The main principles of passive devices based on graphene and carbon films in microwave - THz frequency range

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Abstract. The ability of thin conductive films, including graphene, pyrolytic carbon (PyC), graphitic PyC (GrPyC), graphene with graphitic islands (GrI), glassy carbon (GC) and sandwich structures made of all these materials separated by polymer slabs to absorb electromagnetic radiation in microwave-THz frequency range is discussed. The main physical principles making a basis for high absorption ability of these heterostructures are explained both in the language of electromagnetic theory and using representation of equivalent electrical circuits. The ideas of using carbonaceous thin films as the main working elements of passive radiofrequency (RF) devices, such as shields, filters, polarizers, collimators, are proposed theoretically and proved experimentally. The important advantage of PyC, GrI, GrPyC, GC is that, in contrast to graphene, they can be either easily deposited onto a dielectric substrate or are strong enough to allow their transfer from the catalytic substrate without a shuttle polymer layer. This opens a new avenue towards the development of a scalable protocol for cost-efficient production of ultra-light electromagnetic shields that can be transferred to commercial applications. A robust design via Finite Element Method and Design of Experiment for RF devices based on carbon/graphene films and sandwiches is also discussed in the context of virtual prototyping.

Keywords: graphene, electromagnetic shielding, thin film, absorption, filter, robust design.

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1 Introduction

Electromagnetic (EM) properties of graphene and its ability to absorb light and EM radiation of longer wavelengths is very dependent on the frequency range of EM wave, more precisely on what is the dominant contribution to graphene ac conductivity, either inter- or intra-band transitions [1,2]. It is already well-known that one monolayer of graphite is absorptive in optics, providing $\pi\alpha = 2.3\%$ absorption of light of visible spectra, which comes from its conductivity originated by inter-band transitions. When one switches to the lower frequencies, THz-subTHz
ranges, the inter-band transitions could lead to a much higher absorbance, at the level of 15-20% per plane, depending on the graphene conductivity caused in its turn by the doping level [3,4].

Graphene, being light, flexible, shatter-proof is very attractive for conventional electronic nano-devices as well as for being used in electronic components in post-silicon age. Its ability to absorb large amount of RF radiation might be used for the design of different passive devices. The purpose of the present paper is to demonstrate some of these possibilities while underlying them with the main physical principles of such devices operation on the basis of graphene.

One more important goal is to introduce other conductive carbonaceous films that could be produced or transferred more easily than CVD graphene. These films could swap graphene in the same applications as building blocks of passive RF devices.

2 Physical background

2.1 Main principles

In the following, the words "thin conducting layer" will refer to carbon-based thin film. It can be pyrolytic carbon a few nm thick. Ultimately, it can also be an individual graphene plane or several graphene planes. In the later case, the conductance scales linearly with the number $N$ of graphene planes when no crystallographic correlation is assumed between the successive atomic lattices. This property is realized in a random stacking of CVD graphene planes [5] and in graphene/PMMA multilayers [3]. In these multilayers, the PMMA spacers used for the transfer of graphene are 0.5 μm thick or less. They are so thin at the wavelength scale that play no role in the electromagnetic properties of the system and can safely be forgotten for frequencies up to the THz range.
When a thin – at the scale of the skin depth – conducting layer is located at the interface between two dielectric media, the component of the electric field, which is parallel to the interface induces a current density (A/m) \( J = \sigma E \), where \( \sigma \) is the conductance (sheet conductivity) of the layer. The current density, in its turn, is responsible for a discontinuity of the parallel component of the magnetic field according to the equation \( \mathbf{n} \times (\mathbf{H}^+ - \mathbf{H}^-) = \sigma E \) where \( \mathbf{n} \) is the local normal unit vector. By contrast, the normal component of the electric field is continuous across the interface.

From there on, the Fresnel formula for the reflectance \( R \), the transmittance \( T \), and the absorptance \( A \) of a plane wave incident on the interface can readily be derived for the geometry illustrated in Fig. 1(a) [6]. The results for the transverse electric (s) polarization are

\[
R_s = \frac{|n_I c \frac{\theta_I - n_T c}{\theta_I + n_T c} \frac{\theta_I - \sigma/\varepsilon c}{\theta_I + \sigma/\varepsilon c}|^2}, \quad T_s = \frac{2n_I n_T c \frac{\theta_I \text{Re}[\varepsilon c]}{\theta_I + n_T c} \frac{\theta_I + \sigma/\varepsilon c}{\theta_I + \sigma/\varepsilon c}}{|n_I c \frac{\theta_I - n_T c}{\theta_I + n_T c} \frac{\theta_I + \sigma/\varepsilon c}{\theta_I + \sigma/\varepsilon c}|^2}, \quad A_s = \frac{4n_I c \frac{\theta_I \text{Re}[\varepsilon c]}{\theta_I + n_T c} \frac{\theta_I + \sigma/\varepsilon c}{\theta_I + \sigma/\varepsilon c}}{|n_I c \frac{\theta_I - n_T c}{\theta_I + n_T c} \frac{\theta_I + \sigma/\varepsilon c}{\theta_I + \sigma/\varepsilon c}|^2}
\]

where \( c \frac{\theta_I}{\theta_T} = \sqrt{n_I^2 - n_T^2} \frac{\sigma}{\theta_T} \frac{\theta_T}{\theta_I} \) can be an imaginary number when the incidence angle \( \theta_I \) exceeds the critical angle. The formulas for the transverse magnetic (p) polarization follow from the above by the substitution of \( 1/cos \) for all the cosine functions. When the sample is placed inside a waveguide and illuminated by a pure guided wave, all the cosine functions must be set to unity (incidence at angle \( \theta_I \) with \( c = \sqrt{1 - (\omega/c_0)^2} \)) and the indices of refraction must be reduced by the factor of \( \sqrt{1 - (\omega/c_0)^2} \), where \( c_0 \) is the cut-off frequency [4]. In free space and normal incidence, the absorptance reaches a maximum value \( n_I/(n_I + n_T) \), when \( \sigma \) is real and \( \sigma/c_0 = n_I + n_T [7] \).

