Experimental Characterization of Positive and Negative Dielectric Constants and Artificial Anisotropy of Metamaterials in the Microwave Range

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Abstract. This paper could be considered as a part of the invited talk “Metamaterials and Their Characterization in the Microwave Range” presented on the 8th International Workshop and Summer School on Plasma Physics (IWSSPP’2018, Kiten, Bulgaria), which has been dedicated to the classification, development, characterization and main applications of the metamaterials as one of the key issues of the modern technologies in the microwave, terahertz and optical regions. The experimental characterization of the actual equivalent dielectric parameters of the engineered artificial metamaterials is an important task in their design, nevertheless the existing relatively good correlation between the numerical simulations of these materials by 3D simulators and their measurements. The aim of the presented investigations is to give a helpful comparison between the most popular characterization methods based on free-space, waveguide and resonance measurements in the microwave range. The description of these methods has been based on the following principle: different metamaterials with surface metal inclusions have been selected and their main characteristics – equivalent dielectric constants in different directions (dielectric anisotropy) and the equivalent conductivity have been compared. A special attention has been directed to the description of measurements of positive and negative values of the permittivity, detection of artificial magnetic properties and resonance behaviour of the conductivity of the considered metamaterials.

1. Introduction
Each metamaterial could be considered as an assembly of multiple individual elements (unit cells; “meta atoms”). These unit cells are made from conventional materials (dielectric, magneto-dielectric or metal inclusions); however they are usually arranged in specific periodic patterns and the whole metamaterial gains its properties not from the concrete inclusions’ composition, rather from their well-designed meta lattice (meta crystals). Thus, the precise chosen shape, geometry, size, orientation and arrangement of the unit cells influence the electromagnetic waves to create specific properties that are unachievable by the conventional materials. Metamaterials attain the desired effects by incorporating structural elements with well-chosen sizes (from several nanometres up to several centimetres) in comparison to the wavelength. With other words, the electromagnetic waves show different behaviour in different metamedia depending on whether they have wavelengths comparable to or much greater than the characteristic size of the building blocks (unit cells or inclusions). On this base a useful metamaterial classification has been accepted in two main groups. One group is associated with the so-called artificial "band-gap structures” (wavelength is comparable with the “meta atoms” periodicity
length), e.g., photonic, plasmonic, phononic, and magnonic crystals [1]. The other group is associated with the so-called “effectively continuous metamaterials” (the wavelength is much greater than the characteristic size of the building blocks; typically from $\lambda/10$ to $\lambda/1000$); these materials show effectively continuous properties, not achievable from the ordinary materials (they go beyond natural materials – metamaterials). The local effects in similar metamaterials dominate, while the non-local effects could be described in many cases introducing chirality (bi-anisotropy) coefficients and effective (equivalent) permittivity/permeability parameters. Similar metamedium has to be considered as an effective continuum; the modelling of such complex media could be done by a homogenization (averaging) of the microscopic Maxwell equations to derive their unusual material properties [1].

We will concentrate our investigations on the second type of metamaterials. There exist three main ways to construct different artificial engineered effectively-continuous metamaterials by surface metal inclusions on substrates. Artificial dielectrics could be constructed by manipulation of the capacitance of electrical dipoles ordered in a lattice. Their equivalent (or effective) dielectric constant $\varepsilon_{eq}$ will increase along the wire length (see figure 1(a)), but it will be practically equal to the substrate dielectric constant perpendicularly to the wires (expressions have not been presented; the given dependencies are selected for an illustration only). This is an example for realization of an anisotropic dielectric; quasi-isotropic dielectrics could be realized by crosses – wires in the both parallel directions. This simple example shows a construction of artificial materials with metal inclusions, for which $\varepsilon_{eq} > \varepsilon_{sub}$, where $\varepsilon_{sub}$ is the dielectric constant of the hosting substrate/dielectric. If an additive or subtractive technology (e.g. 3-D printing) has been applied to mix two or more dielectrics in a homogeneous structure (all-dielectric metamaterials), then different situations with $\varepsilon_{eq} > \varepsilon_{sub}$ or $\varepsilon_{eq} < \varepsilon_{sub}$ could be realised (see the next section).

