Characterization of multilayer nano-absorbers

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Abstract. Full characterization of thin nano-carbon and nano-ferrite absorbers was performed in the frequency range 5-40 GHz by a combination of two microwave methods – a resonance one using a pair of cylindrical resonators with different excited modes, and a broadband one, where the samples cover a 50-ohm microstrip line. The results show a reliable extraction of the complex dielectric and magnetic parameters of 20-60-μm thick absorber layers by the resonance method considered (including the anisotropy of these parameters) and a successful verification of their values in the wide frequency range.

1. Introduction

The nano-absorbers are very promising materials for applications in the modern microwave 5G communication systems and antennas, where a reliable suppression of parasitically excited spurious fields must be achieved. The microwave engineers need to know these materials’ dielectric and magnetic properties in order to perform an accurate design of the devices where these absorbers are to be incorporated.

Characterizing such lossy and thin materials with complex dielectric and magnetic properties is not an easy task. The thin absorbers are very important for antiradar and antenna applications [1, 2], making the knowledge of their parameters an important [3]. One of the most powerful methods suitable for absorbers is based on the free-space technique – a plane sample placed between two antennas [4, 5]. However, the method needs relatively big specimens and its accuracy for multilayer samples is not satisfactory. Another broadband technique relies on measuring the S parameters of waveguides with samples (coaxial [6], rectangular [7, 8] or stripline). The waveguide method allows separate measurements of the samples’ permittivity and permeability; however, it only yields the parallel (to the sample surface) components of these parameters. Among the wideband methods one should mention the one based on coplanar waveguides – the thin film is placed just below the coplanar conductor [9]. The common disadvantages of the wideband methods are the needs of relatively big and well-prepared thin samples and their moderate accuracy, which depends on the applied extraction approach and noisy measurements. From this point of view, the resonance measurements can ensure a considerably better accuracy [3] of separate determination of the dielectric and magnetic parameters by different resonance modes with maximums of the electric (E) or magnetic (H) field within the sample. However, the resonance curves in the case of very lossy absorbers could be fully destroyed.

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making the resonance measurements impossible. There exist several resonance solutions for such absorbers. One of them is to use high-quality (high-Q) resonators – dielectric, split-dielectric or whispering-mode resonators [3]. Another approach is the perturbation techniques with very small samples (examples have been given in [2]). The latter approach uses samples of small dimensions (disks, prisms, etc.) and thin films.

In this paper, we propose the use of a combination of an accurate resonance and broadband transmission-line methods to determine separately the dielectric and magnetic constants and loss factors of multilayer absorbers in the frequency range 0-40 GHz. The resonance measurements are based on the two-resonator method, which allows determination of the parallel and perpendicular dielectric parameters (i.e., the so-called dielectric anisotropy [10]), the equivalent magnetic constant, and the equivalent conductivity [11]. We have already applied this combination of methods to study anodic aluminium nano-porous metasamples with pure dielectric or combined dielectric and conductive properties [11], high-conductivity metamaterials with metal surface inclusions, and carbon-containing materials [12]. The objects of the research presented below are absorbers with nano-carbon and nano-ferrite inclusions with applications in a wide frequency range.

2. Methods and samples

2.1. Samples

A set of thin absorbers with nano-carbon and nano-ferrite inclusions were prepared. The absorber under test (AUT) has a thickness $t_{AUT}$ in the interval 20-60 $\mu$m. Due to fact that these AUT cannot form an independent sample for reliable microwave measurements, all the AUT layers produced were deposited on a 100-$\mu$m thick plastic polyester support (PE) and covered by ~30-$\mu$m thick protective lacquer L for bigger mechanical stability – see the models and the photograph in figure 1.

