to 268% as the micro-wire length decreased from 4 to 1 mm [186]. This is attributed to the decrease of the electric resistivity in the multi-wire system compared to the single wire, in spite of the increase of the demagnetising effect [180] for single wire. Based on the outstanding magnetic properties of microwire composites, it shows a promising application perspective. Indeed, the polymer matrix provides a ‘platform’ to assemble the microwires and exhibit the properties that is significantly different from the single wire, which is exactly what composites are for [205].

4.3. GSI effect

As mentioned above, the stresses can play an important role in investigating the GMI materials, and these can be treated in two respects. One concerns the stress influence on the GMI behaviour, and a number of works have been published on this topic for various materials including ribbons [206–208] and microwires [209–211]. Second is the so-called giant stress-impedance (GSI) effect,
which refers to a stress-induced variation of impedance in magnetic materials. This effect in the intermediate frequencies has been evaluated on amorphous wires and ribbons. Shen [212] reported that the GSI effect of a CoSiB microwire reached about −35% for stress $\sigma = 140$ MPa, which is five to six times more sensitive than a conventional semiconductor stress sensor. One kind of Fe-based ribbons was also found to possess an SI ratio of 20% for $\sigma = 84.8$ MPa with the stress perpendicular to the geomagnetic field [213]. In this regard, the wire-based composite is expected to be a good candidate for studying the GSI effect over a wider stress range, since it can be treated as a multi-layer medium consisting of a metallic core, a glass coat and a composite matrix. Such a hierarchical structure would enable a sensitive response to the applied stress for the composite media, where the coupling of internal and external stresses is likely to be able to manipulate the GMI and GSI behaviour. Both aspects of the GSI effect of microwire composites were investigated by Qin et al. [188].

Fig. 23 illustrates the magnetic field dependence of the GMI ratio taken at $f = 1$ MHz under various applied tensile stresses for the single-wire composite of four layers obtained by the method described in Section 3.2.1. Here the tensile stress was applied along the microwire axis. It can be seen from the figure that the maximum GMI ratio decreased as the applied stress was increased. While the GMI ratio decreased gradually with the applied magnetic field ($H_{dc}$) for the unstressed composite, the case is different for the stressed composites. With increasing $H_{dc}$, the GMI ratio first increased, then reached a maximum, and finally decreased for the stressed composites. The magnetic field at which the GMI ratio reached maximum can be considered as the circular anisotropy field ($H_k$) induced by the applied tensile stress.

Fig. 24a illustrates the tensile stress dependence of the maximum GMI ratio and the anisotropy field. Clearly, the GMI ratio decreased slightly from 255% for $\sigma = 0$–253% for $\sigma = 150$ MPa and drastically reduced to 151% for $\sigma = 600$ MPa by more than 100%. An opposite trend was observed for the $\sigma$ dependence of $H_k$. A subtle increase of $H_k$ by 8 A/m for $\sigma = 150$ MPa was followed by a further increase of 112 A/m for $\sigma = 600$ MPa. The $\sigma$ dependence of longitudinal coercivity ($H_c$) was also measured and the result is shown in Fig. 24b. The drop of $H_c$ was observed at $\sigma = 600$ MPa. For the case of the unstressed composite, the gradual decrease of the GMI ratio with $H_{dc}$ is likely to be caused by decreasing circular permeability due to the rotation of magnetic moments. However, an opposite effect

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**Fig. 24.** (a) Tensile stress dependences of anisotropy field ($H_k$) determined from GMI profiles and maximum GMI ratio. (b) Longitudinal coercivity vs. applied tensile stress. Reproduced with permission from [188], copyright 2011 Elsevier.
observed for the stressed composites arises from the fact that the application of tensile stress to the microwire with a negative magnetostriction strengthened the circular magnetoelastic anisotropy field, giving rise to the occurrence of a maximum in GMI that shifted towards higher $H_{dc}$ as $\sigma$ was increased.

The occurrence of such a maximum in GMI for the stressed composites can be attributed to the competition in the rotational magnetisation processes between $H_{dc}$ and $H_k$. The decrease of the GMI ratio with $\sigma$ at $H_{dc} = 0$ is attributed to a reduction in the circular permeability due to the domain wall displacement.

Furthermore, it should be noted that, since the microwires were embedded in a polymer matrix, any heat treatment or variation coming from the polymer matrix is expected to have a significant influence on the GMI properties of the microwire and hence the composite. To gain insight into this, the influences of annealing treatment and the number of layers on the GMI properties are studied. The results are shown in Fig. 25. For the case of as-prepared samples, the four-layer composite exhibited a higher maximum GMI ratio (255%) when compared with the eight-layer composite (216%). Meanwhile, the annealed composite samples (treated at 100 °C for 3 h) exhibited higher maximum GMI ratios (282% and 248% for the four-layer and eight-layer composites, respectively) than the as-prepared ones. These findings are explained as follows. In this kind of composite, a higher stress will be induced as the number of composite layers increases. In the present study, the eight-layer composite may impose more stress in the through-thickness direction than the four-layer composite. As a result, the GMI ratio decreased and $H_k$ increased for the as-prepared composite of eight layers when compared with the as-prepared composite of four layers. On the other hand, the annealing treatment is believed to have relieved the stress between the microwire and matrix produced during the curing process. This explains consistently the larger GMI ratio and smaller $H_k$ obtained for the annealed composite samples relative to the as-prepared ones.

It is also interesting to see that the GMI behaviour observed for the as-prepared eight-layer composite is similar to that of the four-layer composite subject to a tensile stress of 450 MPa. This clearly suggests that the applied tensile stress and residual stress imposed by the composite matrix on the microwire could affect its GMI behaviour in a similar fashion. To understand this quantitatively, the contribution of the applied stress and the matrix-induced residual stress to the anisotropy field were calculated, respectively.

In a cylindrical coordinate system $(z, r, \phi)$, there exist three components for the residual stress in $z$ (along the wire axis), $r$ (radial direction) and $\phi$ (azimuthal direction): $\sigma_{zz}$, $\sigma_{rr}$ and $\sigma_{\phi\phi}$, respectively. The magnetoelastic energy density is given by [214]:

$$U_{me} = -\frac{3}{2} \mu_0 \left( \sigma_{zz} x_z^2 + \sigma_{rr} x_r^2 + \sigma_{\phi\phi} x_\phi^2 \right),$$

(4.2)
where $\lambda_s$ is the saturation magnetostriction constant. $z_i$ denotes the component of the unit magnetisation vector. The residual stresses are assumed to be a function of $x(r/a)$ only, $a$ being the wire radius.

The anisotropy field can then be deduced by the following equation [214]:

$$H_k = \frac{3|\lambda_s|}{M_s} \left( \sigma_{zz} - \sigma_{\phi \phi} + \frac{1}{(1 - k)p^2 + k} \sigma_{zz} \right).$$

(4.3)

where all symbols have the same meaning as in Eq. (4.2).

Taking into account the residual stress imposed by the composite matrix, which is primarily along the negative radial direction ($\sigma_{rr}$), Eq. (4.3) can be modified to:

$$H_k = \frac{3|\lambda_s|}{M_s} \left( \sigma_{zz} - \sigma_{\phi \phi} + \sigma_{rr} + \sigma_{zz} \right);$$

(4.4a)

$$\sigma_{zz} = \frac{1}{(1 - k)p^2 + k} \sigma_{zz}.$$  

(4.4b)

For four-layer composite, when $\sigma_{zz} = 450$ MPa, given $k = 0.5$ and $p = 0.67$, one can receive the effective contribution to $H_k$, $\sigma_{zz} = 619$ MPa. For the eight-layer composite, according to the difference of its anisotropy field relative to that of unstressed four-layer composite ($\Delta H_k = 0.5$ Oe), the concerned stress $\sigma_{rr}$ is given by:

$$\sigma_{rr} = \frac{\Delta H_k M_s}{3|\lambda_s|}.$$  

(4.5)

Given the typical numerical values: $M_s = 400$ G, $\lambda_s = -10^{-7}$, $\sigma_{zz} = 667$ MPa is received. The similar numerical values of $\sigma_{rr}$ and $\sigma_{zz}$ explain the roles of applied stress and residual stress on influencing the magnetoelastic properties and GMI properties. This result affords significant technical applications in the composite industry in terms of probing the residual stress and tailoring the related manufacture conditions.

The GSI effect in the microwire and the microwire-based composites with four and eight layers was investigated. The results are shown in Fig. 26. It can be readily seen that the stress-impedance ratio $[\Delta Z/Z]$ decreased monotonously with stress for all the samples. Interestingly, while the maximum stress impedance ratio $([\Delta Z/Z]_{\text{max}})$ is 38.1% for the single microwire, it is reinforced for the composite samples (41.5% and 43.0% for the four-layer and eight-layer composites, respectively). These results indicate that the prepared composites are very appealing candidates for stress sensing applications.

In the composite, in addition to the residual stress frozen-in between the glass and metallic core, there exists residual stress at the interface between the microwire and polymer matrix. The enhancement of...
the GSI effect in the composites clearly points to the important coupling between internal and external stresses that coexist in these materials.

4.4. Mechanical properties

Since the fabricated composite is intended for structural applications, its mechanical properties are one of the major concerns. Also, the influence of microwires on the mechanical integrity of the composite is a key issue for the success of such sensor-embedded technology. Therefore, it is necessary to discuss the mechanical properties of the composite matrix influenced by the embedded wires [197,215–217].

Qin et al. [197,217] found that, in the dilute composite with glass-fibre reinforced epoxy as a matrix, the fibre shows little effect as shown in Fig. 27, which displays the stress–strain curves obtained for blank composite samples, 10-wire, and 50-wire samples under a maximum force of 30 kN [197]. The tensile strengths are summarised in Table 1. It is observed that all the mechanical parameters are very close to each other for the different types of samples. In terms of the tensile strength, the coefficients of variation (CV) for each type of sample are 6.3, 5.6 and 3.8. Upon performing cross comparison of their average properties, the CV is 3.7. The negligible wire effect can be understood from the basic law of mixture that postulates a considerable volume fraction of fillers needed to realise reinforcement.

