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Editorial

This collection contains the proceedings of the 21st European Conference on Composite Materials (ECCM21), held in Nantes, France, July 2-5, 2024. ECCM21 is the 21st in a series of conferences organized every two years by the members of the European Society of Composite Materials (ESCM). As some of the papers in this collection show, this conference reaches far beyond the borders of Europe.

The ECCM21 conference was organized by the Nantes Université and the Ecole Centrale de Nantes, with the support of the Research Institute in Civil and Mechanical Engineering (GeM).

Nantes, the birthplace of the novelist Jules Verne, is at the heart of this edition, as are the imagination and vision that accompany the development of composite materials. They are embodied in the work of numerous participants from the academic world, but also of the many industrialists who are making a major contribution to the development of composite materials. Industry is well represented, reflecting the strong presence of composites in many application areas.

With a total of 1,064 oral and poster presentations and over 1,300 participants, the 4-day event enabled fruitful exchanges on all aspects of composites. The topics that traditionally attracted the most contributions were fracture and damage, multiscale modeling, durability, aging, process modeling and simulation and additive manufacturing.

However, the issues of energy and environmental transition, and more generally the sustainability of composite solutions, logically appear in this issue as important contextual elements guiding the work being carried out. This includes bio-sourced composites, material recycling and reuse of parts, the environmental impact of solutions, etc.

We appreciated the high level of research presented at the conference and the quality of the submissions, some of which are included in this collection. We hope that all those interested in the progress of European composites research in 2024 will find in this publication sources of inspiration and answers to their questions.

Each volume gathers contributions on specific topics:

- **Vol 1. Industrial applications**
- **Vol 2. Material science**
- **Vol 3. Material and Structural Behavior – Simulation & Testing**
- **Vol 4. Experimental techniques**
- **Vol 5. Manufacturing**
- **Vol 6. Multifunctional and smart composites**
- **Vol 7. Life cycle performance**
- **Vol 8. Special Sessions**

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HIGHLY ELECTRICALLY CONDUCTIVE NANOCOMPOSITES FOR STRAIN SENSING DEVICE IN STRUCTURAL HEALTH MONITORING

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Keywords: Conductive nanocomposite, strain sensor, metasurface, spray deposition

Abstract

Chipless, passive and wireless sensors are one of the most important expected developments in wireless sensor technology for Structural Health Monitoring (SHM) because of their compactness and the absence of a battery or chip for the sensor operation. Strain sensitive resonant antenna reflectors based on the strain sensing metasurfaces (i.e. frequency-selective surfaces) are employed for this application. The variation of the spectral response of the metasurface with external perturbation results in an intrinsically effective sensing mechanism. Highly electrically conductive nanocomposites are required for this application. In this work, nanocomposites at high graphite nanoplatelets content (70 wt%) are fabricated by a spray deposition process on a polyetherimide (PEI) support. Experimental tests have been conducted to investigate the electromagnetic response of the material in the X band (8-12 GHz), showing an electrical conductivity of about 2.0 x 10^4 S/m. The design of the metasurface geometry and numerical simulations have been introduced that account for the electromagnetic properties of the material in the non-naturally frequency selectivity response under the mechanical strain in the X band.

1. Introduction

Chipless passive wireless sensors (CLPWs) are among the most important developments in wireless sensor technology. Their compactness combined with the unnecessity of battery or chip for their operation, make these sensors interesting in various fields, medical diagnostics, security, predictive maintenance, and environmental safety [1]–[3]. The recent progress in the materials field goes towards this development. Specifically, metamaterials and metasurfaces are very well-suited for telemetric sensing applications. They are collections of far-subwavelength resonating structures, typically aligned in a regular crystal lattice designed to interact with the free-space propagating electromagnetic waves. These resonators are designed to interact with the free-space propagating electromagnetic waves, rather than being excited directly by a waveguide or transmission line. Due to external perturbations, the spectral resonances can change, resulting in an intrinsic effective sensing mechanism.