A compelling aspect of some metamaterials has been the ability to create artificial magnetism – materials with magnetic response without the use of inherently magnetic materials. Now the construction of artificial magnetics means to manipulate the magnetic flux density through the magnetic dipoles (loops) in a cell. In this example the most popular unit cell has been used – the so-called split ring resonator (SRR). The equivalent (or effective) magnetic constant $\mu_{eq}$ could be manipulated easier in the interval $0 < \mu_{eq} < 1$ and even $\mu_{eq} < 0$ (a “ferrite-like” behaviour), which is difficult by the natural magnets (see figure 1(b)). Finally, the design of the artificial resonant metamaterials includes the proper choice of the resonances for $\varepsilon_{eq}$ and $\mu_{eq}$ and allows selecting intervals, where the equivalent parameters are positive $\varepsilon_{eq}, \mu_{eq} > 0$ (right-hand materials, RHM), or both they are negative $\varepsilon_{eq}, \mu_{eq} < 0$ (left-hand materials, LHM) and to construct variety of metamaterials with unusable properties (see

![Image](http://people.ee.duke.edu/~drsmith/index.htm)

**Figure 1.** Illustration of possible frequency behaviours of the effective (equivalent) dielectric $\varepsilon_{eq}$ or magnetic $\mu_{eq}$ constants for three types of artificial materials with surface inclusions; examples for: (a) artificial dielectric with non-resonance behaviour of $\varepsilon_{eq}$; (b) artificial magnetic with resonance behaviour of $\mu_{eq}$; and (c) artificial dielectric with resonance behaviour of $\varepsilon_{eq}$ (source: http://people.ee.duke.edu/~drsmith/index.htm)
figure 1(c)). In many cases the “plasma-like” behaviour, $0 < \varepsilon_{eq} < 1$ and even $\varepsilon_{eq} < 0$, is welcome in many applications, e.g. in the antenna design. Metamaterial research is strongly interdisciplinary and involves a lot of researches in the whole world [2]. One of the important areas, directly related to this research, is the metamaterial characterization. In many applications it is necessary to know the equivalent material parameters of the metamaterials. The main parameters, which are informative for the properties of the metamaterials, are: equivalent (effective) dielectric constant $\varepsilon_{eq}$ and equivalent (effective) magnetic constant $\mu_{eq}$ (equivalent permittivity and permeability); equivalent refractive index $n_{eq}$ (especially in the optical and THz ranges) and equivalent conductivity $\sigma_{eq}$. All of these parameters can have as positive, as well as negative values, which determine the specificity of their experimental characterization. The needs for knowledge of the actual equivalent dielectric characteristics of the engineered artificial metamaterials is important in the modern design, nevertheless the proven relatively good correlation between the numerical simulations of these materials by 3D simulators [3, 21] and the performed experimental investigations.

The aim of the presented here investigations is to consider and compare the most popular methods for experimental characterization of engineered metamaterials in the microwave range, based on free-space, waveguide and resonance measurements in the microwave range. The presentation is based on the following principle: two different metamaterials with surface metal inclusions have been selected and their main characteristics – equivalent dielectric constants in different directions (dielectric anisotropy) and the equivalent conductivity have been determined by the supported in the laboratory measurement equipment and methods, including two authorship ones [4, 5]. Positive and negative values of the permittivity and resonance behaviour of the conductivity of the considered metasurfaces have been measured and compared. Due to the fact that the metamaterials have more or less expressed anisotropy of these parameters – different values in different directions, depending on the orientation and sizes of the unit cells, we consider in the next section of the paper some issues, concerning the origin of anisotropy of some artificial materials.

