Abstract

The design concept of effective microwave absorbers and compact matched loads based on 3D-printable lossy nanocarbon-based composites with filler content above the percolation threshold is proposed. The DC-conductive ($\sigma_{DC} = 0.39 \, \text{S m}^{-1}$) 3D-printable filament based on poly(lactic) acid filled with 12 wt.% of multiwalled carbon nanotubes was used. The electromagnetic properties of 3D-printed pyramidal regular structures were experimentally investigated and numerically simulated in 12–18 GHz (Ku-band) and 26-37 GHz (Ka-band) frequency ranges. Within the proposed model the structures under study were considered as graded refractive index material. The optimal geometrical parameters of designed microwave components were successfully evaluated using numerical modeling. Tested components demonstrate remarkable shielding efficiency (> 20 dB) within whole Ku- and Ka-bands and are suitable for practical application related to effective absorption of microwave radiation. The production of 3D-printable materials with controlled and predicted losses offers the possibility for miniaturization of 3D-printed microwave components, such as absorbers and loads. The developed technique, estimating the geometrical parameters of the components vs dielectric properties of the conductive filament, could be used as a versatile platform for predesign of compact microwave devices taking into account constituent dielectric parameters of available printable materials and filaments.

Keywords: 3D-printing, conductive 3D-printable filament, microwave electromagnetic response, Ku-band, Ka-band.

(Some figures may appear in colour only in the online journal)
compatibility [1–3]. The waveguide matched load is a common example of a device, which absorbs electromagnetic energy almost without reflecting the incident electromagnetic wave. Usually, the design of such components is based on a long wedge or pyramid placed in the center of the waveguide [4]. The wedge is made of lossy material (e.g. dispersed carbonyl iron particles in the epoxy resin) and its top is oriented to the incident wave source. The existence of the optimal value of complex dielectric permittivity (or electrical conductivity in the range of "several Siemens per meter" for microwave frequencies) for effective absorption in the material was shown previously [5, 6]. Nevertheless, in practice, it is often difficult to achieve optimal dielectric permittivity and control simultaneously both material losses and its mechanical properties. The losses in available conventional materials are often below optimal values leading to an increase in the length of the wedge to achieve the necessary level of electromagnetic attenuation.

The fabrication of 3D-printable components and materials with controlled and predicted losses offers the possibility for miniaturization of absorbers and loads. We will show that DC-conductive filament of intermediate electrical conductivity (i.e. \( \approx 1 \text{ S m}^{-1} \)) is a universal mean for microwave attenuation elements design by 3D printing. We will present the laboratory process for conductive filament production and then use it for 3D-printing of pyramidal structures to prove the proposed concept of light and compact matched loads.

The periodic pyramidal structures are used as broadband absorbers for the anechoic chambers [7, 8]. Viskadourakis et al investigated the shielding efficiency of the lossless pyramidal structure in 3.5–7 GHz [1]. Normikman et al studied the hexagonal pyramids [9] in 1-20 GHz, Jenkins [10] applied the pyramidal structures as antenna for 3.3–8.0 GHz frequencies. However, all the mentioned components were produced from non-conductive plastics. The development of the lossy filament opens new possibilities for the 3D printing techniques in the field of electromagnetic interference (EMI) shielding applications [3].

The aim of the present paper is to study the possible application of 3D printing technology for the shielding components and matched loads design. The method of the optimal geometrical parameters evaluation is presented. The effectiveness of the method is verified by experimental studying the shielding performances of the printed structure.

2. Model for electromagnetic properties simulation

The classical matched load working principle is based on a smooth transition from an empty waveguide to a waveguide filled with lossy material. The smooth transition is necessary to vanish the reflected power from inhomogeneity inside the waveguide. Usually, the lossy region is made in the shape of a long wedge or pyramid and placed in the center of the waveguide, which top oriented to the incident electromagnetic wave (figure 1 inset). The coordinate of the base of pyramid is as follows [11, 12]. In references [13, 14] we showed experimentally and numerically that the scattering parameters of the homogenized layer are equivalent to the initial structure for spheres, hollow spheres, corrugated composites and similar structures inside the waveguide.