Back to 1980s, Goto and co-workers [215,216] had done excellent work in this regard. Goto and Nishio [215] studied the reinforcing effect of metallic wires with and without glass coats on the epoxy matrix and reported that the mechanical properties of epoxy is strongly enhanced for the wires without glass-coat, the tensile strength reaching 600 MPa at 30 vol.% wire volume. While the glass-coated wires do not show as good reinforcing effect due to the poor bonding between metallic core and glass coat. These effects are readily demonstrated in the fracture morphology images (Fig. 28). Apparently, the quality of interfaces play a critical role in determining the ultimate mechanical performance of the

![Stress–strain curves of blank composites (free of wires), composites containing 10 wires and 50 wires measured by a 30 kN load cell. Reproduced with permission from [197], copyright 2010 Elsevier.](image)

**Table 1**

<table>
<thead>
<tr>
<th></th>
<th>Blank sample</th>
<th>10-Wire sample</th>
<th>50-Wire sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>Young’s modulus (GPa)</td>
<td>14.22 (3.1°)</td>
<td>14.48 (1.1)</td>
<td>14.97 (3.0)</td>
</tr>
<tr>
<td>Tensile strength (MPa)</td>
<td>951.48 (6.3)</td>
<td>935.18 (5.6)</td>
<td>1003.80 (3.8)</td>
</tr>
</tbody>
</table>
wire composites. These fine wires after removing glass coats are also found capable of improving the thermal properties of the composite by increasing the glass transition temperature as the wires can hinder the molecular motion.

Although the microwires can reinforce the polymer materials with adequate concentration and proper orientation, a trade off is often inevitable if one tend to integrate the structural function and other functionalities which favour not necessarily the large concentration or unidirectional fillers. For the designers of such multifunctional composites, it is necessary to maximize the most desirable function for targeted application but with a cost of other functionalities as low as possible.

5. Microwave tunable properties of microwire composites

Further to the understanding of the GMI/GSI behaviours of microwires and their composites, this section targets the microwave tunable properties of the microwire composites, i.e. tunable electromagnetic properties by magnetic field [12,30,41,43,51,197,217–222], stress [12,50,51,55,175] and temperature [41,218]. The tunable property is actually the so-called cross-variable response unique to multifunctional composites, i.e., a given field could control two or more variables, or a variable can be switched by two or more external fields. To achieve such tunability or adjustability is essential for microwave applications such as tunable microwave devices [12] and remote interrogated sensors [223]. It will also be called for to realise reconfigurable local network environment, beam-steering antennas, and microwave remote sensing and control. Most proposed methods have been based on biased ferroelectric, ferrite or magnetic composite substrates and reconfigurable resonant elements implementing active devices or a system of micro actuators [224–226]. These technologies each have their advantages and limitations such as high power consumption, low operational speed, limited frequency band and high cost. The dilute composites with ferromagnetic microwires were proposed in this context by Panina and co-workers [12,51]. Together with following studies [30,174,175,197,219,221,222], the possibility of tailoring the collective dielectric response of the wire media by changing the local magnetic properties with external stimuli without changing the structural parameters has been demonstrated.

In what follows the tunable properties will be discussed in three categories: magnetic field tunable properties, stress tunable properties and temperature tunable properties. In each section, both the wires and their composites will be discussed. Note that all the composites under discussion here are non-percolating due to either the periodical arrangement of the wires with fixed spacing or the existence of glass coat for random wire composites. Thus there is no concern that the formation of conductive network will hinder the interactions with the microwave.

5.1. Field tunable properties

5.1.1. Field effect on the impedance of single wire

The static magnetic field is essential to generate the GMI effect. With our focus on the gigahertz frequency, the numerous studies on the megahertz frequency (see [7] and references therein) will
be skipped. At high frequency, the GMI effect is believed to be caused by the natural ferromagnetic resonance in the outer layer of wires occurring at a relatively small ac field [26,28,29,227]. The resonance frequency is dependent on the anisotropy field, anisotropy angle and external field (if it exists) [50,55,197,228,229]. At around resonance frequency, the field sensitivity could reach maximum [230]. The typical field effect is shown in Fig. 29. Note that the real (imaginary) part of $Z$ corresponds to the imaginary (real) part of circumferential permeability [28]. Similar effects are also shown elsewhere [18,231–233].

5.1.2. Continuous-wire composites

For continuous-wire composites, the dependence of effective permittivity on the external field is established via the field dependence of plasma frequency according to Eq. (2.21). As plasma frequency is dependent on the interwire-spacing and wire diameter following Eq. (2.16), these two geometrical parameters are critical in governing the effective response of the composite to the external field. Actually this is quite reasonable since these two parameters constitute the basic mesostructure, and are decisive in the dielectric heterogeneous composites [234–236]. Also the tunable properties are largely determined by the local magnetic properties of the wires. Therefore, the following discussion of tunable properties of continuous-wire composites is carried out from these three critical influencing factors, i.e., interwire spacing (also known as cell parameter, periodicity), wire diameter and local magnetic property of wires.

5.1.2.1. Influence of wire periodicity. Fig. 30 displays the dependence of complex effective permittivity on the frequency with magnetic field as a parameter for composites with wire Co$_{68.7}$Fe$_{3}$Ni$_{1}$B$_{13}$Si$_{11}$Mo$_{2.3}$ with different periodicity. The dependence of the effective permittivity is well displayed in both graphs below the corresponding plasma frequency.

It can also be seen that, with increasing wire periodicity from 7 to 15 mm, the frequency dispersion of effective permittivity on the magnetic field is remarkably depreciated as the tunability (defined as the ratio of variation of the electromagnetic parameter to that of the corresponding field [224]) reduced from 0.16 m/A to $7.7 \times 10^{-3}$ m/A. This means that a small wire periodicity is preferable for a field-tunable property. However, a decrease of wire periodicity will increase the plasma frequency and hence the skin effect. If the skin effect is too strong, the field effect will be rather weak. Therefore, the wire diameter may also need to be decrease to compensate the decrease of skin depth. Thereby, there exists an optimum value of wire periodicity matching the diameter for the microwave tunable properties. This is consistent with the theoretical prediction that for $b = 0.5$ mm, the best tunability will be achieved for a wire radius of 5–10 μm [237] (see Figs. 31 and 32).

Fig. 33 shows the frequency dependence of the reflection parameter ($S_{11}$) taken at different magnetic fields for the composites with $b = 3$ mm, 7 mm and 9 mm. It can be seen that the shape of the curves varies remarkably as the wire periodicity increases from 3 mm to 9 mm. For the $b = 3$ mm sample, the spectra can be divided into two frequency zones at 7.6 GHz. Below 7.6 GHz, the reflectivity
decreases as the magnetic field is applied. This is due to the absorption effect. However, the opposite trend is observed for $f > 7.6$ GHz. For the $b = 7$ mm and 9 mm samples, one more zone is found for $f > 16.3$ GHz and $f > 14.4$ GHz. The frequency at which the signal of $S_{11}$ changes with magnetic field is considered the characteristic frequency. It is worth noting here that as $b$ increases from 3 mm to 9 mm, the characteristic frequency decreases from 7.6 GHz to 3.8 GHz (between zones 1 and 2). One can also see that the characteristic frequency (between zones 2 and 3) decreases from 16.3 GHz for the $b = 7$ mm sample to 14.4 GHz for the $b = 9$ mm sample. For the $b = 3$ mm sample the characteristic frequency cannot be determined due to the limited measurement frequency range.

![Fig. 30. Frequency plots of the real and imaginary part of effective permittivity for composites containing long continuous wires with the external field as a parameter. (a) wire spacing $b = 7$ mm; (b) $b = 9$ mm. Reprinted with permission from [365] copyright 2012 Elsevier.](image1)

![Fig. 31. Effective permittivity, real part, as a function of external field for composites containing continuous wires with different wire periodicity $b = 7, 9$ and 15 mm. Reproduced with permission from [365] copyright 2012 Elsevier.](image2)
but it appears to be higher than those for the \( b = 7 \text{ mm} \) and 9 mm composites. This finding points to an important consequence that the characteristic frequency shifts to a lower value for composites with larger wire periodicity in the reflection spectra.

Fig. 34 shows the magnetic field dependence of the reflection parameter \( S_{11} \) taken at 900 MHz. The sensitivity of \( S_{11} \) to the magnetic field is positively correlated to \( b \). For the composites with \( b = 9 \text{ mm} \), \( S_{11} \) falls from \( -1.8 \text{ dB} \) at \( H_{ex} = 0 \) to \( -7.5 \text{ dB} \) at \( H_{ex} = 500 \text{ A/m} \).

5.1.2.2. Influence of wire diameter. It is well known that the wire diameter has a strong impact on the GMI properties of microwires [157,159,238,239]. Fig. 35 shows that composites containing wires with a larger diameter presents a higher field tunability than the other with a smaller diameter, which can
be explained by the wire geometry dependence of the GMI effect and associated skin effect. It has already been demonstrated that, the GMI effect is positively correlated to the wire diameter \[240\]. Accordingly, the dielectric response of the composite containing wires of larger diameter is stronger than otherwise \[43\].

**Fig. 33.** Experimental reflection spectra \((S_{11})\) for composite sample of 640 \(\mu\)m thick and 50 cm \(\times\) 50 cm in-plane size with continuous amorphous wires spaced at 3 mm (a), 7 mm (b) and 9 mm (c). Reprinted with permission from [365] copyright 2012 Elsevier.

**Fig. 34.** Reflection parameter as a function of external field for composites containing continuous wires with different wire periodicity \(b = 3\), \(7\) and \(9\) mm at the initial frequency of 0.9 GHz.
A comparison between the transmission spectra (cf. Fig. 36) reveals that the diameter of the wire has a profound impact on the intensity of $S_{21}$ but much less effect on the tunability. The variation of diameter of several microns has a negligible influence on the plasma frequency of 5 GHz, and the

![Graphs showing S-parameters for different conditions](image)

**Fig. 35.** Frequency dependence of real part of effective permittivity with external field as a parameter for composites containing wires of different radius $a$. Reproduced with permission from [365] copyright 2012 Elsevier.

**Fig. 36.** Frequency dependence of $S$-parameters with external field as a parameter for composites containing wires of different radius. Reproduced with permission from [365] copyright 2012 Elsevier.
frequencies at which $S_{21}$ reaches the minimum are about 3 GHz for both of them. It follows that the plasma frequency probably decides the patterns of transmission spectra.