Because the metamaterial properties typically depend on the shape, size, orientation, and proximity of the conducting elements, techniques that alter the geometry of the conducting elements can provide an excellent means for tuning or switching the metamaterial response [4]. Owing to their versatility, metasurfaces are currently proposed for several applications. In the recent literature, sensors for strain detection are found [5]–[8]. They are mostly based on shifting of frequency selective surfaces and are characterized by varied sizes and sensitivity. However, reversibility over cycling deformation is not fully addressed in these preliminary works. For real applications strain sensing sensors based on metasurfaces would require elevated levels of stretchability other than high stretchability and should retain functionality even when being mechanically deformed.

The fabrication of metasurfaces can be complex especially when the periodic structure is not a simple pin or patch. Methods like drilling and milling are limited to simple structures [9]. Additive Manufacturing (AM) is a promising candidate to implement metasurfaces. The design flexibility combined with the possibility of employing a wide variety of materials makes this technique suitable for the rapid prototyping of strain sensors. 3D printed nanocomposite with metal nanofillers, such as silver nanowires (AgNWs) and nanoplatelets (AgNPs) are reported to exhibit the highest electrical conductivity of 10^5 S/m [10], [11], followed by carbon nanotubes (CNTs) (10^4 S/m) [12], [13] and graphene nanoplatelets (GNPs) (10^3-10^4 S/m) [14], [15]. However, this technique limits the electrical conductivity of nanocomposites due to the low content of conductive nanoparticles that can be added to the insulating polymer and produce samples with fraction-of-a-millimeter thicknesses. In this work, a spray deposition printing process is employed to produce thin GNPs/Epoxy nanocomposite coating with high electrical conductivity to be employed in the modelling and fabrication of strain-sensing metasurfaces.

2. Modeling sensor

The physical principle of the sensing mechanism is given by the shift of resonance frequencies due to strain. Design-level simulation provides metasurface geometries optimized for the specific mechanical strain detection, at the frequencies of interest, 10 GHz (X band). The proposed metasurface geometry can decouple surface strain components. Full-wave electromagnetic modelling is employed for optimization.

