Creation of metasurface from vertically aligned carbon nanotubes as versatile platform for ultra-light THz components

G V Gorokhov1,2, D S Bychanok1,3, P P Kuzhir3,1, D V Gorodetskiy5, A G Kurenya5, O V Sedelnikova5, L G Bulusheva5 and A V Okotrub3,5

1 Institute for Nuclear Problems, Belarusian State University, 11 Bobruiskaya str., 220030, Minsk, Belarus
2 Physics Faculty, Vilnius University, Sauliškė 9, 10222, Lithuania
3 Tomsk State University, 36 Lenin Ave, Tomsk 634050, Russia
4 Institute of Photonics, University of Eastern Finland, Yliopistokatu 7, FI-80101 Joensuu, Finland
5 Nikolaev Institute of Inorganic Chemistry, SB RAS, 3 Acad. Lavrentiev Ave., 630090, Novosibirsk, Russia

E-mail: glebgorokhov@yandex.ru

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Abstract
Here a simple and reproducible method for obtaining terahertz metasurfaces formed from multiwall carbon nanotubes (MWCNTs) is presented. The metasurfaces were obtained from a vertically aligned array of MWCNTs using a laser engraving technique followed by polymer covering. The structures under study demonstrate frequency-selective reflection in terahertz range following the Huygens–Fresnel formalism. For a normal incidence of the electromagnetic wave, the model for numerical calculation of backscattering from the metasurfaces was proposed. Lightweight and compact MWCNT-based metasurfaces are capable to replace conventional pyramidal absorbers and could serve as a versatile platform for scalable cost-efficient production of ultra-light electromagnetic components for THz applications.

Keywords: terahertz absorption, carbon nanotubes, diffraction gratings, periodic structures

Some figures may appear in colour only in the online journal.

1. Introduction
Carbon nanotubes (CNTs) are well-known as a perfect filler [1–6] and even the substrate [7] for producing lightweight composites for electromagnetic applications. Along with graphene [8] the unique properties of CNTs allow creating compact durable and/or flexible [9] electromagnetic components and nano-devices, such as antennas [10–12], interconnects [13, 14], polarizers [15, 16], sensors [17], detectors (see [18] and Refs therein) and emitters of sub-mm waves radiation (see [19] for review).

Such wide applicability of CNTs is due to their unique electromagnetics arising from plasmon-polariton (i.e. slowed-down surface wave) propagation along CNT axis [20], as well as so-called ‘finite length’ effects (i.e., localized plasmon resonance) inherent for micron-length single-walled nanotube at THz and far-infrared frequencies [21, 22]. The valuable skin effect caused by electromagnetic radiation screening in low frequency ranges up to microwaves was predicted and experimentally observed for long multi-walled carbon nanotubes (MWCNTs) [23, 24]. The recently demonstrated negative photo-induced conductivity of CNTs [25] supported the possibility of ultra-fast tuning of their THz optical density that opens a way for the development of electromagnetic devices.

To summarize, CNT-based percolated composites [4, 6, 26], films [1, 17], meshes and sponges [27] behave like ultra-lightweight quasi-metals. When single-walled CNTs are relatively short (about hundreds of nanometers), the materials’ conductivity spectra exhibit peaks located at THz range. In case of long single-walled [28] and MW CNTs, and CNT bundles [29], the conductivity peak shifts from THz range towards much lower frequencies (hundreds of MHz and GHz).

However, when individual CNT’s electromagnetic parameters are not comparable with the wavelength, CNT-based
array, film or composite acts as a macroscopically homogenous structure due to slowing down effect. Thus their electromagnetic response is governed by the averaged conductivity, rather than ‘fundamental’ electromagnetics of individual nanotube. In such a case, one typically has porous conductive structure with broadband absorption [27], which properties are dependent on the CNT array/film density, conductivity and geometry of individual tubes forming the array, inter-tubes contact resistance, etc. In order to reach the electromagnetic properties required by a particular application the metamaterial paradigm [30–33] can be applied to the CNT array. The possible solution is combining the intrinsic properties of nanotubes with particular patterning, which providing constructive interference of electromagnetic waves [34].

The idea of this communication is to propose the versatile platform for scalable cost-efficient production of ultralight electromagnetic components based on patterned vertically aligned arrays of MWCNTs. We demonstrate that MWCNT arrays grown via conventional aerosol assisted synthesis may be easily machined using a laser engraving technique in order to obtain complex geometry structures. As a proof of concept, several 3D-metasurfaces have been successfully produced of pyramidal CNT arrays with pyramids height about 0.1–0.5 mm (see figure 1) have been successfully produced and experimentally examined in 0.1–1 THz frequency range. At the same time, the possibility of tuning the electromagnetic response of such metasurfaces was studied theoretically in 0.01–10 THz frequency range. The studied 3D CNT-based metasurface covered with a thin layer of insulating polymer is proved to be effective anti-reflection coating and reflection selective surface for THz range, supporting the ideology of scalable protocol of producing 3D metasurfaces composed of CNT-arrays as microwave-to-THz components.