The phase shift of $S_{11}$ in the presence of an external field (cf. Fig. 37) suggests a promising sensing application of the composite. For CP3, the phase going through $\pm \pi$ is completely suppressed when it is under a small field of 25 A/m. For CP4, the concerned phase shifts to a lower value from 3.7 in the absence of a field to 3.0 in the presence of a field of 70 A/m. These considerable changes suggest the ferrromagnetic microwires enable their composite with a self-monitoring capacity: any stress change that occurs to the wire through the composite can be detected via the microwave tunable spectra.

5.1.2.3. Influence of wire composition. Following from the very strong dependence of the GMI property on the composition of microwires [6,7,157,159], the change of local magnetic properties with the composition is expected to vary the field effect. Fig. 39 shows a comparison between the composites containing wires of same geometry but different composition. Both wire-composites present rather good dispersion properties but differing field effect. This is attributed to the difference in soft magnetic properties as shown in the magnetisation curves of the two wires (Fig. 38).

The scattering spectra for the two composites with periodicity of 7 mm are presented in Fig. 40. In the spectra of $S_{11}$, the two composites possess almost the same characteristic frequencies but differing

![Fig. 37. Frequency dependence of phase of $S_{11}$ with external field as a parameter for composites containing wires of different radius. Reproduced with permission from [365] copyright 2012 Elsevier.](image)

![Fig. 38. M-H curves of Co$_{68.7}$Fe$_{2}$Ni$_{1}$B$_{12}$Si$_{11}$Mo$_{2.3}$ (a) and Co$_{67.05}$Fe$_{3.85}$Ni$_{1.44}$B$_{11.53}$Si$_{14.47}$Mo$_{1.66}$ (b) measured in the field along the wire axis.](image)
field tunability. Comparison of spectra S22 shows that both the shift of resonance and resonance frequency are smaller for CP1.

5.1.3. Short-wire composites

In a short-wire composite, short wire pieces may be uniformly dispersed in a random manner [30,221,222] or in a periodical manner [174,175,177,197,219,241,242]. In this section the magnetic bias (field) effects of short-wire composites are presented and discussed within the theoretical framework detailed in Section 2.2.3.

Fig. 39. Frequency plots of the effective permittivity, real part, for composites containing long continuous wires \((Co_{68.7}Fe_{4}Ni_{1.3}Si_{1.7}Mo_{2.3})\) and \((Co_{67.05}Fe_{3.85}Ni_{1.44}B_{11.53}Si_{14.47}Mo_{1.66})\) with the external field \(H_{0}\) as a parameter. Wire radius \(a = 10 \mu m\); wire periodicity \(b = 7 \text{ mm (upper plot)}\) and \(b = 9 \text{ mm (lower plot)}\).
Fig. 40. Frequency dependencies of magnitude of S-parameters for composite containing amorphous wires Co$_{67.05}$Fe$_{3.85}$-Ni$_{1.44}$B$_{11.53}$Si$_{14.47}$Mo$_{1.66}$ spaced at 7 mm. (a) $S_{11}$, (b) $S_{21}$ and (c) $S_{22}$.

Fig. 41. Effective permittivity spectra of a short-wire composite with varying magnetic field in relative to anisotropy field (500 A/m). The material parameters are given in the graph: $l$ is the wire length, $a$ is the wire radius, $n_c$ is the ratio of wire number to the area containing them.

Fig. 41 shows the dispersion of effective permittivity for a short-wire composite with the parameters $(l,a,n_c)$ detailed in the graph. The value of $\varepsilon'$ is rather small due to the low concentration of the microwires (Fig. 41a). Nevertheless, a dependence of $\varepsilon'$ on the applied magnetic field is
demonstrated. The transformation of resonance to relaxation can be inferred from the frequency evolution of these curves. The anisotropy field can be used as a critical value to distinguish the frequency dependence of $\varepsilon'$ at the studied frequency range (1–5 GHz). The same trend is also observed in the frequency plots of $\varepsilon''$ (Fig. 41b). This is explained as follows. When $H_{ex} < H_k$, the impedance is increased with $H_{ex}$. Therefore, the internal loss increases and the relaxation dispersion occurs. The relaxation behaviour is fully achieved when $H_{ex} = H_k$, whereby the impedance reaches a maximum. Further increase of $H_{ex}$ results in a reverse trend. Note that the dielectric response to the magnetic field is not seen until the field is 250 A/m; this can be attributed to the relative insensitivity of magnetoimpedance for this range of magnetic field.

As with the complex permittivity spectra, the transmission is increased as the field increases with a concomitant resonance–relaxation change, as seen in Fig. 42a. Strikingly, the transmission spectra present a large transmission of ca. 90%, which corresponds to a very large return loss (see Fig. 42b). With the same spectra zoomed at 1–5 GHz (Fig. 42c), the resonance–relaxation transformation was clearly observed with increasing magnetic field. The resonance/relaxation frequency shifts to higher value with increasing magnetic field. The phase shift $\Phi$ is also shown in the reflection spectra as depicted in Fig. 42d.

By decreasing the wire periodicity from 20 mm to 5 mm, the area concentration of wires is greatly increased from 0.06 cm$^2$ to 0.24 cm$^2$. As a result, the values of measured S-parameters are largely increased while their field dependencies remain unchanged (Fig. 43a–d). Inasmuch as the wire geometry (length, diameter and aspect ratio) remains unchanged, there will not be significant changes in

![Fig. 42.](image-url)

(a) Transmission.

(b) Reflection.

(c) Reflection.

(d) Reflection phase.
the dispersion behaviour as far as the composite mesostructure is concerned. It should be noted that, although the phase shift remains unchanged when the field increases from 500 A/m to 1000 A/m, a

![Graphs of various properties](image-url)

**Fig. 43.** Experimental dispersion of (a) real and (b) imaginary part of effective permittivity; (c) measured transmission and (d) reflection spectra of the short-wire composite with the magnetic field as a parameter; (e) presents the phase of reflection coefficient. The material parameters are given in the graph by the same symbols as in Fig. 41.
reduction from 500 A/m to 100 A/m is found to cause a phase reversal between $-\pi$ and $\pi$ in the reflection spectra (Fig. 43e).

5.2. Stress tunable properties

Due to the stress effect on the impedance of amorphous wires, the stress will have significant impact on the prorogation of microwave when they pass through the microwire(s). This is characterised by the variation of electromagnetic parameters (reflection, transmission, permittivity and permeability) with stress. This is the basic working principle for microwave NDT methods [243–249]. Compared to other NDT methods employing ultrasound [250, 251], infrared thermography [251–253], radiography [254, 255], radioactive computed tomography, and ground-penetrating radar (GPR) [256, 257], microwave proves to be advantageous due to the fact that microwaves can penetrate deep inside the composite, scatter little compared to acoustic waves, offer excellent contrast between matrix and reinforcing fibres, have good resolution, are not hazardous, cost-effective compared to radioactive methods, and robust to environmental conditions unlike infrared methods [243–249]. Both near-field [258, 259] and far-field free space [243] characterisation of composite structure have been proved to be useful in detecting the debonds and delaminations of composite structure, demonstrating the usefulness of microwave NDT technology in structural health monitoring applications. However, without embedding sensors, it is hard to detect the local damage. Most recently, the potential of using carbon fibres themselves as antenna and sensors to precisely detect the damage in CFRP has also been demonstrated [260]. However, the interactions of CFRP and microwave are limited by the high conduction loss of CF due to the high volume fraction. The use of microwires as sensor elements is advantageous for their strong interactions with microwave and high sensitivity to external fields. In what follows, we will discuss the stress effect on the wire impedance and the stress tunable effects of microwire composites at gigahertz frequencies.

5.2.1. Stress sensing based on microwires

The stress influence of GMI or stress-impedance effect has been mostly discussed in the megahertz frequencies due to the application interest [211, 261–266]. Interested readers are kindly referred to [7, 160, 6] and the references there. The focus here is on the stress effect of GMI behaviour in the gigahertz frequency.

At gigahertz frequencies, the skin effect is very strong with a small skin depth (e.g. around 1.2 $\mu$m at 1–10 GHz [51]. The permeability is contributed by the outer layer of the microwire. For Co-based microwire with the circumferential domain structure in the outer layer, the permeability is determined by the natural ferromagnetic resonance [227]. According to the Landau–Lifshitz–Gilbert (LLG) equation, the permeability is strongly dependent on the anisotropy field of the wires. Considering the significant stress impact on the anisotropy field of wires [52, 214, 239, 267, 268] and anisotropy angle [50, 55, 228], the permeability (or impedance) spectra can be regulated by the variation of internal stress due to that of geometry [268–271] or glass-removal [272]; and external stress [197] as shown in Fig. 44a. The resonance frequency can be regulated by the parameter of the microwires and the number of wires. Therefore, this effect could be utilised, beyond stress sensing, for detecting and locating damage in the microwire-based composites [260], which is of much interest in engineering applications. In Fig. 44b and c, the sensing resolution is obtained from the shift of resonance with stress/strain with values of 1.06 MHz/MPa and 134.5 kHz/microstrain, respectively. These results lead to an important revelation that the microwires can be used as stress sensors in a wide frequency range provided the permeability can be obtained. Compared with the newly proposed SRR-based sensor with a sensitivity of 5.148 kHz/microstrain [73], the microwires are much more cost-effective and possess a higher Q factor and sensitivity. The susceptibility of permeability to stress can be tailored either by tuning the composition, geometry, and microstructure of the microwires [268] or through developing composites containing magnetic fillers [273] and non-magnetic fillers [274].