The three-layer samples include one AUT layer of several types (see the legend in the figure caption of figure 1). One AUT type is a single layer, which consists of nano-carbon particles of diameter ~3-4 nm, marked as nC. Another AUT layer (nF) consists of nano-ferrite Fe$_3$O$_4$ particles of diameters 20-30 nm. For comparison, a classical AUT (F) with an ordinary ferrite BaFe$_{12}$O$_{19}$ layer with a larger grain diameter ~3-4 $\mu$m is added to the investigated set of absorbers in order to perform a useful comparison for nF and F. Finally, several mixed AUTs were prepared by the three types of inclusions considered: nFF: PE + L + nF + F (mixing ratio F : nF = 1 : 1); nFC: PE + L + nF + nC (nF : nC = 2 : 1) and nFFC: PE + L + nF + F + nC (nF : F : nC = 1 : 1 : 1).

Further, disk-shape samples of diameter $D_{AUT} = 30$ mm were cut by a special cutter from the produced three-layer flat specimens. The samples have a total thickness $t_{AUT} = 150-190$ $\mu$m, which includes the thickness of PE $t_{PE} = 100$ $\mu$m, covering lacquer L thickness $t_L = 30 \pm 5$ $\mu$m and the AUT layer in the middle (the extracted thickness values vary in the interval $t_{AUT} = 20-60 \pm 5$ $\mu$m). Three models of these samples were adopted: 1) a single-layer approach with a thickness $t_{WS}$ (a whole sample with averaged parameters in the whole volume; course model); 2) a two-layer approach (sample AUT + L with a thickness $t_L = t_L + t_{AUT}$) (the influence of the support PE layer is excluded) and 3) a three-layer approach, where the AUT is in the middle (the influence of the covering protective layer L is separated from AUT). Our goal was to determine the layers’ parameters for all approaches considered.

![Diagram](Image)

**Figure 1.** Samples: (a) three-layer; (b) two-layer; (c) single-layer (whole sample); (d) Photograph of samples. **Legend:** AUT: absorber under test; PE: polyester support; L: PE + covering lacquer L; nC: PE+L+ nano-carbon layer; F: PE+L+ ferrite BaFe$_{12}$O$_{19}$ layer; nF: PE+L+ nano-ferrite Fe$_3$O$_4$ layer; nFF: PE+L+F+nF; nFC: PE+L+F+nC; nFFC: PE+L+nF+F+nC (F:nF =1:1; nF:nC =2:1; nF:F:nC =1:1:1).
2.2. Two-resonator method for extraction of dielectric and magnetic parameters of nanoabsorbers

Resonance measurements of AUT samples were performed by the two-resonator method described in [10] for multi-layer samples. Figure 2 illustrates a pair of measurement cylinder resonators used in the experiments; they are with equal diameters, which coincide with the sample diameter $D_{AUT}$, but with different heights. One of the resonators (R1) supports symmetrical $TE_{0mn}$ modes with the electric field perpendicular to its axis. Contrariwise, the other resonator (R2 with a three times smaller height) is designed to support symmetrical $TM_{0mn}$ modes with the electric field oriented along the resonator axis. In both cases, the AUT lies perpendicularly to the resonator axes: on a foam support with half-resonator height in R1 or directly on the resonator bottom in R2. Therefore, the AUT influences the resonance frequency and unloaded quality (Q) factor either in a parallel or in a perpendicular direction to the existing $E$ fields of the modes considered in R1 or R2, respectively. Thus, measuring the AUT in the R1 resonator yields the parallel dielectric parameters ($\varepsilon_r$ and tan $\delta_r$), while the measurements in R2 provide the perpendicular dielectric parameters ($\varepsilon_i$ and tan $\delta_i$); therefore, the actual anisotropy of the AUT can be evaluated with a sufficiently high accuracy (1-3 % measurement errors for $\varepsilon_{r//}$ and 2-5 % for tan $\delta_{r//}$ for the whole-sample approach; the errors for the two-layer approach increase up to 5-8 %, and to no less than 10-15 % for the single AUT approach for $\varepsilon_{i//}$).