The present paper is organized as follows. Section 2 contains the details of ordered MWCNT arrays preparation, engraving and experimental terahertz measurements. The basic principles of investigated structure electromagnetic response modeling are described in section 3. Herein, the theoretical approach is compared with the experimental and absorption properties of the ordered CNT arrays are discussed. Finally, the Conclusions section summarizes the findings and gives an outlook on potential application of such metasurfaces.

2. Experimental

2.1. Preparation of materials

Vertically aligned MWCNT arrays were grown on silicon substrates using an aerosol-assisted catalytic chemical vapor deposition (CCVD) method described elsewhere [35].

A silicon substrate was placed into a tubular oven constantly flowed with argon and heated at 800 °C. The synthesis was carried out using 2 % ferrocene (Fe(C5H5)2) solution in toluene (C6H5CH3). As a result, with the use of 2.5 ml of the reaction mixture an aligned MWCNT array of ∼ 250 μm height was obtained. To examine the morphology of MWCNTs, the pristine sample was investigated by transmission electron microscopy (TEM) using a JEOL 2010 microscope. TEM image of obtained MWCNTs is shown at figure 1(a). Average diameter of nanotubes is ∼ 6 nm.

The plane-parallel MWCNT array was transformed to the array of pyramids using laser engraving. The industrial laser engraver (Winseal, China) with 20 Wt CO2 laser and 20 mm/s scanning speed was used. Grating period was 250 μm. To study the structure of the engraved sample and to prove the preservation of nanotubes after the laser treatment, the obtained sample was investigated by scanning electron microscopy (SEM) using a JEOL JSM 6700 F microscope. The SEM images of engraved pyramids are presented in figures 1 (b) and (c).

After the engraving array of pyramids was covered with epoxy resin in order to protect its fragile structure. The viscous epoxy resin (Crystal 76) was dropped and then spread over the engraved surface under the vacuum. Total height of pyramids after all manipulations measured by means of optical microscopy was 232.9 ± 11.9 μm with 54.9 ± 6.9 μm uncut layer at the bottom. Maximal thickness of epoxy resin between pyramids was 77.0 ± 4.5 μm. General view of structures after covering with epoxy resin is presented in figure 1(d). The impact to electromagnetic response of the investigated system done by polymer cover is discussed in the section 3.

2.2. Terahertz measurements

The THz measurements were carried out using a commercial THz time domain spectrometer ‘TSPEC’ by EKSPLA in 0.1–1.0 THz frequency range. The sample was placed normally to the initial electromagnetic wave. According to its functioning principle, the THz spectrometer registers the waveform of THz electrical field with perfect reflector (as a reference) and with sample (as experimental data). For the purpose to increase signal-to-noise ratio each measurement is...
 averaged over 1024 frames. In order to switch between time and frequency domains, the Fourier transform was used. The reflection coefficient is evaluated as a ratio between powers of electromagnetic waves reflected by the sample and the reference.

3. Modeling

3.1. Periodic structure contribution

Let us consider the backscattering of plane wave from the infinite array of conductive square pyramids with height $h$ and base width $l$ (see figure 2).

There are several approaches to calculate the amplitude of the signal reflected by such a structure. In the low frequency region, the wavelength is much higher than the characteristic lateral size of pyramids that allows to implement the long-wave approximation through the Huygens–Fresnel principle. At low frequencies, the wavelength is much higher than the pyramid height $h$. The total contribution of the edge 1 is:

$$E_{11} = \frac{E_0 \cos \Theta \exp(i\omega t - ik\Delta_x)}{S_{edge}} dS = \frac{E_0 \cos \Theta \exp(i\omega t - ik2x)}{h^2} 2\pi dx,$$

where $k$ is the wave vector, $h$ is the height of pyramid.

The total contribution of the edge 1 is:

$$E_{11} = \frac{E_0 \cos \Theta \exp(i\omega t)}{h^2} \int_0^h 2\pi \exp(-ik2x) dx = \frac{E_0 \cos \Theta \exp(i\omega t)}{2\pi h^2} [\exp(-ikh)(1 + ikh) - 1].$$

The contribution of the edge 3 is the same as equation (2). Contributions of the edges 2 and 4 may be obtained from equation (2) by substitution of $\cos \Theta$ for $\sin \Theta$.