It is worth mentioning that the stress sensitivity of impedance for single wire can be modulated by the dc bias field. When the external magnetic field is equal to the value of the anisotropy field, the stress sensitivity reaches maximum. This is demonstrated in Fig. 45 [275], where the maximum sen-
Sensitivity is shown at 15 Oe. The reason for this is well explained by the dependence of permeability on the anisotropy angle, formulated as

\[ \mu = 1 + 4\pi \cos^2(\theta) \chi. \]  

(5.1)

The application of magnetic field along the wire axis increases the circumferential magnetic permeability by rotating the magnetisation vector towards the wire axis. On the other hand, when a stress is applied along the microwires with a negative magnetostriction, the magnetisation vector rotates away from the axis direction. As a result, the circumferential magnetic permeability is decreased [50]. It is expected, therefore, that the application of longitudinal stress will compensate the effect of the magnetic field. The influence of stress is more obvious with a high magnetic field. Indeed, with reference to Fig. 45, the maximum applied stress of 1263 MPa encourages the absorption back to the original value by offsetting the effects of the magnetic field. The magnetic field along the wire axis is desirable for the absorption of microwires and can also be utilised to increase the stress sensitivity of absorption. Similar results were also reported in [50,55]. Upon analysing the data in Fig. 45, it can be obtained that the sensitivity of absorption to stress increases by ca. 39 times when the field increases from 0.75 Oe to 15 Oe. This has profound implication in designing wire-based stress sensors.
5.2.2. Stress tunable properties of composites

5.2.2.1. Stress tunable properties of composites in free space. Fig. 46 [175] shows the complex permittivity spectra for as-prepared intact rubber-based composite prepared by the method described in Section 3.2.2 and after being damaged with the occurrence of wire breakage. There are pronounced changes for both the real part $e'$ and the imaginary part $e''$ of effective permittivity. In particular, a drastic change of $e'$ is seen with a reversal of sign from negative to positive when the wire breakage happened to the composite in question.

For the composites containing Fe$_{4.84}$Co$_{56.51}$B$_{14.16}$Si$_{11.41}$Cr$_{13.08}$, there is no response at all for the composite subjected to a load range from 0 to 4 kg (not shown here). However, a stress tunable behaviour parallel to the field tunable behaviour is observed in the transmission spectra (Fig. 47). Interestingly, the composite shows a similar response to stress and magnetic field, although the evolution of transmission with magnetic field is steadier than that with stress. To gain a deeper insight into such stress tunable characteristics, Fig. 48 is plotted to show the calculated stress (resp. magnetic field) tunability versus stress (resp. magnetic field) at 1, 4.8 and 8 GHz, which are lower, equal and higher in relative to the plasma frequency ($f_p$), respectively. The overall evolution of tunability remains the same trend at all three frequencies. In comparison with the single peak feature displayed in the magnetic field dependence of tunability (Fig. 48b), both a maximum and a minimum appeared in the stress dependence of tunability.

The primary principle pertinent to the stress tunable phenomenon is as follows. For composite containing ferromagnetic wires exhibiting giant magnetoimpedance effect at microwave frequencies, the effective permittivity may depend on a dc magnetic field via the corresponding dependence of the surface impedance. The surface impedance can also be changed by applying a stress which modifies the magnetic anisotropy and domain structure in wires. Thus, the effective permittivity may also depend on the external stress or strain. It follows that the stress-impedance (SI) property of microwires is critical to the susceptibility of the whole composite to the stress. SI depends strongly on the magnetoelastic characteristics of the microwire, which are conditioned by a number of factors: composition, domain structure, geometry, etc. This accounts for the observed insensitivity and sensitivity of the microwire composites in terms of their permittivity to the external stress when one deals with different wires. Although, all these amorphous wires can be expected to show a drastic change when breakage occurs in the composite, the choicest wires have to be evaluated when it comes to a more delicate stress sensing application.

The single peak presented in stress tunability of $S_{21}$ ($n_{S_{21}}(H)$) is associated with the anisotropy field. By contrast, a more complex relationship of $n_{S_{21}}(\sigma)$ merits more discussion. It is well

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**Fig. 45.** (a) Axial field dependence of absorption in presence of varying stress at 9 GHz for a Co$_{67}$Fe$_{3.9}$Ni$_{1.4}$B$_{11.5}$Si$_{14.5}$Mo$_{1.7}$ wire of total diameter 30.6 $\mu$m and glass coat 5.2 $\mu$m. (b) Tensile stress dependence of absorption (impedance) at varying magnetic fields for the same wire. Reprinted with permission from [275] copyright 2011 Elsevier.
established that the Co-based microwires with a negative magnetostriction have a bamboo-like domain structure, consisting of an inner core and an outer shell [7]. The surface impedance depends on the circumferential anisotropy at the outer shell. When a stress is applied along the axis of a microwire with negative magnetostriction, a magnetoelastic field is induced in the circumferential direction and this drives the spins rotating toward that direction. As a result, the circumferential magnetic permeability is decreased and hence the surface impedance is also reduced. It is expected, therefore, that the application of a longitudinal stress will compensate the effect of the magnetic field. This explains the maximum that occurred at around 13 kPa (Fig. 48a). Afterwards, with increasing stress, the well defined circumferential anisotropy may remain unchanged and hence the surface impedance shows very little variation to the incremental stress, giving rise to a minimum of tunability at 40 kPa. Larger stress than 40 kPa may depreciate the circumferential anisotropy and increase the surface magnetoimpedance, which accounts for the recovery of the increased tunability with stress.

It should be noted that tens of kPa imposed on the composite yields hundreds of MPa on each wire, according to a simple calculation as follow. As the present case meets the isostrain condition, the following equation holds

\[ \varepsilon_c = \frac{\sigma_c}{E_c} = \varepsilon_w = \frac{\sigma_w}{E_w}, \]

where \( \varepsilon_c \) and \( \varepsilon_w \) denote the strain for composite and microwires, respectively; \( \sigma_c \) and \( \sigma_w \) the stress exerted on the composite and microwires, respectively; \( E_c \) and \( E_w \) the Young's modulus of the composite and microwires, respectively. Using the law of mixture, the stress each microwire experiences is given by

![Complex permittivity spectra of as-received composite and after damage by tensile stress.](image)

Fig. 46. Complex permittivity spectra of as-received composite and after damage by tensile stress. Reprinted with permission from [175] copyright 2011 AIP.
Fig. 47. Effect of stress (left) and magnetic filed (right) on transmission spectra of microwire composite containing Fe₄Co₉₈.₇Ni₁₂Si₁₁Mo₂.₃ microwires. Reprinted with permission from [175] copyright 2011 AIP.

Fig. 48. (a) Stress and (b) field dependence of tunability at 1, 4.8 and 8 GHz, plasma frequency $f_p = 4.8$ GHz. The ordinate profiles are normalised by $10^{-4}$ and $10^{-5}$, respectively. Reprinted with permission from [175], copyright 2011 AIP.
\[ \sigma_w = \frac{\sigma_c}{(E_m/E_w)f_m + f_w}, \]

where \(f_m\) and \(f_w\) are the volume fraction of the matrix and microwires, respectively. Due to the significant difference between the Young’s modulus of rubber matrix (2 MPa) and of microwires (100 GPa), 10 kPa on the composite can result in 500 MPa on the microwires. This is within the reasonable stress range as commonly discussed in literature, in terms of the stress effect on GMI properties of microwires (see, e.g. [53,239]).

5.2.2.2. Stress influence of electromagnetic properties measured by spectroscopy. For composite containing ferromagnetic wires exhibiting giant magnetooimpedance effect at microwave frequencies, the effective permittivity may depend on a dc magnetic field via the corresponding dependence of the surface impedance. The surface impedance can be changed by applying a stress which modifies the magnetic anisotropy and domain structure in wires. Thus, the effective permittivity may also depend on the external stress or strain. Following the stress tunable theory proposed by Makhnovskiy and Panina [12], Qin and co-workers [174,276] approached the strain effect on the electromagnetic responses from the technological aspects of the multifunctional composites.

Co56.51Fe4.84B14.16Si11.41Cr13.08 glass-covered magnetic microwires having a total diameter of 29.4 mm and silicone rubber were used for the preparation of composite materials. Continuous microwires with the same length of 70 mm were embedded in a parallel manner into the silicone rubber matrices, which were bonded by silicone resin (see Section 3.2.2 for further details). The number \(n\) of the microwires in composites varied with \(n = 6, 12, 17\) corresponding to the wire spacing \(d = 2, 1, 0.8\) mm. For comparison, copper wires with a diameter of 60 mm were also used as fillers. The resultant composites are of uniform dimension of 70 mm \(\times\) 13 mm \(\times\) 1.8 mm.

The electromagnetic measurement was carried out with a wave vector of the electromagnetic field perpendicular to the wires using a modified microwave frequency-domain spectroscopy. The quasi-TEM transverse electromagnetic mode, which is the only mode that propagates in the structure, is obtained by using a modified microwave frequency-domain spectroscopy. The Nicolson-Ross procedure for the transformation of the load impedance by a transmission line, are determined by the transmission \(S_21\) and reflection \(S_{11}\) parameters. A vector network analyser (Agilent, model H8753ES) with SOLT calibration was used to measure the 5 parameters of the cell containing the sample under test within the frequency range between 300 MHz and 5 GHz. Details of the instrumental were discussed in Section 2.2.2.2. All measurements were done at ambient temperature.

Fig. 49 shows the permittivity spectra for composites containing different amount of microwires and 12 copper wires. For the unstressed sample, the real component of permittivity \(\varepsilon'\) increases with the wire amount. For the same amount of wires, composite containing copper wires displays a larger \(\varepsilon'\) than that with ferromagnetic microwires. The same trend remains when the strain reaches 2.4% as the tensile stress is applied along the longitudinal direction of the composite. Noticeably, there appears a broad relaxation peak at about 4 GHz for \(n = 12\) sample when \(\varepsilon''\) starts to show relatively stronger stress sensitivity, which increases with the wire amount. The sample with copper wires presents a sharper peak at 4.5 GHz. As \(n\) increases to 17, the peak position and the evolution trend with the strain remain unchanged, the shape becomes asymmetric, though. In contrast, the sample with the copper wires exhibits a complex evolution of the resonance peak with...
the strain, which is the resonance peak occurring at the unstressed state increases with a small strain of 2% but disappears at larger strains. To exclude the influence of geometrical factors of the composites and the microwires, Fig. 52 was plotted to show the frequency dependence of $\tan \delta$ (ratio of $\varepsilon''$ to $\varepsilon'$), and the evolution of peak features with strain remains the same trend as in Fig. 51.

Strain dependence of effective permittivity is quantitatively analysed by the Gaussian molecular network model (GMNM) [277]. Fig. 53 shows $\lambda$ dependence of $\Delta$ with varying amounts of wires, where $\Delta = (\varepsilon'(\lambda) - \varepsilon'(\lambda = 0))/\varepsilon'(\lambda = 0)$. This relationship is well fitted by a functional form $k(1 + \lambda - 1/(1 + \lambda)^2)$, where $k$ is a constant, the value of which varies with the wire amount as shown in Fig. 53.