If the thin conducting layer is composed of \( N \) non-interacting graphene planes, the conductance is proportional to \( N \). Accordingly, an optimum value of \( N \) exists for which the absorptance is
maximum. For GHz frequencies, this property is best demonstrated in a waveguide. The condition for maximum absorption writes $\sigma/\varepsilon_0c = 2\sqrt{1 - (\omega_c/\omega)^2}$ for an air filled waveguide. The optimum number of graphene planes is therefore smaller than it would be in free space ($\omega_c \rightarrow 0$) due to the square root factor. In the experiment of Ref. [3], this factor represents 0.72 at 30 GHz for the TE$_{10}$ mode of the rectangular waveguide used.

In free space, assuming $\sigma$ real, the small-angle variation of the absorptance is given by

$$A_{s/p} = A_c \left(1 \pm K \frac{\theta}{2}\right) \quad \text{with} \quad A_0 = \frac{4n_0\sigma/\varepsilon_0c}{(n_1 + n_2\sigma/\varepsilon_0c)^2} \quad \text{and} \quad K = \frac{(n_1 + n_2)(\varepsilon_1 - n_1) - n_2\sigma/\varepsilon_0c}{n_1(n_1 + n_2\sigma/\varepsilon_0c)}$$

(2)

for $s$ (+ sign) and $p$ (- sign) polarization. $A_s$ and $A_p$ behave diametrically opposite with increasing incidence angle $\theta$, raising or decreasing depending on whether the conductance $\sigma$ is smaller or larger than $(n_1 + n_2)(2n_1/n_2 - 1)\varepsilon_0c$. For small incidence angles, the average absorptance $(A_s + A_p)/2$ (non polarized radiation) is constant up to fourth order in $\theta$.

Fig. 1. (a) A plane wave is incident on a thin conducting layer (central green line) located at the interface between two semi-infinite dielectric media with index of refraction $n_i$ (incidence medium) and $n_T$ (transmission medium). (b) Same as in (a) when the thin conducting sheet is sandwiched by two dielectric slabs of finite thickness $d_1$ and $d_2$, and dielectric constant $\varepsilon_1$ and $\varepsilon_2$. In both cases, the angles $\theta_i$ and $\theta_T$ are related by the Snell-Descartes law $n_i \sin \theta_i = n_T \sin \theta_T$.

Fig. 2 illustrates the electromagnetic characteristics of a thin conducting layer of conductance $\sigma = 0.7\varepsilon_0c$ computed with the above formulas in free air. Such a layer could be realized by stacking...
two CVD graphene planes on their PMMA transfer films stretched on a frame. The absorptance of the s-polarized wave (solid curve in Fig. 2a) increases with increasing the incidence angle and reaches its maximum value of 0.5 at the angle \( \theta_I = \arccos(\sigma/2\epsilon_0c) = 69.5^\circ \). By contrast, the absorptance of the p-polarized wave (dashed curve in Fig. 2a) decreases monotonously vs \( \theta_I \). The average of the s and p absorptance (dot-dashed curve in Fig. 2a) stays roughly constant between 0 and 60\(^\circ\). The p reflectance remains small for all incidences and almost vanishes above 70\(^\circ\) (dashed line curve in Fig. 2c). The p transmittance increases monotonously vs \( \theta_I \) (dashed line curve in Fig. 2d). The reflectance and transmittance for the s polarization behave diametrically. As schematized in Fig. 2b, a thin conducting layer of the kind discussed here becomes a polarizer at grazing incidence [8]: almost 100\% of the reflected radiation is s polarized and a big fraction of the transmitted wave is p polarized.

![Graph showing absorptance, reflectance, and transmittance](image)

Fig. 2. Absorptance (a), reflectance (c) and transmittance (d) vs incidence angle computed for a thin conducting layer with conductance \( \sigma = 0.7\epsilon_0c \) at 30 GHz and suspended in air. The drawing in (b) illustrates how such a conducting layer (thick green line) may behave as a polarizer at grazing incidence. The blue full lines and red dashed lines correspond to the s and p polarizations, respectively. The black dot-dashed lines correspond to non-polarized radiations.

2.2 Equivalent circuit approach

For most applications, graphene has to be supported by a substrate, which thickness can not be neglected as compared with the wavelength of interest. The system graphene plus substrate can
be oriented with the thin conducting layer facing either the incidence medium or the emergence medium. One may, therefore, consider exploring the properties of a heterostructure composed of a thin conducting layer located between two dielectric slabs as illustrated in Fig. 1(b).

From the electromagnetic point of view, this system is equivalent to an electrical circuit [9]. The equivalent circuit for the $s$ polarization is shown in Fig. 3(a). The generator accounts for the power that is required to maintain the electromagnetic radiations in a stationary state in spite of the dissipation by Joule effect in the thin conducting layer. For reasons detailed in the Introduction, it is desirable to maximize the absorption over the reflection in electromagnetic shielding layers, very much like to radar absorbing media. One needs therefore to design the heterostructure of Fig. 1(b) in such a way as to dissipate the largest possible power in the thin conducting layer. According to Norton’s theorem, the electrical circuit of Fig. 3(a) is equivalent to the simplified version shown in Fig. 3(b) where $Y_N$ is the total admittance of the real circuit seen by the load, here the thin conducting layer. $I_N$ and $Y_N$ are independent on the value of $\sigma$. As a result, the power absorbed by the load is maximum when its conductance matches the complex conjugate $Y_N^*$ of the external admittance. The simple equation $Y_N^* = \sigma$ allows one to optimize the heterostructure of Fig. 1(b) from the point of view of power absorption for the $s$ polarization.