The cross-section of a waveguide along the plane perpendicular to the pyramid base consists of the rectangle-like lossy region and air region. The homogenization procedure, in this case, means that the air regions can be averaged with lossy regions according to their relative surface fractions \( S(x) \). The dependence of the effective refractive index \( n \) on the coordinate \( x \) is as follows [11, 14]:

\[
n(x) = n_p(1 - S(x)) + n_0 S(x),
\]

where

\[
S(x) = \begin{cases} 
\frac{1, x < -(h_0 + dh)}{1 - \left( \frac{h_0 + dh + h}{dh} \right)^2}, (h_0 + dh) < x < -h_0 \\
0, -h_0 < x < 0 \\
1, x > 0 
\end{cases}
\]

and \( n_p = \sqrt{\varepsilon} \) is the refractive index of pyramid’s bulk material, \( n_0 = 1 \) is the refractive index of air. The spatial distribution of refractive index \( n(x) \) equation (1) is parabolic and presented in figure 1.

The relative amplitudes of reflected \( S_{11} \) and transmitted \( S_{21} \) trough the pyramid signals may be easily calculated using a multi-layered approach developed in optics and discussed in detail in [13–15]. The shielding efficiency \( SE \) is defined [16, 17] as \( SE_T = -20 \log_{10} S_{21} \). Similarly, the efficiency due to reflectance is \( SE_R = -20 \log_{10} S_{11} \).
The presented model gives the dependence of the shielding efficiency of pyramids placed in the waveguide transmission line on their geometrical parameters \((h_0, dh)\) and dielectric properties \((\varepsilon)\). Important to note, that in case of an array of pyramids inside the waveguide or in the free space it is enough to consider and perform averaging and homogenization within one unit cell. The model was implemented using MatLab software.

3. Samples preparation and characterization

3.1. Conductive filament production

The DC-conductive Graphene3D filament is based on the poly(lactic) acid (PLA) Ingeo\textsuperscript{TM} Biopolymer PLA-3D850 (Nature Works) with a 12 wt.-%-content of -OH modified multiwalled carbon nanotubes (MWCNTs) supplied by TimesNano, China. The following procedure was used to prepare DC-conductive Graphene3D filament ready for further 3D-printing.

Firstly, the masterbatch of 12 wt % MWCNTs was prepared by melt mixing of the filler and the polymer in the twin-screw extruder (COLLIN Teach-Line ZK25T) by setting a screw speed of 40 rpm and keeping the temperature in the range 170–180 °C. After that, the composite pellets were extruded by a single screw extruder (Friend Machinery Co., Zhangjiagang, China) in the temperature range 170–180 °C and a screw speed of 10 rpm for producing filament for 3D printing (FDM) with 1.75 mm in diameter. Below we will call the obtained material as Graphene3D filament.

3.2. Filament properties and printing

Scanning electron microscopy (SEM) analysis was performed to get information about the dispersion of nanofiller in the PLA host and its effect on the microstructure. Figure 2(a) and (b) showed the surfaces of both neat PLA and 12 wt% MWCNT/PLA, respectively, after liquid nitrogen breakage of the filament. Very different fracture surfaces are visible for the tested PLA and composite filaments, which are largely attributed to their brittle or ductile mechanical behavior. The neat PLA surfaces appear very flat due to the ductile fracture type, typical for an isotropic polymeric material. In contrast, a network type structure is developed over the entire surface of the MWCNT/PLA composite, due to the interconnection of well-dispersed MWCNTs and to a fine structure of micro-voids that is typical for a more brittle material.

In general, the network filler–polymer microstructure, formed by the strong and conductive MWCNTs in the PLA matrix is typically associated with percolation, which may result in enhanced mechanical and physical properties of nanocomposites compared to the neat PLA. The details of mechanical properties, electrical and thermal conductivity and electromagnetic shielding efficiency of both neat PLA and 12 wt% MWCNT/PLA filaments, obtained from our previous studies \([3, 18, 19]\) are summarized in table 1. As seen, the addition of 12 wt% MWCNTs enhance significantly mechanical properties of the filament, e.g. tensile elastic modulus and hardness, but decrease twice the elongation at ultimate strength, compared to the neat PLA. Moreover, the composite filament demonstrates twice higher thermal conductivity, compared to the PLA. This confirms the microstructural
prediction, that the percolation structure of 12 wt% MWCNTs in the nanocomposite filament is highly conductive, lossy and stronger, but more brittle, than the neat PLA.