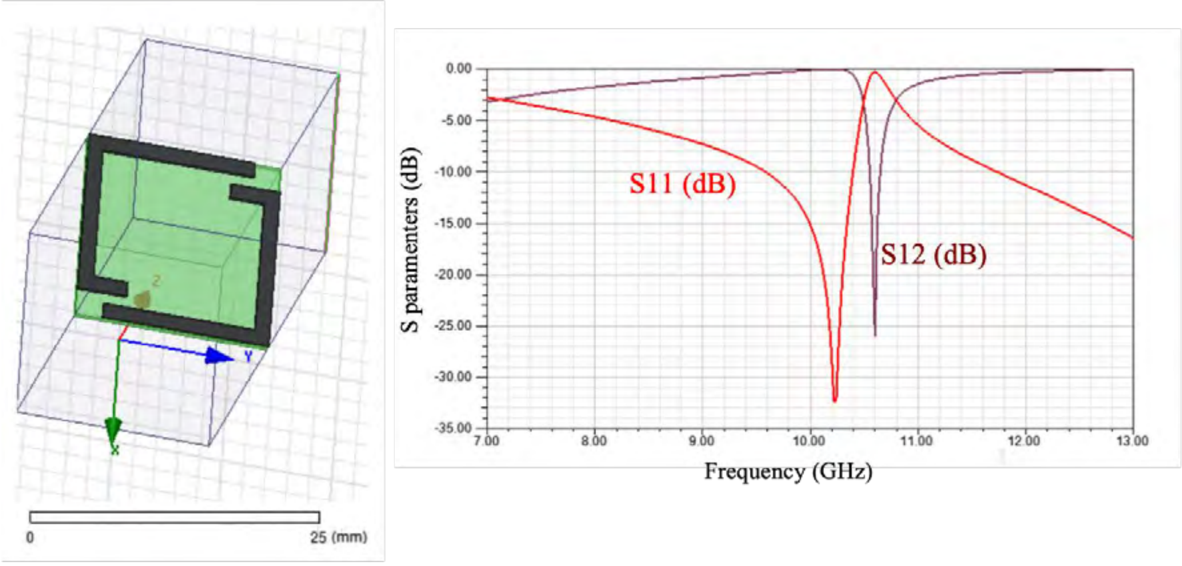


Figure 1. One quarter of metacell for the X band

The size of the unit cell, which is spatially repeated to form a metasurface, directly impacts the final size of the device. Figure 1shows an example of unit cell (only one quarter of metacell is shown for symmetry: metal and substrate are respectively 0.4 mm and 0.5 mm thick and have respectively conductivity 4*10^5 and permittivity 2.5) for the operation of the X band.

It is possible to verify, but it is not reported here for brevity, that a strain along x and y directions produces frequency shift of opposite signs. The above considerations assume metallic behavior of the nano-composite material used to define the resonant rings. It is noted that such assumption is well verified, at the considered frequency, for material conductivity exceeding 10^5 S/m. For a strain-detection able to simultaneously disentangle the x- and y-directed strain, the same principle physical mechanism as above can be used. A proper unit cell design allows adjusting their size to let them resonate at the desired frequency band and vice versa. Increasing the size of the unit cell design allows more than one resonance to come into play, and places more than one zero and/or pole of the transfer function (reflection or transmission) close to each other, to achieve a specific frequency selectivity.

3. Materials production

Graphite nanoplatelets (GNP)/epoxy nanocomposite films have been fabricated with a top-down approach based on a spray deposition process. The GNPs used in this work have a large aspect ratio of 2143 with a lateral size of 30 µm and thickness of 14 nm (supplied by NANESA S.r.l). A three-component resin made of Bisphenol A diglycidyl ether (DGEBA) epoxy resin (ARALDITE® LY 3508), tetrahydro-methyl phthalic anhydride (THMPA) curing agent (ARADUR® 917-1) and 2,4,6-tris (dimethyl aminomethyl) phenol as the catalyst (Accelerator 960-1) was employed in this study, provided by Huntsman corporation. An epoxy mixture with a stoichiometric ratio between epoxy and acyls equal to 0.6 was employed. The substrate used is a polyetherimide film of 100 µm thickness (PEI, Toray Cetex TC1000).

The GNPs/Epoxy nanocomposite coating was deposited on the PEI support using a spray deposition process. Firstly, a dilute dispersion of GNPs in a volatile solvent (acetone) is prepared using a mass volume ratio of 1.4%. The nanoplatelets dispersion is performed using a high-power tip sonicator. Once the dispersion is homogeneous, the epoxy resin is added and further sonicated (Figure 2a). The obtained solution is deposited on the PEI film utilizing an airbrush mounted (Figure 2b). The deposited material is dried all night at room temperature to let the solvent evaporate (Figure 2c). Subsequently, the material is compacted with a calender to promote particle orientation and compactness, obtaining a coating with a metal finish (Figure 2d). Finally, the sample is cured according to the resin polymerization process for 1h30min at 120°C and post-cured for 1h at 140°C.

The thickness (t) of the samples is measured using a digital micrometer by subtracting the PEI substrate thickness of 100 µm. An average value of 13±3 µm is calculated based on several measure points over the area of the sample is considered.