The extraction of the sample parameters is based on the resonance characteristics measured (resonance frequency and unloaded Q factor) in both resonators R1/R2 with AUT that support symmetrical $TE_{0mn}$ or $TM_{0mn}$ modes (e.g. first three with index $m = 1, 2, 3$). The TE modes with index $n = 1, 3, 5, ...$ (with an $E$ field maximum in the AUT plane) ensure determination of the parallel dielectric parameters $\varepsilon_r$, tan $\delta_r$, while the modes with $n = 2, 4,...$ (with an $E$ field minimum in AUT), determination of the perpendicular dielectric parameters $\varepsilon_i$, tan $\delta_i$. The $TM_{0mn}$ modes with index $m = 1, 2, 3$ allow determination of the parallel dielectric parameters $\varepsilon_r$, tan $\delta_r$, while the $TM_{0mn}$ modes with index $m = 1, 2, 3$ and $n = 2$, determination of the parallel magnetic parameters $\mu_r$, tan $\delta_r$ (at this stage, the measurement procedure proposed cannot determine the magnetic loss factors tan $\delta_r$ with a satisfactory accuracy; thus, the results are not presented).

![Figure 2](image_url)

**Figure 2.** Study of nano-absorbers by resonance (a, b) and broadband microstrip-line (c) methods.

The resonance method uses the measured resonance frequencies and quality factors of the exited symmetrical $TE_{0mn}$ (in resonator R1; $m = 1, 2, 3, n = 1, 2$) and $TM_{0mn}$ modes (in resonator R2; $m = 1, 2, 3$); the broadband method is based on covering a 50-ohm microstrip line (MSL) with the sample and exciting a dominant quasi-TEM mode.

2.3. Broadband method for parameters verifications by covering of 50-Ohms microstrip line

The method relies on a measurement structure where the AUT covers a 50-ohm microstrip line (MSL) printed on a standard commercial substrate (in our case Arlon® 25N, 0.508-mm thick). In [2], we already reported the use of a similar structure to characterize the absorbing properties of microwave absorbers. The dominant mode in the MSL has the $E$ field present high above the conductor layout (shown illustratively in figure 2 c) thus penetrating into the AUT. Due to the sample influence on the additional insertion losses $S_{21}$ and insertion phase angle $S_{21}$ in the entire transmission line, their frequency dependencies are representative measures of the AUT parameters, if a reliable extraction
procedure is performed (details are presented in [11]). In fact, the \( E \) fields in the MSL have a mixed distribution (neither purely parallel, nor purely perpendicular); therefore, the covered MSL allows one to extract a mixed (equivalent) value between the parallel and perpendicular dielectric and magnetic constants, \( \varepsilon_{eq} \) and \( \mu_{eq} \). In our case, we simply introduce the parameters obtained by resonance measurements in the built 3-D model of the measurement structure and compare the simulated dependencies with the measured ones for all samples considered. However, there exists a measurement problem: usually, an unavoidable air gap with an effective thickness \( g_a \) appears between the sample bottom surface and the top MSL conductors (evaluated by the help of an isotropic PE sample; \( g_a \approx 2 \pm 0.25 \, \mu \text{m} \); figure 2 c) due to different reasons: small curvatures, roughness, surface irregularities, etc.

3. Results and discussion

First, the absorbing samples considered were measured by a pair of cylindrical resonator R1 and R2 with equal diameters 30 mm and heights 30 and 12.13 mm, respectively. The resonance curves of selected TE modes in R1 and TM modes in R2 are presented in figure 3a,b. Due to the small sample thickness, the resonance curves of the TM modes are sufficiently compact, which allows a reliable mode identification. However, observing the resonance curves and identifying the TE modes for lossy samples are difficult tasks. These circumstances notwithstanding, we succeeded in obtaining satisfactory results following the procedure described in [10]. The extracted pairs of values \( \varepsilon_{//}/\tan\delta_{//} \), \( \varepsilon_{\perp}/\tan\delta_{\perp} \) and \( \mu_{//}/\mu_{\perp} \) are listed in table 1. Pure ferrite (F), nano-ferrite (nF) and mixed (nFF) AUTs