The improvement of Young’s modulus (21%), hardness (11%) and electrical conductivity (10 decades) could be associated with the dense, conductive network structure formed by the carbon nanotubes above the percolation threshold, which allows a transfer of the extraordinary mechanical and electrical properties of carbon nanotubes through the polymer. In contrast, a twice decrease of % elongation of the composite filament compared to the neat PLA may be attributed to the large surface area of the filler which absorbs most of the polymer at the interfaces, as shown in figure 2(b), which leads to increase of the brittleness of the composite material [3, 19]. However, the thermal conductivity of 12 wt% MWCNT/PLA filament was observed only twice higher compared to the neat PLA, in spite of the extremely high thermal conductivity of carbon nanotubes (3000 W/mK). This can be explained by the complex process of thermal diffusion through a polymer, influenced by temperature, crystallinity, macromolecular orientation, etc. Moreover, carbon nanotubes within polymers are usually considered to have many defects that contribute to numerous phonon scattering lowering the thermal conductivity [18].

The fused deposition modeling (FDM-FFF)-type 3D printer X400 PRO German RepRap with an extrusion nozzle with a diameter of 0.5 mm was used. During printing, the filament was heated above its melting temperature and then extruded using a PC-controlled moving nozzle [3]. Thus, the desired 3D-structure is formed as a result of a layered process. The processing parameters of the 3D printing were a temperature of 200 °C, an extrusion speed of 100 mm/min, and the platform temperature of 60 °C. Samples were printed with 100% infill, in a rectangular direction of one layer to another, as shown in figure 2(c).

3.3. Waveguide measurements

The microwave electromagnetic measurements performed using the waveguide method [20, 21]. A vector analyzer MICRAN R4M-18 with 16 × 8 mm² and a scalar network analyzer Elmiika R2-408 R with 7.2 × 3.4 mm² waveguide systems were used for Ku- and Ka-bands, respectively. Real images of waveguide systems are shown in the figure 6. For both setups, the frequency dependencies of the scattering parameters, transmitted/input ($S_{21}$) and reflected/input ($S_{11}$) signals were measured.

4. Results

The complex dielectric permittivity and refractive index of the Graphene3D filament recalculated from experimentally measured S-parameters of the printed plane-parallel layer [20, 21] are presented in figure 3. The filament has high loss tangent (not less than 0.4 within the whole frequency range). Moreover, the material is dispersive, its dielectric permittivity is generally decreasing with frequency. This type of dispersion is typical for nanocarbon composites with filler content above the percolation threshold in microwave frequency range [22, 23].

The minimal height of pyramids and the substrate thickness were computed using proposed model equations (1) and (2). Several simplifications were made. The frequency was fixed as 30 GHz, the effective shielding criteria were introduced as $SE_T > 20$ dB and $SE_R > 20$ (this is equivalent to the absorption of more than 99% of the power of incident wave).

For the computations of $dh$, substrate height was taken as $h_0 = 2$ mm. In this case, $SE$ of the pyramid is dependent only on its height and dielectric permittivity of used material. The combinations of $dh$ and $\varepsilon$ that satisfy the mentioned criteria for $SE$ are presented as the regions in $\varepsilon''(\varepsilon')$ coordinates in figure 4. The decrease of the $dh$ result in narrowing the region of possible $\varepsilon$ combinations. The measured dielectric permittivity of the Graphene3D filament at 30 GHz is $\varepsilon = 16.74 - i6.17$ (figure 3). The minimal pyramid’s height $dh$, required for the effective shielding is 8–9 mm for the Ku-band. Similarly, $dh = 22$ mm was evaluated for the Ku-band.

For substrate thickness $h_0$ computations the obtained $dh = 8$ mm was used. The dependencies $SE_T$ and $SE_R$ on the substrate thickness $h_0$ are presented in figure 5 (filled symbols). The oscillations of $SE_R$ related to the interference, while $SE_T$ increases monotonously. These oscillations may be significantly damped by increasing the height of the pyramids $dh$. The $SE$ vs $h_0$ for the plane-parallel layer ($dh = 0$ mm) are also presented in figure 5 (see open symbols). Both $SE_R$ and $SE_T$ of the plane layer are significantly lower in comparison with the pyramidal structure. The $SE_T$ expectedly increases with the thickness, but the $SE_R$ remains lower than 5 dB. It means that a planar layer cannot simultaneously demonstrate high $SE_R$ and $SE_T$ parameters (or in other words absorption ability) at any substrate thickness.

The proposed graded index approach is useful for the practical design of pyramidal matched loads. It provides...
Table 1. Mechanical and physical properties of the filaments.

| Filament type | Tensile strength (MPa) | Tensile young’s modulus (MPa) | Elongation (%) | Hardness (MPa) | Electrical conductivity (S/m) | Thermal conductivity (W/mK) |
|---------------|------------------------|-------------------------------|----------------|----------------|-----------------------------|---------------------------|
| PLA           | 28.1±3.9               | 593.6±8.5                    | 8.5±1.6        | 176±5          | 8.3×10^{-11}               | 0.18±0.03                 |
| 12 wt.% MWCNT | 23.3±1.9               | 719.9±6.2                    | 4.3±0.4        | 195±9          | 0.39                        | 0.37±0.02                 |

Figure 4. The pyramid height $dh$, required for the effective (20 dB) EMI shielding, presented as the dependence on the dielectric permittivity. Dot stands for the measured $\varepsilon$ of Graphene3D filament at frequency 30 GHz.