2. Origin of the resultant dielectric anisotropy in the artificial materials

The dielectric anisotropy of materials could be expressed as different dielectric constants in different directions, caused by different reasons. The microwave and optical engineers usually connect the anisotropic behaviour with the microwave ceramics and optical lenses; this is one of the oldest known types of material anisotropy. This is the so-called crystalline anisotropy for mono- or polycrystalline materials (optical glasses, ceramics, artificial soft and low-temperature co-fired ceramics LTCC, liquid crystals, etc.). In principle, these materials are homogeneous, but the anisotropy appears due to the existence of different crystallographic axes in the lattices. This anisotropy is usually relatively strong. In fact, another specific group of plasma/ferrite researchers identifies the original concept for the material anisotropy with the magnetic or electric gyrotropy of gaseous or solid-state plasmas and ferrites, all in external magnetic biasing fields. These phenomena belong to the so-called induced anisotropy; the dielectric and magnetic properties of similar natural metamaterials have been described with tensor form of the permittivity and permeability (including non-diagonal components, controllable by external dc biasing magnetic field). The ferroelectric materials and films can also be associated with this group of electrically gyrotropic materials, but in external electric biasing field. Nowadays, a new class of materials appears, for which the measured anisotropy could be considered more as an undesired property. First of all, in this group we can add the commercial engineered reinforced substrates with many applications in the modern RF and microwave electronics. The characterization of the dielectric anisotropy of similar popular materials is very important for the reliability of the modern design of different planar structures on similar substrates, especially in the millimetre-wave range. We measure reinforced substrates from different manufacturers and different types with anisotropy from 1-2 up to 20 % (see below for this parameter) [6]. The anisotropy of similar structures has been caused by the inhomogeneity between the mixed reinforcing fibres net and the applied filling. To this group of anisotropic materials we can add the textile fabrics [7] used for wearable antennas, multilayer antenna radomes [8], 3-D printed dielectrics [9], etc. The artificial
anisotropy of the engineered metamaterials and “band-gap” materials with controllable dielectric constant in different directions is now the new type of anisotropy [10], caused by the chosen shape, geometry, size, orientation and arrangement of the unit cells of the artificial materials. Therefore, it is very similar by origin with the anisotropy, caused from the inhomogeneity of the reinforced and composite samples. Let’s consider how this artificial anisotropy appears?

There exist many different technologies to mix two dielectrics or dielectrics with metal inclusions: series, parallel, mixed, layered (series/parallel), dispersed (uniform/random), pillar, impregnated, etc. The known models of similar mixtures [11] allow to estimate the resultant dielectric constant, but the information for the anisotropy is very coarse and not depends on the size and form of the concrete unit cell. In order to obtain more reliable information for the artificial anisotropy and the origin of this phenomenon, we started in the paper [12] a research for investigation of the anisotropy properties of a big group of artificial materials: textile fabrics [13], 3-D printed dielectrics [14], homogenized metamaterials [15] and even fresh plant biomass [16]. The common issue of these investigations is the possibility to estimate numerically and experimentally the artificial anisotropy of different materials. There exist three cases of dielectric anisotropy of the artificial materials, which dielectric properties can be described by diagonal tensors, namely:

\[
\begin{align*}
(\varepsilon_r)_{\text{Bi-axial}} &= \begin{pmatrix}
\varepsilon_{xx} & 0 & 0 \\
0 & \varepsilon_{yy} & 0 \\
0 & 0 & \varepsilon_{zz}
\end{pmatrix} ; &
(\varepsilon_r)_{\text{Uni-axial}} &= \begin{pmatrix}
\varepsilon_{\text{par}} & 0 & 0 \\
0 & \varepsilon_{\text{par}} & 0 \\
0 & 0 & \varepsilon_{\text{perp}}
\end{pmatrix} ; &
(\varepsilon_r)_{\text{Near-to-isotropic}} &= \begin{pmatrix}
\varepsilon & 0 & 0 \\
0 & \varepsilon & 0 \\
0 & 0 & \varepsilon
\end{pmatrix}
\end{align*}
\]

The most of the metamaterials with non-symmetrical unit cells have bi-axial anisotropy with three different scalar dielectric constants along the axes 0x, 0y and 0z. The uni-axial anisotropy has been expressed in relatively thin structures with plane symmetry, while a near-to-isotropic behaviour is a property of the homogenized and foam-like metamaterials.

In [12-14] we proposed a numerical method for reliable determination of the dielectric constants of artificially constructed materials (incl. metamaterials) in three directions by resonance method. The idea of the method is to build the corresponding unit cell, to reproduce it in a hosting substrate (see figure 2 (a, b) and to put the whole sample in a rectangular resonator, which supports TE and TM modes with three mutually perpendicular directions of the electric fields – figure 3. An independent extraction of the resultant dielectric constant along all three different directions is possible after replacing of the metasubstrate under interest with an equivalent substrate. We selected different standard inclusions (see illustrations in figure 2(c)) – spheres, cubes, cylinders, prisms, disks and some combinations between them, made by isotropic dielectrics or by metals. The results from simulations of several artificial samples with uni-axial anisotropy have been presented in Table 1. We use for a quantitative measure of the resulting dielectric constant/loss tangent anisotropy (parameters \(A_\varepsilon\) and

![Figure 2](Image)