For the circuit of Fig. 3(a),

$$Y_N = Y_1 + Y_2 = \epsilon_\epsilon c \left( \alpha_1 - \frac{\beta_1^2}{\alpha_1 + \eta c} \epsilon_j + \alpha_2 - \frac{\beta_2^2}{\alpha_2 + \eta c} \epsilon_j \right)$$

$$\alpha_j = \frac{\sqrt{n_j^s \tilde{\varepsilon}_j - \tilde{\varepsilon}_j}}{\epsilon_j \left( \mathcal{E}_j - n_j^s \tilde{\varepsilon}_j \mathcal{K}_j \right)}$$ and $\beta_j = \frac{\sqrt{n_j^s \tilde{\varepsilon}_j - \tilde{\varepsilon}_j}}{s \left( \mathcal{E}_j - n_j^s \tilde{\varepsilon}_j \mathcal{K}_j \right)}, j = 1, 2$ (3)

where $k_0 = \omega/c$. The non-standard formalism used for the elements of the circuit of Fig. 3(a) has the advantage to yield an easy generalization of the expression of $Y_1$ and $Y_2$ in the form of a continued fraction when the system has an arbitrary number of dielectric layers [10]. In normal
incidence, (eq. 3) can be used irrespective of the polarization. The case where there is just single
dielectric layer, oriented upwards or downwards with respect to the incident radiations, is now
analyzed under the explicit assumption that both $\epsilon_1$ and $\epsilon_2$ are real.

Fig. 3. (a) Electrical circuit equivalent to the heterostructure of Fig. 1(a) as concerns its
electromagnetic properties for an $s$-polarized incident wave. The expressions written along the
circuit elements are their admittances (see text for the meaning of the symbols). The conductance in
the middle of the diagram – the load – represents the thin conducting layer in the center of the
heterostructure. (b) Further simplification of the circuit (a) as seen by the load according to
Norton’s theorem.

2.2.a Graphene on a substrate facing the incidence medium.
This case corresponds to $d_2 = 0$ in Fig. 1(b). At normal incidence, the expression (eq. 3) of $Y_N$
simplifies in

$$Y_N = \epsilon_0 c \left( \frac{n \epsilon_1 + \epsilon_1 - n_1^2 S}{\epsilon_1 c} \frac{d_1 c}{\phi_1} + n_t \right) \quad \text{with} \quad \phi_1 = \sqrt{\epsilon_1 k_0 d_1}. \quad (4)$$

For real $\sigma$, the admittance matching condition requires $Y_N$ to be real, which is realized when
either $\sin \phi_1 = 0$ (half-wavelength blade) or $\cos \phi = 0$ (quarter-wavelength blade). The first case
is equivalent to the geometry of Fig. 1(a) with its maximum absorptance condition $\sigma/\epsilon_0 c = n_t +
n_T$. The second case demands $\alpha/\epsilon_0 c = \epsilon_1 n_T + n_T$ for optimum absorption and leads to an
absorptance larger than in the first case, provided $\epsilon_1 > n_T^2$. The optimum value of $A$ obtained after
additional calculations is $A_{opt} = \epsilon_1/(\epsilon_1+n_I n_T)$. An illustration of this effect is described in Section 4.

2.2.b Graphene on a substrate facing the emergence medium.

This case corresponds to $d_1 = 0$ in Fig. 1(b). The expression of $Y_N$ is the same as in (eq. 4) after the substitution of the index 2 for the index 1 and $n_I$ for $n_T$. Still for a real $\sigma$, the condition $\sin \phi_2 = 0$ is equivalent to the geometry of Fig. 1(a). The condition $\cos \phi_2 = 0$ is more interesting and leads to the condition of maximum absorption $\sigma/\epsilon_0 c = n_I + \epsilon_2/n_T$, with an absorptance larger than the first case provided $\epsilon_2 < n_T^2$. Here, the optimum value of the absorptance is $A_{opt} = n_I n_T/(\epsilon_2+n_I n_T)$.

It can approach 100% when $n_T \to \infty$. The Salisbury screen invented in 1952 pertains to this geometry [11]. In this device, the emergence medium is a metal ($n_T \to \infty$) thicker than its skin depth, separated from the thin conducting layer by a non-absorbing dielectric slab of thickness $d_2$. The absorptance is maximum for frequencies such that the effective wavelength in the dielectric slab is a multiple of $4d_2$ ($\cos \phi_2 = 0$, or more precisely $\text{Im} Y_N = -\text{Im} \sigma$ when $\sigma$ is complex).

3 Methodology

3.1 Synthesis of carbonaceous materials

Graphene, PyC, GrPyC and GrI are all fabricated by chemical vapor deposition at ~1000 °C temperature regime using methane precursor. Graphene, GrPyC and GrI were grown on a copper foil whereas PyC was grown directly on a silica substrate. Typically, a large area monolayer graphene is grown on a copper substrate [12]. However, copper can be used as a substrate for thicker graphitic film synthesis as well. Annealing the copper substrate or increasing the amount of methane increase the carbon film thickness. This will lead to synthesis of GrPyC and GrI films. Without catalyst metal (i.e. the process on a silica substrate), an amorphous carbon film is
produced with dominating sp2 hybridization [13]. This will lead to ultra-thin and uniform PyC film formation [14].