![Figure 3](image-url)
have relatively stable dielectric parameters in a wide frequency interval (7-32 GHz) and expressed anisotropy: \( \varepsilon_{\parallel} \sim 6.7; \varepsilon_{\perp} \sim 2.5-6 \). The nano-carbon AUT shows a visible decrease of the parallel dielectric constant as the frequency is raised (\( \varepsilon_{\parallel} \sim 4-5 \), the effect has also been reported in [11]); while the perpendicular dielectric constant remains relatively large, \( \varepsilon_{\perp} \sim 16-23 \). However, this is connected with a possible increase of the sample conductivity [11]. Adding nC inclusions to the ferrite AUT, both dielectric constants, \( \varepsilon_{\parallel} \) and \( \varepsilon_{\perp} \), of samples nF and nFFC increase in comparison to the pure ferrite AUT and a stronger frequency dependence arises: \( \varepsilon_{\parallel} \sim 7-2.5; \varepsilon_{\perp} \sim 4.5-22.5 \). Definitely, this is a rather specific behavior connected with the influence of the equivalent conductivity of the samples due to the carbon content. The ferrite samples’ magnetic constants are close to 1: \( \mu_{\parallel} \sim 0.45-0.9; \mu_{\perp} \sim 1.05-1.2 \). The values \( \mu_{\parallel} \sim 1.45-7 \) measured for nano-carbon AUT is a quasi-effect; it has to do with the influence of the sample conductivity on the resonance frequencies (explained in [11]).

The above results allow us to verify their accuracy by covering the MSL and measuring the additional losses and phases – the dependencies in figures 4 a, b. The simulated curves for the phase delay in the MSL after covering by an AUT coincide very well with the measured curves, if an air gap of \( g_{a} \sim 2 \mu m \) is taken into account (figure 4 b). The simulated dependencies were obtained for the different AUTs with parameters taken from Table 1 for the frequency interval 15-20 GHz (single-layer approach). This is a direct evidence that the dielectric and magnetic constants of AUTs measured by the resonance method are sufficiently accurate in a wide frequency range; however, the method of covered MSL yields mixed values of the equivalent dielectric and magnetic constants different from