The effective permittivity ($\varepsilon$) is dependent on the wire concentration ($p_v$) and the averaged polarisability ($\langle a \rangle$): $\varepsilon = \varepsilon_m + 4\pi p_v \langle a \rangle$, where $\varepsilon_m$ denotes the permittivity of matrix [12]. Thus, increasing the wire amount improves the effective polarisation and hence the permittivity. The polarisability is primarily dependent on electric excitation. For the present composite configuration with microwires perpendicular to the electric field vector, although the sample is aligned with the tensile axis of the deformation apparatus, an axial component of electrical field still exists due to the inevitable misalignment of the wires within the sample and/or the possible inhomogeneity of the electrical field in the cell which may affect the electric excitation of the wires. This accounts for the observed dielectric response of the composite samples due to the polarisation and the induced circumferential magnetisation of the wires. For the magnetic microwires, the strain modifies the magnetisation process and hence the circumferential permeability, resulting in the changes of $\varepsilon'$. However, since the experimental data were obtained using the same protocol and measurement cell, the changes of permittivity can be solely attributed to strain.

It is also shown that more microwires induce a sharper peak at ca. 4 GHz, indicating an increased stress sensitivity. However, this argument does not hold true for the composites with $n = 6$ and copper wires which can be understood as follows: for $n = 6$, the wires concentration is not high enough to
satisfy the response of a noticeable peak to the external stress; for copper wires, they have no response to the induced circumferential magnetic field and therefore the composite does not show any stress-induced peaks. Overall, the effective permittivity of wire composites increases with the strain, which is further discussed later based on the GMNM approach. It is worth pointing out that, owing to the

![Graph](image_url)

**Fig. 50.** Strain dependence of $\varepsilon'$ for composites with $n$ as a variable.

![Graph](image_url)

**Fig. 51.** Spectra of $\varepsilon'$ for composites containing ferromagnetic microwires $n = 6, 12, 17$ and $12$ copper wires with strain $\lambda$ as a parameter.

satisfy the response of a noticeable peak to the external stress; for copper wires, they have no response to the induced circumferential magnetic field and therefore the composite does not show any stress-induced peaks. Overall, the effective permittivity of wire composites increases with the strain, which is further discussed later based on the GMNM approach. It is worth pointing out that, owing to the
higher concentration of copper wires as a result of larger diameter than that of ferromagnetic microwires, the composite sample with copper wires shows a larger permittivity than that with the same amount of ferromagnetic microwires.

From the application point of view, it is important to find ways to improve the stress sensitivity. Obviously, this can be approached by increasing the amount of microwires in the composite. However, it should be noted that it does not necessarily mean that more is better. In this work, the sensitivity of permittivity to stress showed little change between the \( n = 12 \) and \( n = 17 \) samples. Somewhat surprisingly it is found that the samples containing magnetic microwires and that containing non-magnetic microwires appear to yield a similar stress sensitivity of \( \varepsilon'' \). This observation suggests an independence of the stress sensitivity in wire composites to the conductivity and magnetic permeability of the wires for a sufficiently high concentration of wires, although the reason for this result is not obvious.

The most striking feature shown in Fig. 51 is the evolution of the peak with the stress for \( n = 12 \) and \( n = 17 \) samples. It is proposed that the gradual spectral change from relaxation to resonance at 4.5 GHz can also be attributed to the influence of stress on wire magnetisation as discussed above, which modifies the eddy current loss and contributes to the steady evolution of the \( \varepsilon'' \) resonance due to the circumferential ferromagnetic resonance.

The loss tangent (\( \tan \delta \)) retains the same changing trend with the strain as \( \varepsilon''(\lambda) \), indicating that such a strain effect is independent of any geometrical factors [278]. It should be noted that such a

![Fig. 52. Frequency dependence of loss tangent with strain as a parameter. Reprinted with permission from [174] copyright 2010 AIP.](image)

![Fig. 53. Variations of \( \Delta \) with strain for composites with differing amount of wires as shown in the open symbols at 4 GHz. The best fitted lines to the function of \( k(1+\lambda-1/(1+\lambda)^2) \) and values of \( k \) corresponding to different \( n \) are also shown. Reprinted with permission from [174] copyright 2010 AIP.](image)
transformation of relaxation to resonance is absent for the \( n = 6 \) sample and composite with copper wires in the spectra of complex permittivity. This suggests the exclusivity of ferromagnetic microwires and the requirement of wire concentration to realise a steady evolution of the peak feature defined by the stress [221].

Now we discuss the strain-permittivity relationship. Basically the GMNM approach is related to the elasticity network of the composite material. Excellent agreement between the experimental results and the GMNM model is observed for all samples (Fig. 53), which suggest the applicability of the model at measured strain range. By introducing more microwires \((n \leq 12)\) into the rubber matrix, a higher stress sensitivity (characterised by \( k \) value in this case) and a closer experimental-model match is obtained. But when \( n \) increases to 17, \( k \) is decreased and a relatively larger mismatch is observed. This can be attributed to the enhanced complexity of the composite mesostructure arising from the larger amount of embedded microwires [277]. Nevertheless, due to the periodical topology of the microwires within the composite, GMNM gives fairly good predictions for all samples.

The significance of wire patterns is manifested in Fig. 54a, in which a nonlinear dependence of \( \varepsilon'' \) on the strain is shown. In this case, the wire starts to snap when the strain exceeds 2.8%. Due to the uneven interfacial properties, each single wire may experience different stress, which results in partial but not total fracture of all wires. We then receive a similar resonance response for two patterns (Fig. 54b). This indicates that the increase of dielectric loss with the stress is compensated with the opposite effect resulting from the reduction of strained wires. There are two possible mechanisms involved in this phenomenon. First is the stress effect: the stress changes the current distribution in the wires and induces higher dielectric loss, and release of stress results in the opposite effect. Second is the shape effect: the wires are fractured to relatively shorter pieces and the anisotropy field of the wire becomes non-uniform and results in the reduction and broadening of resonance linewidth. This leads us to a striking revelation in a larger context that the for functional fillers enabled heterogeneous composites, the macroscopic behaviour is dependent on the collective response of the fillers on one hand, and each single filler on the other hand. This then poses the challenge of how to manipulate some, if not each, of the fillers. In so doing, we will have greater control of the properties of the composite presented to meet specific applications.

5.3. Temperature tunable properties

By analogy with stress, the temperature tunable properties are derived from the temperature dependence of GMI properties. Because the magnetic permeability is sensitive to temperature, the GMI changes rapidly as a function of temperature, especially in the vicinity of the Curie temperature \( T_C \). In general, for Co-based microwires, the GMI effect first increases with increasing measuring temperature due to the internal stress relief and reaches a maximum value near the Curie temperature of the material, then finally decreases at higher temperatures [279,280]. The dramatic change of GMI
at temperatures above the Curie point was attributed to the collapse of the magnetic coupling in the material [281], which has been exploited for the development of temperature sensors [282].

In terms of composites, the magnetic phase transition at \( T_c \) will lead to a large transformation of dispersion of effective permittivity as well as the reflection and transmission coefficients [41]. Fig. 55 shows the theoretical permittivity spectra and transmission/reflection spectra for \( T < T_c \) and \( T > T_c \). By analogy with the influence of stress or magnetic field, there is an anomalous dispersion of \( \varepsilon'(f) \) and relaxation-resonance transformation when \( T \) exceeds \( T_c \). All these features demonstrate strong temperature effects, which can be exploited for temperature sensing. A very promising application is to embed these microwires into the preform of polymer laminates. The microwires can then serve as self-regulating heating elements in the microwave curing process, which is believed to be much more efficient than conventional curing methods [283]. Meanwhile, with proper choice of composition and tailoring, the wires can be made highly sensitive [284] to the temperature to be applied for process monitoring, which remains a challenging issue in composites manufacture [285]. In addition, the wires remain as stress sensing elements in the cured composites for various kinds of structural application. With increasing use of advanced fibre-reinforced composites in industry [286], the addition of microwires may well renovate the composite industry to some extent.

Fig. 55. (a) Calculated dispersion spectra of resonance complex effective permittivity in the vicinity of antenna resonance under the influence of temperature, the inset shows the schematic configuration of the composite. (b) Typical transmission and reflection spectra under the influence of temperature. (c) Typical reflection phase spectra under the influence of temperature [41].
Thin conducting wire structures are common building blocks for preparing metamaterials with negative permittivity of a range of unusual properties. This has generated a considerable interest in wire media and vast literature is devoted to the subject (see e.g. [287–294]). The negative electrical response also suggests that the wire medium is characterised by a low frequency stop band from zero frequency to the cutoff frequency which is often referred to as plasma frequency [295,296]. For the wire radius in micron scale, and lattice constant in millimeter scale, the plasma frequency is in the gigahertz range. In this frequency band, a strong dispersion of the effective permittivity may be used to engineer a specific electric response. However, a single array of non magnetic wires cannot provide negative magnetic permeability, so to obtain simultaneously the negative permittivity and permeability, non-magnetic wires are often combined with another array of SRRs [61]. Such a design suffers from the drawback of having relatively large dimensions and is not suitable for making into a complex shape when required, e.g., to be made into a coating on a curved surface. Another disadvantage is that such design is anisotropic and the negative refraction is limited to only a couple of polarisations of incident plane electromagnetic wave [297–300], impeding interesting applications such as perfect lens [58], where isotropic metamaterial is needed. Most recently, alternative approaches have been proposed capitalising on the magnetic properties of magnetic materials such as ferrites [301,302], yet a combination of non-conducting wires and magnetic materials is still needed. In this context, to pursue the most simplified design, single ferromagnetic wire or their array is proposed to provide simultaneous negative permittivity and permeability, in that the negative permeability can be obtained at frequencies between natural ferromagnetic resonance and antiferromagnetic resonance [231–233,303,304], while the negative permittivity can be obtained below the normalised plasma frequency $f = f_p/\sqrt{\varepsilon_m}$ [48,12]. Thus, as long as the ferromagnetic resonance frequency is not higher than the plasma frequency in the continuous-wire case, or the anti-antenna resonance in the short-wire case, the simultaneous negative permittivity and permeability can be obtained. With a square net configuration, the microwire composite will present isotropic performance.