The experimental receipts for graphene, PyC, GrPyC and GrI are similar to each other. All materials demonstrated here are fabricated in conventional hot wall CVD by using methane precursor. Pyrolytic carbon was grown on a silica substrate at dynamic temperature range starting at 700 C. First the CVD chamber was heated to 700 C in hydrogen atmosphere. Then the temperature was increased in CH4 atmosphere (static 25 mBar) to 1100 C and after 5 minutes the CVD chamber was cooled down to 700 C and the methane atmosphere was replaced with hydrogen (for more details see Ref.[15]). Graphene was grown on a copper foil (99.8 % pure) at 950 C temperature. First the copper foil was heated and annealed (20 min) in H2 atmosphere (5 sccm, 0.5mBar). The graphene film was grown in 950 C using 5sccm H2 + 5 sccm CH4 flow for 20 minutes. GrPyC was grown on copper foil (99.8 % pure) in 1000 C temperature in static CH4 atmosphere (25 mBar) in 30 minutes (see Ref.[16] for details). This process produced graphitic carbon film about 8 nm thick. GrI sample was also grown on a copper foil (99.8 % pure). The copper substrate was first annealed in 1000 C temperature in H2 atmosphere (5 sccm, 0.5 mBar) for one hour. After this the graphitization was done in static atmosphere using methane and hydrogen gas mix (with 1:1 ratio in 8 mBar pressure) for 20 minutes. All the samples were cooled down to room temperature in static H2 atmosphere, ~5 mBar (overnight).

The growth of PyC on a dielectric substrate does not require a metallic catalyst. In about 1000 C temperature the spontaneous methane decomposition leads to C2 and eventually aromatic C6 hydrocarbon species formation. These molecules land on a dielectric surface and evolve forming nanosize graphitic flakes that intervene and form a continuous film. Although the PyC film is continuous and dominated by sp2 hybridized carbon, the film is very amorphous. Therefore,
many properties like electrical conductivity and nonlinear optical effects are suppressed in comparison to graphitic carbon films [15, 17].

Low carbon solubility in copper is responsible for graphitic monolayer growth on the surface of a copper substrate in CVD process. This technique is widely explored and is recognized as one of the most promising method for large area graphene synthesis. The process is based on surface catalysis. The methane molecule is decomposed at the surface of a copper substrate and, due the low carbon solubility of copper; the carbon atom stays at the surface of the copper substrate. Eventually, the surface of copper is covered by sp2 hybridized monolayer of carbons. Once the copper surface is coated with graphene, the catalytic growth of graphite is almost prevented. However, increasing the amount of methane during the process will cause template graphitic film growth on top of the monolayer graphene resulting a thin graphitic film.

![Graphene and PyC films](image)

**Fig. 4.** Graphene (a), GrI (b) and GrPyC (c) transferred on oxidized silicon substrate. (d) A PyC film deposited on a silicon substrate.

Moreover, if the copper substrate is annealed for one hour, we observed graphitic islands grown on a monolayer graphene. These graphitic islands appeared as either round areas or stripes (see Fig. 4). The process temperature of 1000 C is below melting point of copper (1084 C) but is
enough for surface melting. Prolonged annealing time may increase the depth of melted copper slightly increasing carbon solubility in copper resulting multilayered graphitic areas.

A GC was fabricated by spin coating carbon based photoresist (nLOF-AZ2070 diluted with AZ EBR solvent with 1:4 ratio) on a substrate. After the substrate is coated by a resist layer, it is baked on a hot plate (110 °C/ 1 min) and then pyrolyzed in a CVD system. The thickness ratio of the original photoresist film and the GC is 1:10, i.e. the thickness of a 300 nm thick photoresist film led to a 30 (± 3) nm thick GC after the pyrolysis. The thickness of the GC film can be vary by changing the thickness of the photoresist film [18,19].
3.2 Carbon film transfer and characterization

Graphene, GrPyC and GrI grow on a Cu substrate and need to be transferred for further use. GrPyC is mechanically much more robust than graphene, and therefore survives the transfer process from a Cu substrate without a template polymer layer [16] typically used in graphene transfer process to protect and hold graphene.

Transfer process of graphene and GrI is done by using Poly(methyl methacrylate) (PMMA) as a support. First the PMMA layer is spin coated on a graphitic film/copper substrate and baked in about 60 C for 10 minutes. After baking, the backside carbon of the sample is removed by oxygen plasma etching (20 sccm/100 W/2 min) and the copper substrate foil is then wet etched in ferric chloride. The PMMA/graphitic film is next rinsed in water two times to remove FeCl3 remains and deposited then on a dielectric substrate. Remaining water is evaporated by baking the sample on a hot plate in mild temperature.

When all water is dried from the sample the PMMA support is removed by acetone bath (overnight) and then rinsed in isopropanol and water. This procedure will remove most of the PMMA but sometimes leave remains that can be seen in Fig 4.

The PyC film is deposited directly on a dielectric substrate and is usually not needed to be transferred. However, should the film be lifted out from the substrate, it is noteworthy that the adhesion between the PyC film and its silica substrate is rather weak. Depositing PMMA on a substrate and then gently placing the sample in water sometimes lifts the PyC/PMMA film from the substrate. From the water surface, the PyC/PMMA film can be deposited on an arbitrary substrate and the PMMA can be removed similarly as for graphene transfer.

After the carbon films are transferred on the desired substrate, it is important to characterize them. In our experiments, we used Raman spectroscopy, which is well-known technique to
identify the carbon allotropes. Moreover, optical transmission spectroscopy was used to provide some understanding of the linear optical properties of the fabricated films.

![Graph showing Raman and transmittance spectra](image)

Fig. 5. (a) Raman, and (b) transmittance spectrums of graphene, GrI, GrPyC and PyC.

A Raman spectrum of a graphitic carbon material is governed by three main peaks. Those are D (located ~1350 cm\(^{-1}\)), G (located ~1582 cm\(^{-1}\)) and 2D (located ~2700 cm\(^{-1}\)) peaks. Generally, the D peak is a breathing mode, activated by a disordering in the graphite lattice while the longitudinal mode G peak is a trace of a graphitic sp\(^2\) hybridization of carbon atoms. The 2D peak is a second harmonic of D peak but does not require disorder.