**Figure 2.** (a) Universal unit cell; (b) constructed artificial sample with unit cells (inclusions) in a hosting Polycarbonate (PC) substrate; (c) examples for several unit cells [12]: spheres, cubes, cylinders, disks, rectangular net, disks + cylinders (made by dielectrics or metals)
Figure 3. Rectangular resonator with meta substrate with three modes with mutually perpendicular electric fields along the axes: (a) 0x; (b) 0y; (c) 0z with different resonance frequencies in a chosen frequency band (X band in this case)

Table 1. Results for the artificial anisotropy in PC substrate with dielectric/metal inclusions [12]

| Sample †† | $\varepsilon_{\text{incl}}/\varepsilon_{\text{PC}}$ | $\varepsilon_{\text{par}}/\tan\delta_{\varepsilon,\text{par}}$ | $\varepsilon_{\text{perp}}/\tan\delta_{\varepsilon,\text{perp}}$ | $A_\varepsilon/A_{\tan\delta,\varepsilon}$ % ‡‡ |
|-----------|-----------------|-----------------|-----------------|-----------------|
| 0) Pure PC | 1               | 2.75/0.005      | 2.75/0.005      | 0/0             |
| 1) PC + spheres ($d = 0.3$ mm) | 0.364           | 2.714/0.00496   | 2.719/0.00497   | -0.18/-0.20     |
| 2) PC + cubes ($a = 0.3$ mm) | 0.364           | 2.644/0.00489   | 2.644/0.00487   | 0.00/-0.41      |
| 3) PC + cylinders along 0z ($d = 0.3$ mm) | 0.364           | 2.621/0.00481   | 2.660/0.00492   | -1.48/-2.26     |
| 3') PC + cylinders along 0x ($d = 0.3$ mm) (no image) | 0.364           | 2.547/0.00476   | 2.502/0.00465   | 1.78/2.34       |
| 4) PC + disks in plane 0xy ($a = 2.0$ mm) | 0.364           | 2.269/0.00423   | 2.246/0.00396   | 2.25/6.47       |
| 5) PC + cylinder net (0.3 mm) | 0.364           | 2.249/0.00461   | 2.502/0.00465   | -0.20/-0.86     |
| 6) PC + metal cylinders ($d = 0.3$ mm) + disks ($a = 0.7$ mm) | 0.364           | 2.547/0.00476   | 2.502/0.00465   | 1.78/2.34       |
| 6') PC + surface disks (0.7 mm) | 0.364           | 2.497/0.00473   | 2.025/0.00185   | 75.80/87.54     |

†† sample denotation and images are given in figure 2(c)

‡‡ anisotropy: $A_\varepsilon = 2(\varepsilon_{\text{par}} - \varepsilon_{\text{perp}})/(\varepsilon_{\text{par}} + \varepsilon_{\text{perp}})$; $A_{\tan\delta,\varepsilon} = 2(\tan\delta_{\varepsilon,\text{par}} - \tan\delta_{\varepsilon,\text{perp}})/(\tan\delta_{\varepsilon,\text{par}} + \tan\delta_{\varepsilon,\text{perp}})$

Figure 4. (a) Resultant dielectric constant anisotropy $A_\varepsilon$ for isotropic PC with dielectric inclusions like cylinders in different directions for different ratios $\varepsilon_{\text{incl}}/\varepsilon_{\text{PC}}$; (b) Dielectric anisotropy $A_\varepsilon$ of 3-D printed unit cells with different shapes and symmetry (0 – from [12, 15]; 1 – [17]; 2, 3 – [9])
The set of full-antenna $A_{\text{tan}k}$, presented in the last column (see the corresponding expressions in the footnote to Table 1). Data clearly show that the dielectric spherical and cubic inclusions practically don’t change the isotropy of the hosting substrate. The set of full-height via holes (cylinders or disks) along the perpendicular $0z$ axis definitely increases the anisotropy, but it remains small (less than 4-5 % for $A_0$). However, a set of perpendicular metal via holes influences the anisotropy much stronger. Generally, the metal inclusions increase the anisotropy in all considered cases, nevertheless their forms (see the next sections); the influence is stronger in direction along the cylinder axes – ~30-75%. The last fact has been confirmed also for all-dielectric artificial materials – see dependencies for cylinder inclusions in figure 4 (a). The rule has been confirmed – the dielectric constant along the cylinders increases, when the electric field is orientated along this direction, in comparison to the dielectric constant, perpendicularly to the cylinder axis. This effect increases the anisotropy $A_z$ of the whole artificial substrate, which increases with the rise of ratio $\varepsilon_{\text{incl}}/\varepsilon_{PC}$. The positive sigh of $A_z$ means that $\varepsilon_{\text{par}} > \varepsilon_{\text{perp}}$, while the negative sign – that $\varepsilon_{\text{par}} < \varepsilon_{\text{perp}}$. We tested the described effect in the case of all-dielectric metamaterials, made by 3-D printing – dependencies in figure 4 (b). When the dielectric inclusions have been predominantly orientated along a fixed direction (e.g. samples with unit cell 0, 3, 4), the resultant dielectric constant increases along this direction and the anisotropy is relatively big, especially when $\varepsilon_{\text{incl}} > \varepsilon_{PC}$. Contrariwise, when the unit cell is enough symmetrical (e.g. inserted cubes – unit cell 1; spheres, rhombuses, etc.), the anisotropy is very small in all directions. We confirmed numerically and experimentally all of these effects in the papers [12-15].