Theoretically, a calculation is given here to illustrate the possibility of obtaining negative permeability and permittivity. For the configuration shown in Fig. 57a, the permeability can be expressed as [305]

$$\mu_{\text{eff}} = \frac{1}{2} \frac{(\omega_0 + \omega_m)^2 - \omega^2}{(\omega_0 + \omega_m)^2 - \omega^2} \left( 1 + \sqrt{\frac{\sigma}{\omega \epsilon_0} \frac{2\pi a}{b} \left( \frac{a}{b} \right)^2} \right) + 1 \right) \quad (6.1)$$

where $\omega_0 = \gamma H_{dc}$ with $\gamma$ the gyromagnetic constant and $H_{dc}$ the external field. $\omega_m = 2\pi(2\pi a/b)^2\gamma M$ with $M$ the saturation magnetisation of wires of radius $a$ and interwire spacing $b$ and a bulk conductivity $\sigma$. For typical values of Co–Fe–Cr–B–Si wire, $\sigma = 10^{16}\text{C}^{-1}$, $M = 500\text{Gs}$, when $a = 10\mu\text{m}$, $b = 1\text{mm}$, $H_{dc} = 10\text{Oe}$, the ferromagnetic resonance is 704 MHz according to $f_r = \mu_0 r \sqrt{H_{dc} M_s}/2\pi$ [108]. The normalised plasma frequency is 38.9 GHz, when the matrix permeability is 2. In this case, both negative permeability and permittivity are obtained at $1.9$–$21.7\text{GHz}$ [305]. It should be noted that by regulating $a$, $b$ (within the limit) and magnetic bias, the negative index range can be tuned. This could be of great use from the application point of view.

The capability of microwires to realise metamaterial features is demonstrated amazingly in a single wire. Labrador et al. [231] tested a single wire of nominal composition Fe$_{77.5}$Si$_{12.5}$B$_{10}$ in a waveguide by parallelising it with the electric field vector on slices of Rohacell foam under a dc magnetic field. Both negative permeability due to the natural FMR and negative permittivity as an effect of interactions with the waveguide are realised in the X-band. Note that different from the case of short-circuited microwires with waveguide, the electrically isolated microwire behaves as capacitive-loaded, responsible for negative values of the effective permittivity. As the natural FMR occurs at zero magnetic field, magnetic field is then not an essential element in this metamaterial system, which greatly simplifies its structure. On the other hand, application of magnetic field could also add the tunable functionality.
Such a self-contained and versatile fine element is hence established as promising metamaterial building block to configure a series of metamaterials, which are discussed below.

Different approaches were developed to realise the metamaterials based on ferromagnetic micro-wires. Adenot-Engelvin et al. [306,307] fabricated the wire composite as schematically shown in Fig. 56a using CoFeSiB wire with a small negative magnetostriction coefficient of total diameter 9 μm and core diameter 4 μm. The volume fraction of the wires is within the range of 6–11%. The microwave permeability for this composite is shown in Fig. 56b fitted by a model based on the solenoid approach with a unique set of values for resistance (R), inductance (L) and capacitance (C).

The permeability is also found, in this configuration, to be dependent on the loops, in terms of the resonance frequency by analogy with the magnetic field effect [229,232] or stress [197]. Such field effects can be readily explained by the LLG model for computing the magnetic dynamic susceptibility.

In another configuration (see Fig. 57a) [232] constituted by wire arrays of CoSiB with a diameter of 2 – 3 μm, the transmission spectra were obtained as shown in Fig. 57b exhibiting the rise of transmission with the dc field due to a double negative condition obtained. Although there is no matrix involved, such wire arrays demonstrate the potential to make metamaterials from these wires.

Liu et al. [173] proposed a metamaterial configuration by combining the long conductive fibres along the electric field and microwires along the magnetic field as shown in Fig. 58a. As expected, the negative refraction index is shown at a certain frequency range as shown in the numerical results (Fig. 58b), although the significant resonance loss remains a problem for its application.

In comparison to the composites mentioned above, the prepreg-based composites possess much better mechanical properties in addition to the metamaterial particularities [177]. Indeed, regardless of the matrix, the wire arrays alone [232] or simply bonding the wire arrays on the paper [237,241] are able to show negative permittivity (see Fig. 59). But the sine qua non for composites in engineering application is their structural function. In this sense, the proper choice of matrix, fabrication and the resultant structural performance need to be addressed carefully to obtain a truly applicable multifunctional composite.

A very important sine qua non for realising the metamaterial feature at microwave frequency is that the diameter of microwire is comparable to the skin depth, too large a diameter will cause huge reflection [47,237,304]. This argument generally holds true for any functions based on microwave–material interactions other than EMI shielding dominated by reflection. For typical ferromagnetic microwires, σ = 10^{16} S^{-1} (10^5 S/m), μ = 20, the calculated skin depth δ at 10 GHz is about 1 μm [49]. Since the permeability decreases with the frequency, δ changes little. This is larger than even very thin microwires with typical radius of few microns. Although it is still possible to penetrate into most of the inner core in the Fe-based wire [171] or outer shell in the Co-based wire [308], the submicron wires [309–314] or nanowires [315–326] would be preferred in this case. On the other hand, reducing the wire diameter will reduce the volume fraction of wires and hence the permittivity and permeability. One may have to accept that such an unavoidable loss is typical for metal metamaterials [327].

Fig. 56. (a) Schematic view of a sample with 11 metamaterial blocks. (b) Measured permeability and model with R = 0.1Ω, L = 0.09 nH, C = 9.5 pF for the eight-loops wire composite. Reprinted with permission from [306] copyright 2006 Elsevier.
Another restriction is set on the ‘dilute’ condition, i.e., \( b \gg a \), which is necessary to have a relatively smaller carrier density and large effective carrier mass, such that the plasma frequency can be regulated in the 1–10 GHz of application interest. The band stop or band pass filter, for example, can be designed based on the criticality of plasma frequency on transmission \([47,305]\). Also, it is argued that a large number of wires is deleterious since it will absorb most of the electromagnetic wave \([197,304]\). This issue must be addressed before the microwire composites can find metamaterial applications such as cloaking. On the other hand, it is desirable for microwave absorption application. As such, these two functionalities cannot be pursued simultaneously, but this would greatly extend the freedom of tailoring the electromagnetic properties of the microwire composites.

6.2. Microwave absorption capacity of microwire composites

The high-frequency absorption behaviour of amorphous ferromagnetic materials, among others, is of sufficient interest for microwave absorber applications \([229]\). Since amorphous glass-coated microwires possess small dimension (1–30 \( \mu \)m in diameter), high electrical conductivity (\( \sim 6 \times 10^5 \) S/m), high magnetic permeability (\( \sim 10^3 \)), and high mechanical strength (\( \sim 10^3 \) MPa), they can be incorporated into polymer-based composites for creating high-performance microwave absorption \([171,172,176]\) or EMI shielding \([328]\) composite materials. It should be noted that, for microwire
composites, the EMI shielding of interest to us is absorption-dominated shielding [328]. In this sense, we will focus on the discussion of microwave absorption of microwire composites, which is determined by its electromagnetic constitutive parameter, namely, permittivity and permeability through the intrinsic properties of microwires and the mesostructure. The following discussion is carried out based on the different absorption mechanism, i.e. dielectric loss dominated and magnetic loss dominated absorbing.

6.2.1. Dielectric loss dominated absorbing

In the case of dilute composites, the microwire composites exhibit high complex permittivity but a close-to-unity permeability with negligible magnetic loss at gigahertz frequency [12,172,221]. It follows that the absorption feature is mainly determined by the relaxation polarisation.

The concentration of wire amount plays an important role in this case. It has been shown that an increasing amount of wires will improve the absorption [328]. However, it is reported that rather than simple linear dependence of absorption on filler content, there exists a threshold value at which the percolation network is formed if the glass coats at the end of wires were spalled, which is often the case [172] (see Fig. 60a). This is common in the percolating composite systems (see, e.g. [114,329,330]). In detail, when the wire content is smaller than the percolation threshold, the loss

---

**Fig. 59.** Effective permittivity spectra of Co66Fe3.5B16Si11Cr3.5 continuous wire arrays deduced from the scattering spectra with the external magnetic field as a parameter (wire radius $a = 10 \mu m$, spacing between wires $b = 5$ mm). Reprinted with permission from [314] copyright 2011 John Wiley & sons.
tangent increases but without significant increase of dielectric loss as the wire content is increased, whereas further increase of wire concentration gave a sharp increase of dielectric loss due to the wave reflection rather than absorption. This effect overshadows the contribution of increasing loss tangent to the microwave absorption and results in the decrease of microwave absorption. Note that the tunnelling effect is responsible for the conductivity of the composite before the percolating threshold, which is highly desirable for microwave absorption. It should be mentioned that, at this point, micro-wires are not as good as ferrite due to its much higher conductivity, which otherwise would free our concerns on the concentration limitation to preserve the dipole nature [331]. To address the conflict between increasing wire concentration and percolating network, superior glass quality is the key. Therefore, it reveals to us, in addition to the quality of the metallic core, good glass quality is necessary for further improving the microwave absorption. Indeed, it is shown that the percolation threshold decreases due to the decrease of the length of naked metallic core via annealing but the level of maximum absorption is retained. It can then be expected that with a further increase of annealed wire concentration, the absorption can be increased.