Raman spectra of the carbon films were measured by a Via Raman microscope with 514 nm excitation wavelength. Fig. 5 shows Raman spectra of graphene, GrPyC, GrI and PyC. As seen, graphene and GrI are almost comparable with their small D peak but strong and rather narrow G and 2D peaks. The small D peak is expected to originate from the bi-/multi-layer islands in graphene and GrI. In GrPyC, the D peak is significantly stronger in comparison to graphene and GrI because the multilayer areas are grown by template manner without catalyst and this increases the amount of disorder in the material. From the Fig. 5 (a) it can be seen that the PyC film is already a very amorphous material with wide D and G peaks and has negligible 2D peak. Therefore, it can be summarized that graphene, GrPyC and GrI are rather crystalline materials,
while PyC is amorphous graphitic carbon. A careful analysis reveals that there are practically no differences in the Raman spectrum of the PyC and GC [19].

The transmittance spectra of the carbon films were measured by a Perkin Elmer lambda-18 spectrometer in a wavelength range from 250 nm to 750 nm. They are shown in Fig. 5. As it can be seen, all of the films have almost constant transmittance from 500 nm to 800 nm and a transmittance dips at 260 nm. As it is shown in Raman spectra of graphene, GrPyC and GrI possess higher crystallinity in comparison to PyC. The absorption of the GC is about 10 % higher in comparison with PyC of the same thickness [19]. Therefore, the drop in the transmittance for GC and PyC is wider in comparison to other films.

3.3 Sandwich structures fabrication

The sandwich structure of the multilayered graphene/graphitic film, described theoretically in chapter 2 is fabricated by using the PMMA layer as a dielectric spacer between the carbon films. The sample fabrication process follows the transfer process to the point where PMMA/graphene film is deposited on a substrate. When PMMA/graphene sample is on a substrate, instead of removing PMMA in acetone, another PMMA/graphene film is deposited on top of the first layer. By this technique multilayered sandwich structures can be easily stacked with a desired number of layers (see Fig. 6).

![Image](image.png)

Fig. 6. PMMA/graphitic film (Gr-film) sandwich structure. (a) First, (b) second and (c) third layer of PMMA/Gr-film are stacked layer by layer.
Sandwich could be preferable from different points of view. From one side, PMMA is always used for graphene transfer and the procedure of removing PMMA could make the device production more difficult, time and cost consuming. For microwave applications, we do not need to remove PMMA, because: (i) we want to have graphene or other conductive carbon layers decoupled electromagnetically, in order to have the conductance of the overall sandwich structure equal to the sheet conductivity of each layer multiplied by the number of layers; (ii) the structure remains flexible; (iii) the thickness of the PMMA films used, between 100 nm – 1 micron, is thin enough in order not to contribute to the EM response of the sandwich (100 nm – 1 micron of PMMA is transparent for microwave and THz radiation.

3.4 Electromagnetic measurements
The EM interference (EMI) shielding ability as ratio of transmitted/input, $S_{21}$, and reflectance as ratio of reflected/input, $S_{11}$, signals were measured at 26–37 GHz by a scalar network analyzer R2-408R (ELMIKA, Vilnius, Lithuania) at room temperature and normal pressure. The measurement errors is $\delta|S_{21}| = \pm (0.6 + 0.06|S_{21}|)$. Reflectance ($R$), transmittance ($T$), and absorbance ($A$) are obtained from the measured S-parameters as $R = S_{11}^2$, $T = S_{21}^2$, $A = 1 - R - T$. THz measurements (200 GHz – 3 THz) were carried out using time-domain spectrometer T-SPEC Ekspla, Vilnius, Lithuania.

4 Experimental data, microwave range. Electromagnetic interference shielding layer

4.1 Experimental data and comparative analysis.

Figure 7 presents the experimentally measured absorption coefficient of multilayered structure depending on the number of layers and the thickness of the dielectric substrate in such a system.
Fig. 7. Absorption ( ) vs number of graphene layers in sandwich structure and vs the thickness of the dielectric substrate in case radiation comes from the substrate side. Data collected for the frequency 30 GHz.

By selecting the optimum substrate thicknesses and number of layers (therefore the overall conductance), it becomes possible to rise the absorption in the microwave range at 80-95% levels. In this case, as follows from the theoretical description, one of the central parameters defining the electromagnetic properties of the sandwich system is the conductance of the original carbon film, which in its turn is influenced by many factors.

The dependence of the scattering matrix elements of structures consisting of one carbonaceous layers is shown in Fig. 8.
Analyzing the results collected in Figure 8, one may conclude that graphene is the best candidate for EMI shielding in case optical transparency is also needed for instance for electromagnetic interference (EMI) shielding of windows of optoelectronic devices. Moreover, the contribution of reflection ($S_{11}$) in graphene/PMMA sandwich consisting of one graphene monolayer in microwave range is the smallest in comparison with all other carbonaceous films (46% vs 52% for GrI, which is closest to graphene regards to optical density).

At the same time, while not as transparent as graphene is, thin PyC films (5 nm thick), GrI and GrPyC (8-12 nm thick) could be interesting alternative to graphene, as they are still transparent enough to protect optoelectronics devices and provide better (18-20% vs 15% for one layer structure) shielding ability in microwaves.

Thicker PyC and GC, being 25-30 nm thick, absorb already large amount of visible light, close to 50%, and might be used for other tasks where optical transparency is not a must (such as EMC tasks for electronic devices working in THz and sub-THz frequencies).
Although PyC and GC being 25-30 nm thick are not transparent in visible range, there is an important advantage of using them as EMI shield for high-frequency nanoelectronics: they both can be deposited onto the top of dielectric substrate directly (any shape and size), and therefore can be used not as a part of sandwich structure, but as they are.

**4.2 EMI shield for high frequency electronics**

Design of compact effective shielding layers of microwave radiation is a very important goal for many practical applications. For development of 5G communication systems, it is necessary to solve many problems, related to EM compatibility (EMC) and propose materials perfectly absorbing EM radiation at high frequencies, up to THz range. The subject of this subsection is to show at which thicknesses (number of layers in case of sandwich structures) one may expect high enough EMI shielding efficiency (SE) coursed by absorption mostly.