3. Free-space method for characterization of big samples of metamaterials

Let’s now concentrate our efforts on the characterization of metamaterials made from reinforced substrates with metal inclusions on the surface. Depending of the size of the samples, we can apply several known method for experimental determination of their dielectric constant – see figure 5.

One of the most popular methods is the free-space method [18]; it has been already applied to metamaterials [19, 20]. In this method the metasample should be placed in the non-reactive zone between two antennas (often with focussing lens, forming smaller beam spots). The dielectric parameters can be extracted from the measured relative transmission losses (for tan$\delta_{\varepsilon_{\text{eq}}}$) and relative phase (for $\varepsilon_{\text{eq}}$). To avoid diffraction effects, the samples should be relatively big – see illustration in figure 5(a).

In order to test this method, we selected three different metamaterials, made by surface metal inclusion on standard substrate Arlon 25N ($\varepsilon_{\text{par}}/\varepsilon_{\text{perp}} = 3.57/3.35$ [6]). One of them is made by Cu surface disks (metadisks) and the insertion phase has negative sign (phase delay) – see dependencies in figure 6(a). The phase delay of metadisks sample is bigger than for the pure substrate; thus, the dielectric constant definitely increases – see the extracted values of $\varepsilon_{\text{eq}}$ in figure 6(b), when the corresponding metasample has been replaced with an equivalent sample with the same thickness (set of $\varepsilon_{\text{eq}}$-dependencies has been shown). The other sample (called meta) don’t have planar symmetry; due to this fact it shows different insertion phases for vertical and horizontal orientation of the E fields of the incident wave, changing the antennas’ polarization. For horizontal orientation the sample shows again expressed phase delay, but for vertical orientation – it has “phase advance” – the transmitted wave has bigger phase velocity than the velocity without sample. In this case negative values of the dielectric constant could be extracted; see the corresponding dependencies in figure 6(b). It is important to note, that the
Figure 6. (a) Measured relative phase delay/advance by free-space method in K/Ka bands through two types of meta samples with rectangular and disk metal inclusions (sample 1 metasurface: non-symmetrical planar unit cell 3x1.5 mm deposited on the both surfaces of substrate Arlon 25N (10 mils); sample 2 metadisks: symmetrical planar unit cell with disks of diameter 3 mm and distance between them 4 mm, single deposited on Arlon 25N (6 mils) (b) extracted values of the equivalent dielectric constants of the both samples (for sample 1 for vertical and horizontal orientations along the E fields); data for pure substrates are also given performed simulations of the whole measurement structure with incorporated metasamples show very similar values of the phase delay/advance to the measured ones (see the stars in figure 6(a)). In this first example we showed how we can measure positive or negative dielectric constant of metasamples with different polarization of the incident E field. Due to the fact, that the free space method has been implemented by waveguide-fed horn antennas with restricted frequency bands, we cannot be sure for the actual sign of the phase inside the sample body, especially for bigger thicknesses or equivalent dielectric constants. However, the next example gives clearer situation for the sign of the measured phase. One can see in figure 7(a) the phase delay/advance in a meander metasurface (part of antenna convertor from linear to circular polarization) for four different orientations of the structure according the E field of the incident wave. Now the extracted $\varepsilon_{eq}$ values clearly show two zones with $\varepsilon_{eq} > 1$ and $\varepsilon_{eq} < 1$ for three of the cases without any doubts – see the dependences in figure 7(b). The main disadvantage of the free-space method – large flat samples, could be combined with another disadvantage – time-consuming simulations for extraction of the equivalent dielectric constants. Figure 8 gives an illustration for the constructed once or twice splitted models with fed waveguides and large sample in radiation box in order to ensure satisfied distribution of the E field in the sample.
4. Waveguide (transmission-line) methods for wideband characterization of metamaterials