Summarizing the impact of all four edges, it is possible to obtain the scattering parameter $S_{11}'$ (ratio between reflected and incident radiation amplitudes) for the surface paved with pyramids:

$$S_{11}'(\nu, h) = \frac{(1 + 2ikh) \exp[-i2kh]}{2k^2 h^2} - 1.$$

Figure 3 presents the $S_{11}'$ frequency dependence for various $h$. Here, we supposed that pyramids were composed of electric conductor, i.e. the flat surface of such material has $|S_{11}| = 1$.

The increase of pyramid height $h$ shifts the $S_{11}'$ spectrum to the low frequency region. Figure 3 clearly depicts the transition between long-wave approximation through the Huygens–Fresnel theory to the geometric optics region. At low frequencies, the wavelength is much higher than the pyramid height $h$, thus the structure interacts with radiation as a perfect reflector. When the wavelength is comparable with $h$, the decreased with
relatively small oscillations caused by interference. Finally, at high frequencies, the $S_{11}^0$ value is significantly damped and back-reflection becomes negligible.

### 3.2. Dielectric Layer contribution

A widely known example of matching layer is the optical lens antireflective coating, which decreases the difference between refractive indices of free space and lens making their interface less reflective. For the pyramids array covered with dielectric layer the back-reflected signal amplitude is also dependent on the dielectric permittivity $\varepsilon$ and thickness $\tau$ of the latter one. To calculate dielectric layer contribution, it is necessary to take into account the interference between waves reflecting from top and bottom surfaces of dielectric layer covering the pyramids. The electric field $E_I$ in the region above dielectric layer (in free space) and electric field $E_H$ inside the layer may be determined as:

$$
E_I = C_1 \exp[-ik_1 x] + C_2 \exp[i k_1 x],
$$

$$
E_H = C_3 \exp[-ik_2 x] + C_4 \exp[i k_2 x],
$$

where $C_1, C_2, C_3$ are unknown coefficients, $k_1 = \frac{2 \pi \nu}{c}$ and $k_2 = \frac{2 \pi \nu}{\sqrt{1 - \varepsilon}}$ are the wavenumbers in the free space and in the dielectric layer respectively, $\nu$ is the frequency, $c$ is the speed of light. The amplitudes of initial and reflected waved were taken to be $C_3$ and $\alpha C_3$ respectively, a term $(|\alpha| \leq 1)$ implies imperfection of CNT array as conductor. Therefore, $\alpha$ may be considered as amplitude of the signal reflected by a plane surface of conductive material with semi-infinite depth (in considered case, the CNT array) into a medium (polymer) with the dielectric constant $\varepsilon$. Equation (4) should satisfy the following boundary conditions:

$$
E_I(-\tau) = E_H(-\tau),
$$

$$
\frac{\partial E_I}{\partial x} |_{-\tau} = \frac{\partial E_H}{\partial x} |_{-\tau}. \tag{5}
$$

Solving equation (4) and (5) allows to obtain the amplitude of reflected signal from the plane-parallel layer of dielectric with non-perfect back reflector:

$$
S_{11}^d(\nu, \tau, \varepsilon, \beta) = \frac{C_2}{C_1} \frac{\sqrt{\varepsilon} (1 - \beta)/(1 + \beta)}{\sqrt{\varepsilon} (1 - \beta)/(1 + \beta)} \exp[-i 2 \pi \nu \frac{h}{c} x]. \tag{6}
$$

Equation (6) describes the contribution of dielectric layer to the reflection coefficient of epoxy-covered pyramidal CNT array. The parameter $\alpha = \frac{\sqrt{\varepsilon} (1 - \beta)/(1 + \beta)}{\sqrt{\varepsilon} (1 - \beta)/(1 + \beta)}$ is related to the amplitude of reflected signal $|\beta| \leq 1$ from plane back reflector in the free space. When $\beta = -1$, equation (6) coincides with the amplitude of reflected signal from dielectric layer located on the perfect conductor [39] excepting the phase factor $\exp[2 \pi \nu k_1 x]$. In order to represent the normal reflection of THz wave from plane-parallel MWCNT array, $\beta = -0.9$ was used.

![Figure 4. Frequency dispersion of $S_{11}$ scattering parameter of polymer-covered pyramid-based reflector at different dielectric layer thickness $\tau = 0.1, 0.2, 0.3$ mm. (\beta = -0.9, $\varepsilon = 3 - 0.4i$).](image)

Figure 4 shows the frequency dependence of $S_{11}$-parameter for the dielectric layer of $\tau = 0.1, 0.2, 0.3$ mm thickness.