Table 1 presents the optimal thickness of carbonaceous films corresponding to EMI shielding efficiency $S_{21}$ at the level of 8-10 dB for guided waves, 30GHz. In all cases, carbon thin film was put on the top of 1.4 mm thick silica substrate (which permittivity is 3.83) and the radiations come from the substrate side. The calculations were made for the waveguide experiment realization. If the substrate has smaller permittivity, the optimal $l_{opt}$ carbonaceous film thickness corresponding to minimal reflection and maximal absorption at normal incidence will decrease as $\varepsilon = l + \sigma l_{opt}/(2\varepsilon_0 c)$ [20].

For EM attenuation given in Table 1 the absorption value is about 0.84-0.86, whereas $R = 0.02-0.03$ and $T = 0.11-0.013$. In free space, the values of EMI SE will be somewhat higher (11-13 dB).
Table 1. EMI shielding efficiency $S_{21}$ at 30 GHz of the carbonaceous films and sandwich structures. $N_{\text{max}}$ corresponds to number of carbonaceous layers related to maximal shielding efficiency (see Fig.9 for calculations with particular conductivities corresponding to our collection of carbonaceous films).

| Material  | Thickness, nm | $N_{\text{max}}$ | $SE_{15\text{ layers}}, \text{ dB}$ |
|-----------|---------------|------------------|-------------------------------------|
| Gr        | atomic layer  | 19               | 8.3                                 |
| GrI       | 5 nm          | 15               | 9.3                                 |
| GrPyC     | 8 nm          | 14               | 9.5                                 |
| GC        | 30 nm         | 15               | 9.3                                 |
| PyC (5 nm)| 5 nm          | 40               | 5.4                                 |
| PyC (30 nm)| 30 nm       | 9                | 11.9                                |

Fig.9. Example of calculation of optimal number of layers in sandwich structure for particular conductivities of carbonaceous films ($0.28\varepsilon_0\sigma_c$, graphene in this particular case) corresponding to minimal reflection (when radiation comes from the side of 1.44 mm silica substrate) and maximal absorption (>85%).
5 Experimental data, THz range. Passive devices

5.1 Experimental data and comparative analysis

Fig. 10. (a) The transmittance of Graphene and GrI films on the top of quartz substrate (0.5 mm thick) in THz range. The points denote the experimental data. (b) The same for PyC of different thicknesses.

Using the method of conductivity calculations from the measured electromagnetic response of the samples in the THz frequency range (details of the calculation are presented in [4]), it is easy to show that graphene, GrI and GrPyC are characterized by the following conductivity values: $0.21 \varepsilon_0c$, $0.36 \varepsilon_0c$ and $0.37 \varepsilon_0c$. 
Theoretically calculated dependences of absorption and transmission of s and p polarized wave in free-standing thin carbonaceous film vs the angle of incidence and conductivity are shown in Figure 11.

Fig.11. The dependence of the transmission (a) and absorption (b) and (c) of thin carbonaceous film on the incidence angle and film conductivity (in $\varepsilon_0\varepsilon_0$ units).

Analyzing the experimental data obtained in the context of theoretical estimates, we can conclude that the increase of the overall conductance of the thin carbon film or sandwich leads to sharper absorptance angle dependence for s-polarized wave (see also [4]), which can be used in the development of tunable THz passive device.
In this regard, the use of graphene is more favorable in comparison with other types of investigated carbon materials. However, it should be noted that the effects associated with selective transmission of differently polarized waves on the angle of incidence are more pronounced for the films with higher conductivity (therefore for mass production of THz modulator or filter either GC or PyC directly deposited on the top of quartz substrate, or GrPyC which does not need PMMA, could be more appropriate).

5.2. Filter, polarizer, modulator, collimator

The different reaction of thin conductive film, including graphene, PyC, GrPyC, GC to react differently to the polarized radiation can be used for design and fabrication different passive devices for THz applications. As it was discussed in section 2.1 for s-polarized radiation the optimal thickness (number of layer) decreases dramatically with the increase of incidence angle, which means suppressing transmittance ability of thin carbon film for s-polarized waves at almost sliding incidence. In contrast, for p-polarized radiations, the thickness of carbon layer required for maximal absorption increases together with increasing incidence angle, that is carbonaceous film/sandwich becomes transparent for EM radiation. This effect can be easily used for producing filters, modulators, collimators and polarizers for THz radiation. The schematic representation of such devices is presented in Figure 12.
6 EMI shield Robust Design approach: the preliminary phase

With the aim of transferring from lab to industrial scale the application of sandwich structures made of carbon-based thin conductive films separated by polymer slabs as electromagnetic shield, a virtual prototyping approach is considered on the schematic model reported in Figure 13 [21, 22].
In this context, the design by means of computer simulations (computer aided design) leads to maximize the production yield, especially if the tolerances and uncertainties affecting the design parameters are taken into account [23]. This approach is particularly useful for the study and design of innovative device/material where the involved physical mechanisms are still not clear or the developed technology is not well established [24, 25]. By considering a particular system’s performance represented by a function \( f(\chi) \) depending on \( n \) design parameters including “controllable and not controllable” factors \( \chi = x_1, x_2, ..., x_n \), a design that satisfies the customer constraint also in presence of factors’ variations \( \Delta = [\Delta_1, \Delta_2, ..., \Delta_n] \) is named Robust Design [26]. Robust Design requires a preliminary phase in order to define if and how much each factor affects the system’s performance. At the same time, this phase allows to identify an analytic expression of the function \( f(\chi) \) describing the performance in case the problem cannot be expressed in closed form (for example, by using a polynomial best fit approximation) [27]. By starting from a system model and adopting a Design of Experiment (DoE) approach on the simulated data, the use of Dex Scatter Plot (DSP) allows to carry out a screening on the design parameters. The main factor and the type and trend of the dependence with respect to each one of the \( n \) parameters can be highlighted by looking at the slope of the interpolating curve [28]. The use of Response Surface Methodology (RSM) can then be considered in order to obtain the desired analytic function \( f(\chi) \) only on the factors that have been found to have a significant influence [29]. This function can be used as the performance function to carry out an optimization procedure such as the Robust Design [30]. Such a simulation based optimization method, adopted also for the system considered in this paper is described schematically in Figure 14.
6.1 Statement of the problem

For the multilayer shield the considered system’s performance is the shielding efficiency SE computed by means of a system’s model shown in Figure 13. A FEM based commercial software (Comsol Multiphysics® RF Tool) has been adopted for the required simulations.