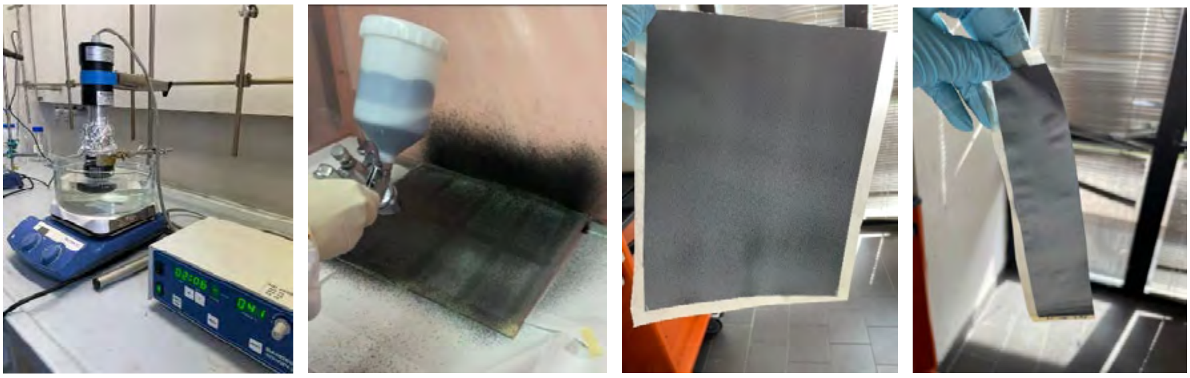

(a) (b) (c) (d)

Figure 2 Steps of the fabrication process of GNPs/Epoxy nanocomposite supported on PEI support. (a) Dispersion of nanoplatelets by ultrasonication. (b) Spray deposition process by using an airbrush. Deposition on PEI substrate (c) prior calendering and (d) post calendering

The sheet resistance (R_{sheet}, Ω/sq) of the coating has been evaluated with a 4-probe system (Napson, Resistest-8A). An average value was obtained considering 5 measure points. The static electrical resistivity (ρ, Ωm) and conductivity (σ, S/m) are computed considering the thickness (t) of the sample, as follows:

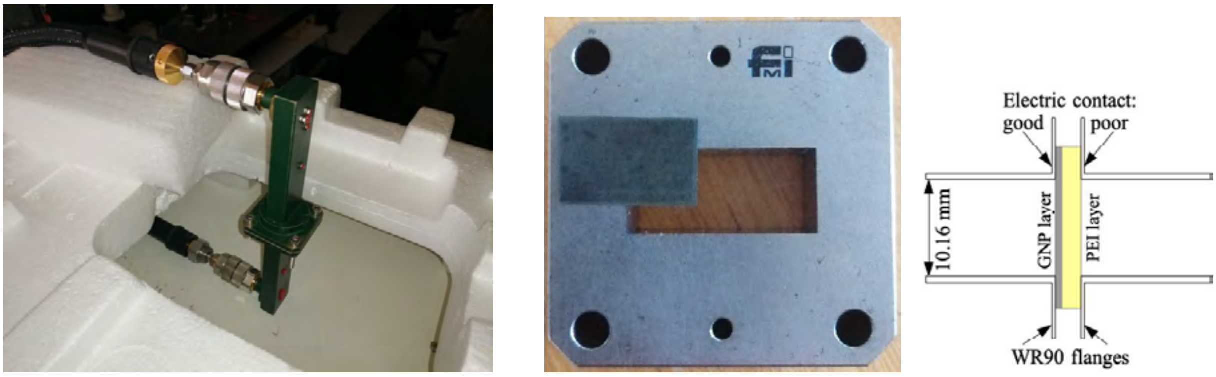
$$\begin{gathered}\rho = R_{sheet} \cdot t \\ \sigma = \frac{1}{\rho}\end{gathered} \tag{1}$$

The conductivity (σ, S/m) as estimated by sheet resistance measurements is 2.05 x 10^4 S/m.

4. Electromagnetic Testing

The electromagnetic characterization of the GNPs/Epoxy nanocomposite in the X band (8-12 GHz) has been performed by putting a thin rectangular sample of the material in the cross section of a WR90 rectangular waveguide (22.86x10.16 mm^2) as shown in Figure 3. A Vector Network Analyzer (VNA) has been used to measure the scattering parameters of the sample. TLR calibration technique has been applied to define the reference planes for the scattering parameters at the connecting flanges in the center of Figure 3.