Another popular method for measurement of metamaterials is the waveguide method – the sample is placed inside a rectangular waveguide to fit the inner cross section (as in [9]). The last circumstance could be considered as a disadvantage – the waveguide cross-section area at higher frequency may be smaller for the chosen sizes of the unit cells and the characterization of the whole metasample could be coarse due to the poor homogenization due to the big inclusions. We applied in this paper another applicable approach – bigger sample can be placed between the open ends two sections of rectangular waveguides. Using a set of rectangular waveguides in wide frequency range (from S up to Ka band, i.e. from ~5 to 40 GHz – figure 5(b)), we managed to measure the phase delay/advance in the considered metamaterial samples from the previous section and to extract the information for the equivalent dielectric constant. In this case the samples have been pressed between the waveguide ends without any air gaps. Different phase behaviour in metasurface 1 in vertical or horizontal directions can be again observed – now in wider frequency range 5-40 GHz. Due to the close proximity of the sample to the waveguide aperture, the matching of the whole structure is poor and unavoidable ripples appear in the phase dependencies. Nevertheless, the extracted $\varepsilon_{eq}$ values are practically the same as in the previous case: $\varepsilon_{eq} \sim 5$-15 for horizontal orientation and $\varepsilon_{eq}$ from -50 to -110 for vertical orientation. The success of the measurements has been ensured due to the small sample thickness and the air-gap absence; if we increase the distance between the waveguide ends, the phase-advance behaviour become strongly influenced – figure 10(a). We verified numerically these effects – the calculated phase delay/advance strongly depends on the gap distance, but mainly for samples with $\varepsilon_{eq} < 1$ and $\varepsilon_{eq} < 0$ – figure 10(b, c). The influence becomes more stable, when the gap distances become comparable with the distances between the antennas in the free-space method. This circumstance could be considered as a serious disadvantage of the popular rectangular waveguide method.

There exist wideband planar transmission-line methods to characterize the dielectric parameters – the
Figure 10. (a) Influence of the air-gap distance between the waveguide ends on the measured relative phase advance by waveguide method; (b, c) simulated relative phase for different air-gap distances for $\varepsilon_{eq} \sim 0$

Figure 11. (a) Measured relative phase delay/advance in metasamples, which cover planar lines (CPW or MSL); (b) Samples' arrangement and measurement setup by vector network analyser in wide frequency band

Figure 12. (a) Measured relative phase delay/advance in Graphene and Graphite samples by covered CPW (diameter ~ 9 mm; thickness 0.9 mm; (b) extracted $\varepsilon_{eq}$ values (inset: dielectric constant of Graphene by [23]))

samples could be placed into or over the corresponding planar line: stripline, microstrip line, coplanar
waveguide. We have developed a method for characterization of the equivalent dielectric constant of dielectric substrates placed on microstrip lines (MSL) or coplanar waveguides (CPW) as overlays [22].

The present investigations show that this method is applicable also to metamaterials. The mentioned planar lines have enough “high” E fields above the microstrip conductor, which can be influenced by the sample presence (predominant parallel fields in CPW and mixed parallel and perpendicular fields in MSL). Really, we manage to measure the phase delay/advance in metasamples on CPW and MSL and they show very similar behaviour as in the previous case – see figure 11. A special distinction is that in order to minimise the strong screening effect of the surface metal inclinations on the dominant modes in the transmission lines, we applied small air gap (0.1 mm). However, exactly this gap cannot give us the possibility for reliable extraction of the dielectric constants, when \( \varepsilon_{eq} < 1 \) (like for the waveguide method). The other problem is the existence of cut-off frequency in CPW (bellow this frequency different mode than dominant one propagates in CPW), and the measurements are not representative. Nevertheless, the covered CPW method is very successful for characterization of metamaterial-like samples. We applied the method for characterization of spectral pure Graphite, pressed thick Graphene and air-filled Graphite, formed as disks – see figure 12. The measured frequency dependencies of the equivalent dielectric constant in these carbon-based materials in the frequency range 2-40 GHz are unique; only one scientific group shows similar results [23]. The proposed measurement setup allows easy realization of applying of external de biasing magnetic field.