By considering the geometry and the domains reported in Figure 15, Planar-Wave condition has been applied at “Port 1” (Electric Field: E₁); “Port 2” (Electric Field: E₂) is the “Output Port”. Perfect Matched Layer (PML) represents “matched loads condition”, necessary to avoid possible reflected incident waves causing simulation errors. The structure is infinitely extended along y-
direction: in Figure 15 “Periodic Condition” on upper and lower boundaries are fixed and “field continuity condition” along y-direction is applied. Over the whole structure the following equation was implemented

\[ \nabla \times \nabla \times \mathbf{E} - \mu_r k_o^2 \left( \varepsilon_r - j \frac{\sigma}{\omega \varepsilon_0} \right) \mathbf{E} = 0 \]

where \( k_o \) is the propagation vector amplitude in vacuum (empty space), \( \varepsilon_r \) is the relative electric permittivity of each material, \( \sigma \) is the electrical conductivity, \( \mu_r \) is the relative magnetic permeability, \( \omega \) is the frequency of the incident wave and \( \mathbf{E} \) is the Electric Field. The post processing extraction of the scattering parameters leads to compute the \( \text{SE} = - S_{21}^2 \) for the considered configuration.

6.2 Design parameters

Three design parameters or factors are considered: the number of replicated “cells” in the structure, as illustrated in Figure 13 (3 in the considered case); \( \sigma \) is the electrical sheet conductivity of the carbonaceous layer (since it is a new-generation material its fabrication intrinsically may suffer from large uncertainties); \( l_{\text{pmma}} \) is the thickness of the PMMA layer. The factor \( \text{NumArray} \) is a discrete controllable parameter ranging between 1 and 9. In the performed evaluations 3 discrete level (i.e. 1, 5 and 9) have been considered. The factors \( \sigma \) and \( l_{\text{pmma}} \) are continuous and partially controllable parameters for which we have adopted a range between 1/10 and 10 times their nominal values, sampled with a 5 level uniform segmentation. The selection and evaluation of the response variable has been obtained by means of a Matlab\textsuperscript{®} environmental routine developed in order to compute the SE at 30 GHz for a given combination of these three factors by exploiting the FEM model described in the previous section.
6.3 Design of Experiments (DoE)

By considering a full factorial approach on the design parameter defined in the previous section, a Matlab® procedure able to represent DoE Scatter Plot after computing of 3x5x5=75 total design was developed. The achieved results are shown in Figure 16.

From the simulated data reported as blue circles in Figure 16 it is possible to observe that the PMMA-thickness effect becomes relevant only for higher number of replicated cells. In fact, for NumArray = 1 (one cell) the SE remains constant with respect to thickness variation whereas a weak dependence is detected only for NumArray=9. Moreover, SE exhibits a quadratic-type dependence both on the conductivity of the thin layer and on the PMMA thickness (dashed lines in Figure X4). In Figure 17 the same simulated data are used to derive the main effect of each factor.
In particular, in Figure 17 the slope of the red-continuous line furnishes the so called “main effect” if a normalization of the factors with respect to their nominal value is considered (i.e. $x_1 = \frac{\text{NumArray}}{\text{NumArray}_{\text{nom}}}$, $x_2 = \frac{\sigma}{\sigma_{\text{nom}}}$, $x_3 = \frac{l_{\text{pmma}}}{l_{\text{pmma}_{\text{nom}}}}$). The number of cells is the most influencing parameter (main factor $= 31.67$) and the PMMA thickness is the less one (main factor $= 3.74$). All the computed main factors are positive, showing that SE increases by increasing each considered parameter.

### 6.4 Response parameters and RSM

According to the dependences found in the previous phase, the RSM Tool function of Matlab is used to calculate the coefficients of the second order approximating function $\text{SE}$ for a fixed value of $\text{NumArray}$:

$$\text{SE}_{\text{NumArray}}(\sigma, l_{\text{p}}) = \gamma_0 + \gamma_1 \sigma + \gamma_2 l_{\text{p}} + \gamma_3 \sigma^2 + \gamma_4 l_{\text{p}}^2 + \gamma_5 \sigma l_{\text{p}}$$
In Table 2 the gamma coefficients of the interpolating functions are reported useful to derive the SE in non-simulated design or to apply optimization procedure considered in the schematic layout of Figure 14.

Table 2. Parameters of robust design

| NumArray | $\gamma_0$ [dB] | $\gamma_1$ [dB m/S] | $\gamma_2$ [dB/m] | $\gamma_3$ [dB m$^2$/S$^2$] | $\gamma_4$ [dB/m$^2$] | $\gamma_5$ [dB/S] |
|----------|-----------------|---------------------|------------------|-----------------------------|---------------------|-----------------|
| 1        | 4.23            | 43.76E-6            | 161.53           | -19.32E-12                  | -2.88E6            | -388.26E-6      |
| 5        | 11.62           | 61.26E-6            | 9.95E3           | -28.87E-12                  | -141.27E6          | 281.38E-6       |
| 9        | 13.89           | 73.57E-6            | 121.76E3         | -37.04E-12                  | -962.32E6          | 603.84E-6       |

In particular, the Robust design of the system with the procedure proposed in [25] and based on the interpolating functions here obtained is still in progress and will appear in a forthcoming paper.