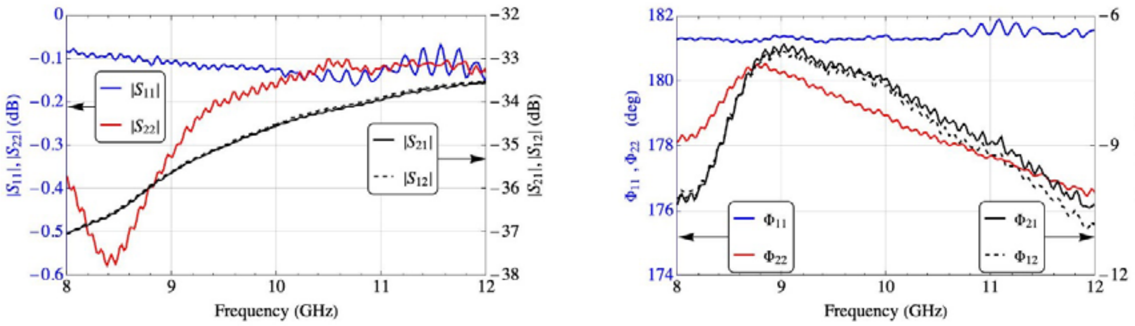
Hence, the scattering parameters S_{ij} shown in Figure 4 are exactly those measured to the left (port 1 of VNA) and to the right of the sample (port 2 of the VNA), in the empty waveguide.



(a) (b) (c)

Figure 3 (a) WR90 rectangular waveguides with the rectangular sample filling the cross section between the connecting flanges in the center of the image. (b) detail of the inner of the WR90 waveguide and a reduced piece of GNPs/Epoxy nanocomposite filling the cross section. (c) detail of the contact between the sample and the waveguide flanges (not in scale).

Amplitudes $|S_{11}|$ and $|S_{22}|$ and phases Φ_{11} Φ_{22} of the reflection coefficients at the two ports are slightly different because the sample is made by two different lossy material layers (the GNPs/Epoxy nanocomposite, d^{GNP}=13 µm thick, and the PEI support, d^{PEI}=100 µm thick) and the effect of the excitation from the two ports of the sample produce different reflections seen at the two ports. The measured amplitudes $|S_{12}|$, $|S_{21}|$ and phases Φ_{12}, Φ_{21} ensure a particularly good reciprocity of the composite medium both amplitude and phase. As a preliminary analysis, the amplitudes of the reflection coefficients are very close to 0 dB (less than –0.2 dB for $|S_{11}|$ and –0.6 dB for $|S_{22}|$) and their phases are close to 180° and these values imply that the composite material has high reflection property, similarly to an ideal conductor that is characterized by total reflection (0 dB amplitude and 180° phase). The differences between S_{11} and S_{22} in the lower part of the band are related to the fact that the wave impinging from the PEI port can flow also in the upper part of the metallic flange of the WR90 waveguide. In fact, the sample placed between the flanges is slightly larger than the cross section to ensure a flat placement between them when they are brought together and screwed. Unfortunately, this kind of placement and overlapping with the flange in the PEI port does not produce perfect electric contact between the PEI part of the sample (dielectric) and port that can cause the difference between S_{11} and S_{22}. On the contrary, electric contact between port 1 and sample is better because the GNP part of the sample is conductive.



(a) (b)

Figure 4 (a) Amplitude and (b) phase of the scattering parameters of the rectangular sample of the GNPs/Epoxy nanocomposite supported on PEI support**.**