6.2.2. Magnetic loss dominated absorbing

In the case of microwire composites with heavy loading, the ferromagnetic resonance may shift to gigahertz frequency due to the enhanced effective anisotropy field via the long range dipolar interactions between wires [171,305]. In this case, the ferromagnetic resonance for the planar microwire composites is given by [305]

\[
\begin{align*}
    f_r &= r \sqrt{4 \pi M_s (H_k + H_n)}, \\
    H_n &\propto iM_s (a/l)^2,
\end{align*}
\]

where \(H_n\) is the created field by neighbouring wires of total number \(i\), or it can be simply understood as an additional anisotropy field induced by wire interactions [180]. Thus, any factors that can influence the anisotropy field should be considered tuning parameters. Directly from Eq. (6.2), it is obtained that a larger aspect ratio \((l/2a)\) will reduce the resonance frequency and increase the absorption according to Snoek’s law; and that the increasing number of wires will enhance the permeability and neighbouring field, and hence the resonance frequency. For Co-based wires, the decrease of metal-to-total diameter ratio \(p\) will elevate the internal stress and consequently the anisotropy field [160,332], thus the absorption should be shifted to a higher frequency. This has been very well analytically explained by Baranov [333]. The dependence of resonance frequency can be expressed as

\[
f \approx f_0 \sqrt{\frac{1 - p^2}{1 + 1.5p^2}} \text { (GHz)}
\]

where

\[
f_0 = 1.5 \sqrt{2} \times 10^6 \text { (GHz)}
\]

Three kinds of microwires of typical positive, negative and vanishing magnetostriction constant with correspondingly different composition [334] are chosen here to shed light on the usefulness of this theory. By using Eq. (6.3), the metal-to-total diameter ratio \((p)\) dependence of resonance frequency is calculated for these three wires. The result confirms the role of wire cross-sectional geometry on the natural ferromagnetic resonance frequency and also reveals that the iron-based wires are more suitable for absorption purpose at relatively higher gigahertz frequencies. It should be, however, noted that the resonance frequency can be shifted to higher frequency if a large enough neighbouring field is yielded with a good number of wires added into the absorbing media or tailored wire geometry (Fig. 61). Another proved method is to make a multilayer structured microwire film that will enhance the anisotropy field [335] and give an improved initial permeability as high as 6000 at 1 MHz. With reduction of the wire length, the demagnetising field increases, and the anisotropy field are reduced accordingly [171,180,204,328], resulting in reduction of absorption via the collaborative effect with
the influence on the neighbouring field $H_n$. In general, optimal dimensions for excellent absorption performance requires a metallic core of diameter 1–3 μm and length of 1–3 mm comparable to the half wavelength for microwires.

Before closing this session, several important aspects of implementation of microwires on absorbers should be highlighted. First, as the anisotropy constant of microwires can be conveniently modulated by the magnetostriction constant that is subject to wire composition and stress conditions (either internal stress from different geometry or external stress applied), tuning of natural FMR resonance and associated absorption features such as absorption maximum and bandwidth is therefore readily accessible. Second, the dispersion of microwires (i.e., mesostructure control) is critical to meet the requirement of suitable dimensions for maximum absorption. This has been experimentally proved in Ref [336]. Tunable absorption can be realised in the absence of magnetic field and prefers a ultra-thin metallic core, making the wires attractive for miniaturization of microwave devices based on these fine elements. The last important aspect meriting our attention is the usage of wires without glass coats. Although they are inferior in terms of as-cast wire quality due to the fabrication limitation

Fig. 60. (a) Fig. 7. Morphology and metal core contact of the short-cut microwires: (a) as-cast; (b) annealed at 450 °C; (c) annealed at 530 °C; (d) metal core contact (as-cast). (e) Calculated reflection losses of planar composites filled with as-cast and annealed microwires (with filling ratio of 15% and thickness of 1.5 mm.) Reprinted with permission from [172] copyright 2010 Elsevier.
as compared to glass-coated wires, it is the metallic core rather than glass coat that interacts effectively with the microwave. As such, applying metallic core only into the absorbing matrix would improve the packing density of the wires [335] and hence the absorbing rate and efficiency.

7. Potential applications

The ultimate goal of developing new materials is for practical applications. Based on the structure and properties of microwires and their composites presented and analysed in previous sections, it is now possible to propose relevant applications. As early as 1991 when the GMI was not defined, a report on the application of GMI was published [337]. From then on, GMI materials have been under idealisation and development for sensor applications taking advantage of their high sensitivity to external field, driving current frequency and tensile stress. Many new types of sensors using GMI effect have been proposed, increasingly widening the application of the GMI. Hitherto, a variety of devices based on GMI microwires, such as magnetic field sensors [338–341], stress sensors [145,211], current sensors [342] and position sensors [340,343], have been commercialised. The detailed mechanism of microwire-based GMI sensors has been detailed in Refs. [7,31,160,161]. Here we briefly summarise the sensing application of microwires as shown in Table 2. In this Chapter, some potential applications are proposed for microwires composites as mapped in Fig. 62.

Thanks to a remarkable microwave tunable dispersion of the electromagnetic parameters by external magnetic fields, stress and temperature, microwire composites find a wide range of applications in, to name but a few, smart coatings, microwave absorbers, non-destructive testing (NDT), stress sensing, structure monitoring, process monitoring and implant accessories (see, e.g., [12,41,174,308,314,328,344]). They are discussed in the following sections.

7.1. Microwave applications

By analogy with the waveguide system containing ferromagnetic wires, the microwire composites have wide application as microwave electronic components [345]. Owing to their metamaterial properties, the microwire composites can be used for general applications of left-hand materials such as high-performance frequency selective surface (FSS) and quality band stopper. Based on the remarkable tunable effect, they can also be applied as tunable filters and phase shifters [43,51]. Their low cost and susceptibility to mass production, combined with their exceptional absorbing capacity in relative to low volume fraction, mean that they can be designed into various shapes of absorber for civil uses, for example, to improve the electromagnetic compatibility of electronic devices and protect them and human bodies from EM and noise pollution, and also for military use as a means to realise camouflage.
of aircrafts or ships by absorbing radar waves. Their military application has been successfully performed by Micromag (Spain), who commercialised the excellent reduced radar cross section (RCS) technology in air, land and sea vehicles. In 2009, a mixed coating consisting of the wires within regular paints, was implemented on the surface of a patrol ship of Spanish navy (see Fig. 63) and achieved significant reduction of RCS leading to increased survivability from 45% to 90% at best [346].

<table>
<thead>
<tr>
<th>Applications</th>
<th>Types of GMI sensors</th>
<th>Features and/or functions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Target detection and traffic control</td>
<td>Position sensors [370]</td>
<td>Low fabrication cost and simple setting; superior to optical based monitoring devices</td>
</tr>
<tr>
<td></td>
<td>Wireless magnetic field sensors [371]</td>
<td>Remote control of industrial process</td>
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<td></td>
<td>Nano-GMI sensor [372]</td>
<td>Measure electric field in high speeds for automobiles</td>
</tr>
<tr>
<td>Aerospace research and application</td>
<td>Magnetic field sensors</td>
<td>Measure ambient magnetic field vector in space</td>
</tr>
<tr>
<td></td>
<td>Gear-tooth sensor [373]</td>
<td>Control speed and position of gears; improve flight safety</td>
</tr>
<tr>
<td>Electronic compasses and information equipment</td>
<td>GMI electronic compasses [371]</td>
<td>Small size and low power consumption</td>
</tr>
<tr>
<td></td>
<td>G motion sensors</td>
<td>Detect both geomagnetism and gravity [375]</td>
</tr>
<tr>
<td>Non-destructive crack detection</td>
<td>Magnetic field sensors</td>
<td>Superior to GMR in terms of sensitivity [376]</td>
</tr>
<tr>
<td>Biomedical and health control</td>
<td>GMI amorphous wire sensor</td>
<td>Capture crack regions [377]</td>
</tr>
<tr>
<td>Stress sensor applications</td>
<td>Brain position sensors [378,379]</td>
<td>Examine function of brain and detect tumour, if any</td>
</tr>
<tr>
<td></td>
<td>Biological detection sensor [380]</td>
<td>Fast identification and diminution of direction threshold of pathogens or other targeted biomolecules</td>
</tr>
<tr>
<td></td>
<td>GMI torque sensor [381]</td>
<td>Simple construction, high accuracy, low power consumption and easy installation</td>
</tr>
<tr>
<td></td>
<td>SI-CMOS-IC multi-vibrator [382]</td>
<td>Very high sensitivity, sense seismovibration of bridges due to cars passing</td>
</tr>
</tbody>
</table>

**Table 2**

**Applications of GMI sensors.**

**Application Map**

**Fig. 62.** Application map of ferromagnetic microwires and their multifunctional composites [218]. Courtesy of Dr. Makhnovskiy of Plymouth University.
The targeting operation frequency range could be met by regulating the wire geometry and patterns. Yet it should be noted that the diameter of microwire also limits its application for higher frequencies. At terahertz (THz), for example, one has to resort to nanoparticles for any microwave application.

7.2. Structural reinforcement and health monitoring

In civil and aeronautical engineering, the microwire can be used as reinforcing elements to make polymer matrix composites with enhanced mechanical and/or thermal properties as discussed in Section 4. In addition, they can also be applied to reinforce glass matrix [347]. Due to its achievable high ductility as demonstrated by Donald et al. [348] (Fig. 65), the microwire glass-matrix composite is expected to meet the requirement of specific application where high toughness and resistance to spalling is necessary.

Another promising application for microwire composites is the evaluation of structural materials by detecting the invisible structural damages or defects. According to the varied scattering response of the defects and main structure to the waveguide, the contrast dielectric images can be established. The sensitivity to the stress of the composite also affords it full credibility in stress-monitoring. With the wires as sensing elements inside the composite, the stress distribution can be imaged vividly. For instance, the composites are treated as being made of a number of blocks, and each block has a sensitive yet different response to the stress exerted on it, which is reflected on the resonance spectrum. This kind of technology is called microwave imaging technology (Fig. 64). Note that the principal
Advantage of this technology is that it not only has the ability to monitor or detect the condition of the materials at service, but can also be used at long range, which means remote monitoring can be achieved with data communication. This can greatly expand the capability of the current microwave NDT technique. A lucrative target to practice this technology is wind turbines, a critical element for wind energy harvesting. By implementing the thin coating layer containing microwires onto the surface of the existing turbines or embedding the wires into the turbine structure to be produced, the microwires would be able to function as dipolar antennas to monitor the health condition of turbines. They are also capable of identifying the approximate location of damages with the wire failure drawing on the length effect on the resonance frequency. In addition, the rotating blades is notorious in causing significant unwanted doppler returns to the illuminating radar, which could be somewhat detrimental to the radar operations as it is a formidable task to filter these unwanted signals [349]. Introduction of microwires can resolve this conflict of interest between the desire to encourage wide use of renewable and green energy and that to maintain the effective operation of those important human safety associated radars used for air traffic control, weather monitoring and marine navigation aids. The last but not least benefit is the attenuation of lightning impacts, which will severely influence the electronic systems in a wind turbine [349]. The threefold benefits will surely avail a niche for microwires being applied in energy sector.