7 Conclusion

We discussed the possibility to use various conductive carbon films as building blocks for passive electromagnetic devices, including EMI shielding layer, filters, polarizers, modulators and collimators for microwave and THz radiation. The peculiarities of different carbonaceous films, including the simplicity or difficulty of their synthesis and optical transparency were discussed. We found that for microwave application, any of the investigated carbon films could be used (optimal thickness and number of layers in the sandwich structures were found). In case, when optical transparency is a necessary condition, graphene, GrI and GrPyC are the best candidates, with an advantage for graphene. In case one needs only shielding ability, PyC and GC are good alternative as they can be deposited on the dielectric substrate directly.

The ability of thin conductive film, including graphene, PyC, GrPyC, GC to respond to polarized radiation can be used for the design and fabrication different passive devices for THz
applications. A demonstrator of PyC to be used as the sensitive element in the THz polarizer was presented.

The final conclusion is that, depending on the particular application that is targeted, both graphene and other carbonaceous films (GrPyC, GrI, PyC, GC) could be utilized. In case one need a shield for optoelectronic window, the best option is graphene, GrPyC and GrI (but graphene is the best because of the best transparency in visible range). In case of filters, collimators, modulators, where dielectric substrate should be also used, PyC and GC, which can be deposited directly without the delicate PMMA shuttle transfer, could be better than graphene.

The analysis provided in the present communication of electromagnetic properties in GHz-THz frequency ranges of many of carbon conductive films together with their optical properties and details of synthesis/transfer process opens an avenue towards the development of a scalable protocol for cost-efficient production of ultra-light electromagnetic shields and other RF passive devices on the basis of graphene and nm-thin carbonaceous films.

Finally, in order to support the scalable protocol of a particular device with a Robust Design approach taking in to account the unavoidable parameters’ variability, the preliminary phase of describing the performance of the device (i.e. the multilayer EMI shield) as a function of relevant parameters based on DoE and RSM tool is here reported. In particular it is shown that from the considered design parameter (i.e. the number of layers in the sandwich structures, the conductivity of the adopted carbonaceous film and the thickness of the dielectric spacer), the number of layers is the most relevant factor for the shielding efficiency whereas the thickness of the PMMA is the less one. Moreover, a quadratic equation describing the shielding efficiency for each considered number of layers is reported and will be used in order to optimize the design by
considering the uncertainty on the conductivity of the adopted carbonaceous film and on the
thickness of the dielectric spacer.

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Caption List

Fig. 1. (a) A plane wave is incident on a thin conducting layer (central green line) located at the interface between two semi-infinite dielectric media with index of refraction $n_i$(incidence medium) and $n_T$(transmission medium). (b) Same as in (a) when the thin conducting sheet is sandwiched by two dielectric slabs of finite thickness $d_1$ and $d_2$, and dielectric constant $\varepsilon_1$ and $\varepsilon_2$. In both cases, the angles $\theta_i$ and $\theta_T$ are related by the Snell-Descartes law $n_i\sin\theta_i = n_T\sin\theta_T$.

Fig. 2. Absorptance (a), reflectance (b) and transmittance (c) vs incidence angle computed for a thin conducting layer with conductance $\sigma = 0.7\varepsilon_0 c$ at 30 GHz and suspended in air. The drawing in (b) illustrates how such a conducting layer (thick green line) may behave as a polarizer at grazing incidence. The blue full lines and red dashed lines correspond to the s and p polarizations, respectively. The black dot-dashed lines correspond to non-polarized radiations.

Fig. 3. (a) Electrical circuit equivalent to the heterostructure of Fig. 1(a) as concerns its electromagnetic properties for an s-polarized incident wave. The expressions written along the circuit elements are their admittances (see text for the meaning of the symbols). The conductance in the middle of the diagram –the load-- represents the thin conducting layer in the center of the heterostructure. (b) Further simplification of the circuit (a) as seen by the load according to Norton’s theorem

Fig. 4. Graphene (a), GrI (b) and GrPyC (c) transferred on oxidized silicon substrate. (d) A PyC film deposited on a silicon substrate.
Fig. 5. (a) Raman, and (b) transmittance spectrums of graphene, GrI, GrPyC and PyC.

Fig. 6. PMMA/graphitic film (Gr-film) sandwich structure. (a) First, (b) second and (c) third layer of PMMA/Gr-film are stacked layer by layer.

Fig. 7. Absorption ( ) vs number of graphene layers in sandwich structure and vs the thickness of the dielectric substrate in case radiation comes from the substrate side. Data collected for the frequency 30 GHz.

Fig. 8. The dependence of the scattering matrix elements (S\textsubscript{11} and S\textsubscript{21}) at 30 GHz, and the optical transmittance values of the carbonaceous film type.

Fig. 9. Example of calculation of optimal number of layers in sandwich structure for particular conductivities of carbonaceous films (0.28ε\textsubscript{0}c, graphene in this particular case) corresponding to minimal reflection (when radiation comes from the side of 1.44 mm silica substrate) and maximal absorption (>85%).

Fig. 10. (a) The transmittance of Graphene and GrI films on the top of quartz substrate (0.5 mm thick) in THz range. The points denote the experimental data. (b) The same for PyC of different thicknesses.

Fig. 11. The dependence of the transmission (a) and absorption (c) and (d) of thin carbonaceous film on the incidence angle and film conductivity (in ε\textsubscript{0}c units).

Fig. 12. Schematic representation of polarizer for THz radiation.

Fig. 13. Sketch of the Implemented Structure

Fig. 14. Simulation based optimization scheme

Fig. 15. Simulated geometry and domains of the device in Figure 13.

Fig. 16. DoE Scatter Plot of SE @ f=30GHz for 1, 5 and 9 replicated cells of the shield.

Fig. 17. Main Factor Plot of SE@f=30GHz
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