| Table 1. Values of the dielectric and magnetic parameters of absorber samples extracted by resonance measurements for three types of approximations: single-, two-layer or whole sample. |
|---|---|---|---|---|---|---|
| Samples | \( f_{max, \mu m} \) | Whole sample approach (mixed parameters for PE + AUT + L) | Two-layer approach (mixed AUT + L) | Single-layer approach (parameters for single AUT layer) | |
| | TE | TM | TE | TM | TE | TM | |
| PE | 100 | 011 | 3.15/0.0061 | - | 3.32/0.0083 | - | 3.32/0.0085 | - |
| | | 021 | 3.16/0.0048 | 2.97/0.0060 | 2.38/0.0068 | 2.35/0.0085 | - |
| | | 031 | 3.15/0.0049 | 2.95/0.0056 | 2.35/0.0085 | 1.58/0.026 | - |
| L | 30 | 011 | 2.96/0.0070 | 2.54/0.0079 | 2.32/0.0083 | 1.54/0.020 | - |
| | | 021 | 2.97/0.0060 | 2.52/0.0073 | 2.38/0.0068 | 1.73/0.030 | - |
| | | 031 | 2.95/0.0056 | 2.51/0.0062 | 2.35/0.0085 | 1.58/0.026 | - |
| nC | 25 | 011 | 4.75/0.037 | 3.47/0.019 | 7.65/1.59 | 4.20/2.14 | 13.6/200 | 18.8/11.0 |
| | | 021 | 4.30/0.052 | 3.24/0.018 | 6.82/5.36 | 4.04/1.54 | 12.8/210 | 15.5/9.0 |
| | | 031 | 4.25/0.050 | 3.21/0.017 | 3.49/5.50 | 3.56/2.20 | 4.93/1500 | 22/5.5 |
| F | 25 | 011 | 3.72/0.0175 | 2.96/0.086 | 4.05/0.66 | 2.00/10.0 | 5.98/1.09 | 2.36/75 |
| | | 021 | 3.70/0.021 | 2.85/0.020 | 4.00/2.0 | 2.60/70.0 | 5.92/7.80 | 2.61/5.2 |
| | | 031 | 3.75/0.027 | 2.88/0.010 | 4.15/2.7 | 2.35/16.0 | 6.23/24.4 | 2.38/30.8 |
| nF | 20 | 011 | 3.35/0.028 | 2.68/0.036 | 3.75/0.58 | 2.05/1.93 | 5.88/160 | 3.4/168 |
| | | 021 | 3.34/0.045 | 2.78/0.028 | 3.73/4.1 | 2.54/5.5 | 5.95/210 | 4.07/85 |
| | | 031 | 3.32/0.034 | 2.72/0.024 | 3.71/1.6 | 2.24/26 | 5.87/400 | 3.72/11 |
| nFF | 20 | 011 | 3.48/0.020 | 2.77/0.060 | 4.12/0.33 | 2.21/8.47 | 6.79/147 | 5.2/1050 |
| | | 021 | 3.47/0.025 | 2.78/0.018 | 4.11/0.43 | 2.53/20.0 | 6.91/159 | 6.0/56 |
| | | 031 | 3.48/0.035 | 2.73/0.012 | 4.18/1.07 | 2.26/14.0 | 6.90/480 | 5.5/23 |
| nFFC | 25 | 011 | 5.30/0.079 | 3.00/0.074 | 9.21/0.81 | 2.75/90.0 | 17.45/3100 | 8.8/930 |
| | | 021 | 4.52/0.113 | 3.06/0.090 | 7.02/1.35 | 3.34/21.0 | 12.72/300 | 22.5/162 |
| | | 031 | 3.25/0.045 | 3.06/0.025 | 3.47/4.20 | 3.11/19 | 4.88/2100 | 16.5/24 |
| nFC | 60 | 011 | 5.94/0.120 | 2.85/0.053 | 8.84/3.34 | 2.57/73 | 12.1/670 | 4.43/11 |
| | | 021 | 4.41/0.110 | 2.88/0.089 | 5.82/2.5 | 2.67/80.4 | 7.52/82.6 | 4.70/13 |
| | | 031 | 2.84/0.055 | 2.82/0.036 | 2.51/0.77 | 2.62/144 | 2.59/730 | 4.60/28 |

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the parallel and perpendicular ones; the method cannot separate the influence of the dielectric and magnetic properties.

The additional losses simulated and measured for pure ferrite AUT are also in relatively good agreement (figure 4 a). Problems appear for the samples with nC inclusions, namely, the measured \( \tan \delta_{//,\perp} \) values cannot fit well the measured losses. In this case, we include additionally in the simulations an equivalent conductivity \( \sigma_{eq} \) of nano-carbon samples (e.g. \( \sigma_{eq} \approx 0.3-0.5 \text{ S/m} \) for the frequency interval 15-25 GHz). The influence of this parameter fully explains the bigger losses measured for samples nC, nFFC and nFC (figure 4 a); the equivalent conductivity of these samples increases with the frequency.

![Figure 4](image)

**Figure 4.** Additional losses (a) and phase delay (b) of MSL covered by AUT with parameters taken from table 1. Legend: solid curves – measurements; dashed curves – simulations by ANSYS HFSS®.

4. Conclusions

In conclusion, the methodology proposed provides fully applicable results for nano-AUTs that are as thin as 20-60 \( \mu \text{m} \), which allows one to simulate reliably microwave devices with incorporated nano absorbers. The combination applied of two methods, resonance and wideband, seems to be useful for materials with mixed dielectric and magnetic properties and measurable conductivity. The resonance method, based on selected modes with different orientation of the electric fields with respect to the sample surface allows a complete and separate characterization of the dielectric and magnetic parameters and their possible anisotropy. This is achievable at fixed frequencies, but when one applies a broadband method where the AUT samples cover a 50-ohm microstrip line, the accuracy of these parameters can be verified in a wide frequency range (5-40 GHz in this research).

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