It should also be noted that the research in SHM of composite structure is not without limitations. To date all work only deals with the dielectric matrices such as non-conductive polymer matrices or glass-fibre reinforced composites but not the carbon-fibre composites, in that the conductive carbon fibre is likely to result in huge conduction loss at a high volume fraction (60–70% for commercial carbon fibre composites), making it a formidable task to quantify the effect of microwires, if any, through the results, for example, from the free-space measurements. This is however not unsolvable, and the microwires could be set on the top of carbon fibre prepregs during the preform preparation or simply serve as functional coat or tape attached on the surface of carbon fibre composites. In this way, however, one may only be able to detect a surface crack but not the inner damage. On the other hand, the microwires coating on the carbon fibre could form a double-layer absorber capitalising on the absorbing properties of microwires and the quasi-reflective manner of carbon fibre composites [350]. How to implement the microwires into carbon fibre composites is an intriguing topic worthy of further study.

7.3. Other applications

Combining the remote stress control with the advantage of the small size, the wire inclusions can be made into micro antennas embedded into implants for biomechanical applications [218].

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**Fig. 64.** Proposed microwave contrast imaging technique for stress monitoring and structural interrogation [218]. Courtesy of Dr. Makhnovskiy of Plymouth University.
example, microwires have been proposed to be deployed in a prosthetic device as shown in Fig. 66. They are expected to meet the needs of immediate diagnosis of interfacial conditions at the micro level and to analyse the elastic deformation inside the materials. Yet such application remains at a conceptual level. Development of the relevant device has to rely on a full knowledge of the wire properties under different conditions, precise control of wires in the device design, and robust processing of signals. Compounded by the strict requirement of medical devices, this is non-trivial task but, without a doubt, is a promising direction for exploitation of microwire-based materials and devices.

Other possible applications rely on the combination of microwires and other functional materials, for example, carbon nanotubes. As is known, carbon nanotubes can be used as gas sensors owing to their affinity to gas molecules resulting from extremely high surface-to-volume ratio. They are capable of detecting explosive gases such as hydrogen or toxic gases. The mechanism behind is the dc resistance of CNTs changes sensitively with absorption of analytic molecules. However the low sensitivity (typically 2–10%) calls for a significant improvement by modifying this technique. Phan et al. [199] reported the CNT casted on magnetic ribbon could enhance the impedance of original ribbon and varies sensitively with the content. Furthermore, with enhanced GMI effect, the advanced microwires/CNT hybrid filler can then be incorporated into polymer matrix to develop a multiscale hybrid composite for microwave tunable and absorption applications. Therefore, development of microwire/CNT filler could be very useful to extend the sensing capability of microwires.

Another example is fibre-Optic magnetometer made from a magnetostrictive element attached to a length of optical fibre, capable of detecting external field via a phase change produced [351–353]. This
technology makes good use of the magnetomechanical effect (DE effect) addressing the magnetic field dependence of Young’s modulus of magnetostrictive microwires. This effect can also be used for biosensing application [354]. It is interesting to note that on the one hand optical fibre and magnetic microwire tend to be competitive in the sensing market; on the other hand, they can be coupled to realise sophisticated functionalities.

8. Summary and directions for future research

8.1. Concluding remarks

Both fundamental and technological aspects of microwire composites have been thoroughly reviewed in this article. The primary conclusions arrived at as follows:

1. The multifunctionalities of microwire composites stem from the wire inclusions, which can act as both reinforcement and functional fillers. According to the effective medium theory, the effective electromagnetic properties of wire-composites are determined by the topology of the wire patterns (mesostructure) and the local properties, i.e., the intrinsic properties of microwires, including geometry, dielectric and magnetic properties. By tailoring the microwires and regulating the composite mesostructure, the electromagnetic properties which characterise the microwave interactions with the composite can then be modulated to realise a variety of functionalities such as tunable properties, metamaterial properties and microwave absorption.

2. The microwire composites can be fabricated through different means from hand lay-up to open-mould casting, depending on the choice of materials. The selection of matrix draws on the structural requirement, ranging from elastomer to epoxy to glass-fibre prepreg with distinguished Young’s modulus. Mass production is limited by the wire patterns and geometry; it remains a technical challenge to realise industrial scale fabrication with precise control of composite structure following the optimised design.

3. Microwire-composites present significant GMI and GSI effect enabled by the wire inclusions. The GMI effect can be regulated via extremely small loading of wire fillers, which is desirable for the sensing applications. An enhanced GMI performance was obtained in the multi-wire composites through the collective response of the microwires in the presence of an external magnetic field. The wire-composite also shows superior stress-sensing resolution and the GSI effect of the wire-composite can be well optimised by the number of composite layers and annealing. As evidenced by the structural examination and tensile tests, the extremely small volume fraction of microwires (≈0.01 vol.%) allows the wire-composites to retain their mechanical integrity and performance.

4. The field-tunable properties of the composites in the microwave frequency range were found to vary remarkably with changing wire periodicity, wire geometry and composition. It is possible to optimise the microwave dielectric properties and transmission/reflection patterns of the composites through modulating these wire parameters and the mesostructure constituted by the wire topology. This can be utilised to manipulate the composite architecture and extend the limitation of the effective frequency range.

5. For a microwire composite panel, when the wire breakage occurs due to the applied stress, there appears a significant change in the sign and magnitude of the effective complex permittivity below plasma frequency. The magnetoelastic characteristics defined by the composition are critical to the stress sensitivity of dielectric response. When comparing the stress tunability and magnetic field tunability, much more complex dependencies of tunability on external stimuli were observed at the measured frequency range. The high frequency-domain spectroscopy characterisation further reveals that, with increasing wire concentration, the composite exhibits larger permittivity and strain sensitivity. There is a pronounced dependence of permittivity spectra featured as relaxation to resonance transformation with increasing strain and further confirmed with evolution of loss tangent. The relative variation of $\varepsilon'$ versus strain follows the Gaussian molecular network model very well.
6. Ferromagnetic microwires offer a solution to realise isotropic double negative medium \( \varepsilon < 0, \mu < 0 \) with relatively simple structure consisting of only wire arrays, as opposed to the conventional complex structure constituted by the conducting wires and magnetic materials or SRRs. By regulating the wire diameter, the wire spacing and magnetic bias, the negative index frequency range can be well tuned, which has been demonstrated in a number of configurations.

7. The microwave absorption of microwire composites originates from the dielectric loss and magnetic loss, the domination of which seems to depend on the filler content. For dilute composites, the glass-coat quality plays a vital role in reducing the reflection loss by impeding the formation of a fully conductive network. For heavy-loaded wire composites, the absorption feature is closely correlated with the FMR. Therefore, the local static magnetic properties and the effective static magnetic properties as well as the size effect determine the dynamic magnetic response of the wire composites and hence the absorption characteristics. As such, sufficient freedom is available for one to modulate the EM parameters and control the absorption features (absorption maximum and bandwidth) for the targeted applications. As concerns the EMI shielding, the absorption is identified as the main contributor. Rather low filler loading yields high shielding efficiency for the microwire composites and make it superior to other shielding materials.

8.2. Outlook for future research

The present review detailed the multifunctionalities of microwire composites. Inspired by current active study on this subject, some relevant directions are proposed as follows:

- The interfacial properties of the microwire composites require more detailed study, which is absent as yet. Chemical coupling agent such as silane can be applied to improve the interfacial strength between the glass coat of microwires and polymer matrix. Varying pull-out test methods (see e.g., [355]) and Raman spectroscopy can be used to perform a precise measurement of the bonding strength and interfacial stress transfer [356].
- Field/stress tunable properties of composites containing short or long continuous wires with either helical or circumferential anisotropy should be investigated with or without an external stress by varying the geometry of the wire and permittivity of matrix within a reasonable range using the flexible planar coil, which may indicate a variety of options for designing the microwave tunable structure under the external magnetic field [12].
- Considering the sensitivity of the MI of microwires to temperature [160], the temperature tunable properties would be of great use in composite process monitoring. The possibility of microwires serving as temperature sensing elements in composites manufacture realises the added values of microwires in the preform process. This would make such microwire composites more useful and promising for industrial applications. Analytical results of typical dispersion of effective permittivity and transmission/reflection spectra with temperature have been shown in Ref. [41]. It is promising therefore to direct efforts into this direction. Further, in view of the efficiency of microwave in activating cross-linking for polymers [357,358], the microwave–material interactions can also be exploited for the microwave curing of the polymer composites [359]. Although a fluoroptic thermometer is immune to microwave [360], it would be more convenient for microwires to serve as both heating and sensing elements in processing.
- To maximise the functionality and add more values to the resultant composite, a plausible approach is to develop hybrid composites incorporating both microwires and other functional fillers such as carbon nanotubes. Because they serve quite a few similar purposes, e.g., mechanical reinforcements [2], metamaterial units [361], and EMI shielding elements [362], they are likely to enhance the relevant performance of a hybrid composite. Further, their electromagnetic functions apply to different frequency ranges [363], and the combination of them may greatly extend the applicability of the resultant hybrid composite. In addition, magnetic nanoparticles can also be considered to coat on the surface of microwires to realise biphase magnetic filler with improved soft magnetic characteristics, which is desirable for some microwave applications hinged on high permeability and low hysteresis.
Since the impedance match of the front absorber can be improved by the non-zero surface impedance of carbon fibre composites as the substrate [364], design of a multilayer structure absorber based on microwires and carbon fibre composites could be very promising with the increasingly heavy use of carbon fibre in all kinds of vehicles. The manipulation of arrangement of carbon fibres and glass-coated microwires in normal manner could also realise metamaterial features [173], which can be exploited for cloaking application.

The objective of demonstrating the stress tunable properties is accomplished. However, such a technique is not instrumented yet. A lot of work needs to be done in order to realise a visual device with high-definition tomography. Hopefully such an instrumentation will renovate the composite industry in the pertinent aspects.

Overall, the ultimate goal is to devise a smart composite that is highly susceptible to a range of energy signals and capable of an efficient and effective transformation to electric signals. The functional fillers are expected to maximise the all-round performance of the resultant composite indiscriminately.

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