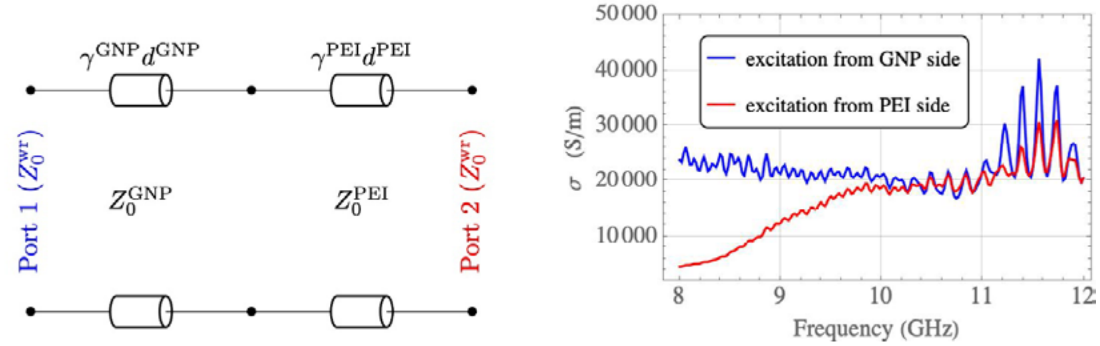
4.1. Electromagnetic properties estimation

The estimation of the conductivity of the GNPs/Epoxy nanocomposite can be obtained by the equivalent circuit shown in Figure 5(a). In fact, the two materials (GNPs/Epoxy nanocomposite and PEI) can be seen as two transmission lines characterized their modal impedance based on their conductivity σ and dielectric permittivity $\varepsilon=\varepsilon_0\varepsilon_r$ where ε_0 is the vacuum permittivity and ε_r the relative permittivity:

$$Z_0 = \frac{j\omega\mu}{\gamma} \tag{2}$$

$$\gamma = \sqrt{j\omega\mu_0(\sigma + j\omega\varepsilon_0\varepsilon_r) + \left(\frac{\pi}{a}\right)^2} \tag{3}$$

For the GNPs/Epoxy nanocomposite we can assume the hypothesis of good conductor which implies $\sigma \gg j\omega\varepsilon_0\varepsilon_r$. In Figure 4(a), $Z_0^{PEI}, Z_0^{GNP}, \gamma^{PEI}, \gamma^{GNP}$ refer to the modal impedance and propagation constant in the PEI or in the GNPs/Epoxy nanocomposite. Finally, Z_0^{wr} refers to the modal impedance of the empty WR90 waveguide, where the scattering parameters are measured and normalized. By the transmission line theory, we can calculate the theoretical value of the reflection coefficient seen at the two sides and by equating them to the experimental values we can extract an estimation of the conductivity of the GNPs/Epoxy nanocomposite, assuming ε_r=3.15 and $\frac{\sigma}{\omega\varepsilon_0\varepsilon_r} = 0.0013$ at 1 MHz for the PEI. The estimated values are shown in Figure 5(b) in the hypothesis of excitation from the GNP side (port 1, blue line) or the PEI side (port 2, red line).



(a) (b)

Figure 5 (a) Equivalent circuit of the GNPs/Epoxy nanocomposite supported on PEI. (b) Estimation of the conductivity of the GNPs/Epoxy nanocomposite.

The results in Figure 5(b), about the estimated conductivity of the GNPs/Epoxy nanocomposite, show a good agreement between the measurements made at the two ports of the sample. The differences in the lower part of the frequency band are related to the differences between S_{11} and S_{22}, as previously discussed.

4. Conclusions

This research serves as a significant starting step in the development of nanocomposite-based metasurfaces for structural health monitoring. The initial design of strain-sensitive resonant antenna reflectors has yielded important insights into the potential of this emerging technology to detect strain within the X-band frequency range (8-12 GHz) using chipless, passive, and wireless sensors. The modeling highlighted the importance of high conductivity for the successful operation of such metasurfaces as strain sensors.

A high electrical conductivity (approximately 20,000 S/m) was achieved using graphite nanoplatelets supported by a polyetherimide (PEI) dielectric film. The application of a spray deposition process to manufacture these nanocomposite layers proved to be an effective method for applying thin, uniformly conductive layers onto non-conductive substrates. This manufacturing technique is not only innovative but also scalable, making it suitable for widespread commercial use.

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