The typical value for epoxy resin permittivity in THz frequency region $\varepsilon = 3 - 0.4i$ was used. Figure 4 depicts the typical interference oscillations, which are absent for non-covered pyramids. When the dielectric layer becomes thicker, these oscillations shift to the low-frequency region.

### 3.3. Combination of dielectric layer and structure contribution

The real pyramidal array was impregnated with epoxy resin to overcome the pristine pyramids brittleness. The electromagnetic response of such structure is defined by both contributions from dielectric layer and from pyramidal back reflector. Due to surface tension forces, the epoxy resin unevenly covers the CNT pyramids array. As a first approximation, we considered the case when the thickness of the epoxy increases linearly with approaching the base of the pyramid (figure 2(d)). In this case the amplitude of reflected signal from the unit cell may be calculated as:

$$
S_{11} = \frac{2}{l} \int_0^l S^d_{11}(\nu, \frac{h}{l}, x, \varepsilon, \beta) \times \exp[-i 2 \pi \nu \frac{h}{c} x] dx, \tag{7}
$$

where $l$ is the half length of pyramids base, $h$ is the height of epoxy layer near the pyramids base. The first multiplier in equation (7) related to the dielectric layer contribution, the second is connected with the phase shift caused by the structure.

### 3.4. Comparison between experiment and modeling

The comparison between experimentally measured amplitude of the signal reflected by the array of pyramids covered
the electromagnetic radiation in the submillimeter frequency defines the dumping rate of the reflected signal.

ness of the dielectric cover, while the height of pyramids in the THz range. Its position is determined by the thick-

The concept of frequency selective reflector based on pyramidal metasurfaces was implemented and investigated. The impact of pyramids height and dielectric covering layer to the electromagnetic response of investigated structures was theoretically described in a wide frequency range. The possibility to control the reflective behavior of the metasurfaces was theoretically substantiated.

The metasurface was produced by the laser engraving of vertically aligned MWCNT array followed by the dielectric layer covering. In accordance with the theoretical prediction, the experimental reflection spectrum exhibited the local minima in the THz range. Its position is determined by the thickness of the dielectric cover, while the height of pyramids defines the dumping rate of the reflected signal.

The present design of metasurfaces effectively absorb the electromagnetic radiation in the submillimeter frequency range is one of the numerous examples of THz component (such as frequency selective surfaces, filters, lenses, attenuators, etc) that is possible to be realized using perfect absorption ability and electromagnetic response peculiarities of 3D-patterned CNT arrays. Targeting to the technology readiness level 3, introduced experimental observations supported by simple analytical model provide a solid laboratory-proved background for the scalable cost-efficient technological protocol of ultra-lightweight THz components.

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ORCID IDs

G V Gorokhov https://orcid.org/0000-0003-3102-8930
D S Bychanok https://orcid.org/0000-0002-7055-4274
P P Kuzhir https://orcid.org/0000-0003-3689-0837
D V Gorodetskiy https://orcid.org/0000-0002-3446-7480
A G Kurenya https://orcid.org/0000-0001-6571-2218
O V Sedelnikova https://orcid.org/0000-0002-0491-3208
L G Bulusheva https://orcid.org/0000-0003-0039-2422
A V Okotrub https://orcid.org/0000-0001-9607-911X

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Figure 5. Amplitude of back-reflected signal $S_{11}$ from periodic pyramidal CNT-array covered with epoxy resin layer (symbols correspond to the experimental data, line—to the modeling results).

with epoxy resin (figure 1 (d)) and fitted values obtained by equation (7) is presented in figure 5.

The experimental data are in good agreement with modeling results. Mean absolute percentage error value was 6.4 %. The difference between experimental results and modeling simulation may be related to the non-linear dependence of epoxy layer thickness in the region near the pyramids base. The model curve in figure 5 was obtained with the following set of parameters: $\beta = -0.9$, $\varepsilon = 3 - 0.4i$, $h_e = 0.09$ mm, $h = 0.25$ mm. The results showed that the produced metasurface acts as reflection selective surface with maximal absorption near $\nu_0 = 700$ GHz. Below $\nu_0$ the amplitude of signal reflected from CNT array is 0.8, while above $\nu_0$ the $S_{11}$ amplitude is near 0.4.

Conclusions

The concept of frequency selective reflector based on pyramidal metasurfaces was implemented and investigated. The impact of pyramids height and dielectric covering layer to the electromagnetic response of investigated structures was theoretically described in a wide frequency range. The possibility to control the reflective behavior of the metasurfaces was theoretically substantiated.

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The presented design of metasurfaces effectively absorb the electromagnetic radiation in the submillimeter frequency range is one of the numerous examples of THz component
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