Figure 5. The dependence of the $SE_T$ and $SE_R$ on the substrate thickness. Pyramid heights are $dh = 8$ mm (close symbols) and $dh = 0$ mm (open symbols).

Figure 6. 3D printed pyramidal structures and waveguide systems for Ku-band on the left and for Ka-band on the right.

$dh = 22$ mm (Ku-band) are the minimal parameters, which allow to meet 20 dB level for both $SE_T$ and $SE_R$.

The printed samples and the experimentally measured in Ku- and Ka-bands shielding efficiency are presented in figures 6 and 7, correspondingly.

Both structures for Ku- and Ka-bands demonstrate a high level of $SE_T > 20$ dB and $SE_R > 20$ dB, even despite some printing issues (see figure 6), and may be used as effective matched loads or absorbers in anechoic chambers. Usage of lossy material with high $\varepsilon'$ and $\varepsilon''$ allows to obtain similar shielding performance with smaller pyramids (see table 2). Even more, in contrast to the results listed in table 2, we demonstrate that both transmitted and reflected signals are well attenuated. It is possible due to the combination of high Ohmic losses within the material bulk and waves scattering due to the sample’s geometry. Important to note, that high Ohmic losses in considered materials were achieved because of forming DC conductive MWCNT-based network within the polymer matrix. Due to depolarization effects [23] the MWCNT agglomerates in the composite below percolation weakly interact with microwave radiation. Nevertheless, when agglomerates are incorporated in the percolative network of...
Table 2. Comparison of the shielding performances of the pyramidal structures.

| Material parameters | $d_h$, mm | $h_0$, mm | $S_{ER}$, dB | $S_{ET}$, dB | Frequency, GHz | Reference |
|---------------------|-----------|-----------|---------------|--------------|----------------|-----------|
| $\varepsilon = 2$, $\tan\delta = 0.2$ | 90 | 25 | 45 | n/a | 10 | [24] |
| $\varepsilon = 2.492$, $\tan\delta = 0.956$ | 130 | 20 | 42.93 | n/a | 10-15 | [9]*hexagonal |
| $\varepsilon = 2.9$, $\tan\delta = 0.084$ | 130 | n/a | 43.294 | n/a | 10-15 | [25]*triangular |
| BlackMagic 3D | 2 | 2 | 12 | n/a | 5.5 | [26] |
| VeroBlack | 80 | 20 | 20 | n/a | 100 | [27] |
| $\varepsilon = 1.5$, $\tan\delta = 0.26$ | 40 | 20 | 40 | n/a | 30 | [28] |
| $\varepsilon = 18.66$, $\tan\delta = 0.38$ | 22 | 2 | 40.15 | 28.09 | 17.4 | this research |
| $\varepsilon = 15.55$, $\tan\delta = 0.39$ | 8 | 2 | 23.88 | 39.17 | 35.7 | this research |

DC-conductive composite (which is exactly corresponds to our experimental situation) they contribute to effective scattering and attenuation of the electromagnetic waves.

5. Conclusions

The DC-conductive filament of 12 wt% MWCNT/PLA, suitable for 3D-printing was developed for effective absorbers and compact matched loads design. The pyramidal structures were 3D printed and experimentally tested in Ku- and Ka-bands. The pyramid height and the substrate thickness were obtained through the optimization of the shielding efficiency versus the complex permittivity of the filament. Measured shielding efficiency of the printed samples is higher than 20 dB both for reflected and transmitted signals in the investigated frequency ranges.

To conclude, the lossy periodic pyramidal structures 3D-printed from conductive filament are perspective for the fabrication of effective absorbers and waveguide matched loads. The developed technique for the pyramid parameters evaluation may be effectively used as a pre-experimental step since it takes into account the material properties (both for lossy and lossless), required frequency range, and the substrate thickness. This opens the road toward robust pre-design of compact microwave components taking into account constituent dielectric parameters of available printable materials and filaments.

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Figure 7. Shielding efficiency and S-parameters (inset) of the 3D printed samples in Ku- and Ka-bands.
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