Figure 12(a) gives the direct influence of the magnetic field on the phase delay in the carbon-based samples and possibility to extract the equivalent magnetic constant. We will continue this research.

5. Resonance methods for characterization of metamaterials' anisotropy

The resonance methods for characterization of dielectric materials are usually more accurate than the broadband ones. Our variant, the two-resonator method [4, 6], additionally allows determination of the dielectric anisotropy, figure 5(c, d). However, in the case of metamaterials we met some difficulties applying two-resonator method. First of all, the exited resonance modes should be clearly recognized for the resonator with/without sample – figure 13(a). If \( \varepsilon_{eq} > 1 \), the process is relatively easy; the modes for resonator with sample lie below the resonances in the empty resonator, where the reliable identification is possible. Thus, we managed to confirm all the measured \( \varepsilon_{eq} \) values (\( > 1 \)) in this case by other wideband methods (figures 6(b), 12(b)). The problems appear, when \( \varepsilon_{eq} < 1 \), especially for big \( |\varepsilon_{eq}| \) values. In this case the identification is very difficult and even impossible; the modes under interest should lie far above the modes in the empty resonator, where a lot of high-order modes exist. Moreover, multiple resonances appear in this case due to resonance effects in different parts of the whole metasample, as the simulations predict (insets in figure 13(a)). However, for simple metamaterial-like homogenous samples we manage to measure values \( \varepsilon_{eq} < 1 \). Figure 13(b) illustrates two high-order modes in resonator with Graphite sample above 30 GHz. The modes with sample clearly lie above the modes in the empty resonator; thus we extract values \( \varepsilon_{eq} \sim 0.55-0.85 \).

6. Characterization of the equivalent conductivity of metasurfaces

The last example in our considerations is a resonance method for characterization of the conductivity of the metamaterials. Nowadays the need to know the actual values of the conductivity of different metals or other materials becomes actual due to the requirements of the modern design and the new technologies that use artificial metalized plastic details, 3-D printed metallic structures, absorbers and metamaterials. We proposed in [5] an efficient resonance method for determination of the equivalent conductivity; the idea is to replace one flat wall of the measurement resonator with optimized volume-to-surface ratio – figure 14(a). Due to the high own Q factors in the measurement resonator and the preliminary performed calibration with reference metals (Ag, Cu and Al), the proposed setup allows characterization of low-conductivity materials (for example carbon-based plates), including metasurfaces – see the results in figure 14(b). These are our first results for determination of conductivity of metamaterial samples; definitely, resonance behaviour of some metasurfaces has been detected. There are many new possibilities for development of this method [24].
Figure 13. (a) Measured excited TE$_{011}$ modes in cylinder resonator with meta-samples; (b) two high-order modes excited in cylinder resonator with Graphite sample (the measured $\varepsilon_{eq}$ values are $\sim 0.55$-$0.85$)

Figure 14. (a) Measurement setup with rectangular resonator with one wall, replaced with the surface under test (SUT) and three reference metals; (b) frequency dependence of the equivalent conductivity by Q-factor measurements for the 6 first order modes. Legend: 1 – metasurface; 2 – metadisks; 3) pure carbon fabrics; 4) carbon fabrics + epoxy; 5 – carbon nanotube (CNT) fabrics + epoxy

7. Conclusions

The characterization of metamaterials is more difficult process compared to the characterization of the standard dielectrics, but fully possible. The free-space method allows measurements of different values of the dielectric constant: $\varepsilon_{eq} > 1$, $0 < \varepsilon_{eq} < 1$, or even $\varepsilon_{eq} < 0$, but needs big samples and serious computer resources. The broadband waveguide methods use smaller samples, but the results depend on the distance between the waveguide open ends for $\varepsilon_{eq} < 1$. Variant with planar covered transmission lines (CPW or MSL) gives accurate results and the models are easy for simulations; however, an influence of the air gap between the sample and line conductor exists and the method usually underestimates the measured dielectric constants. Moreover, the method easy allows investigation of magnetic properties (magnetic metamaterials). The resonance methods give very accurate reliable results, but needs preliminary information for the possible values of the dielectric constant. Finally, reliable determination of equivalent conductivity of metasurfaces by resonance method is also possible with satisfied accuracy.

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