Research Article

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Effect of granulometric distribution on electromagnetic shielding effectiveness for polymeric composite based on natural graphite

<https://doi.org/10.1515/secm-2019-0037>

Received Aug 27, 2019; accepted Dec 01, 2019

Abstract: In this study, the electromagnetic interference (EMI) shielding effectiveness (SE) of polymer composites based on natural graphite in flakes (NGF) and silicone rubber was investigated with the aim to contribute to the development of the technology of electromagnetic shielding materials. This topic has attracted great attention to the aeronautical and aerospace applications, due to the serious problems that EMI can cause to the functioning of electronic devices. According to this, the present work has produced samples of composite materials with variations on the sizes of the filler particles and composition of the samples. The electromagnetic characterization of the samples is given by the Vector Network Analyzer (VNA) in the X-band frequency range (8.2 – 12.4 GHz). The results indicate that the variation of particle sizes is determinant to the SE performance along with the X-band frequency range. Furthermore, the expansion of the range of granulometry allows controlling the curves of the peaks along the X-band.

Keywords: Composite Materials, Electromagnetic interference, Silicone rubber, X-band.

1 Introduction

Radiation-absorbing materials of the microwave frequency range have attracted great attention in the military and civil applications [1]. Due to the increase of EMI that causes serious problems such as malfunction of electronic devices and detrimental effects on human bodies exposed to this radiation [2].

In many years researches are being made on electromagnetic shielding materials with the intent to minimize troubles that the EMI can cause in so many areas of technology, to improve techniques that already exist and also to develop new techniques especially for applications in the aeronautical and military field, which generally use the X-band frequency range (8.2 – 12.4 GHz), such as radars, communication satellites, aircraft navigation systems, and so on. However, the advances in these techniques still face big challenges in characterizing an efficient applicable material for these purposes [3, 4].

Many studies indicate that the two essentials types of elements necessary to develop a shielding material are, the dielectric material as the matrix and the conductive material as the conductor of the incident energy. These two elements combined must have good properties of electrical permittivity, magnetic permeability, ability to induce electric dipoles while dissipating minimal energy in the form of heat, broadband reflection/absorption, lightweight, thin thickness, high mechanical and thermal stability [5–7].

According to the characteristics that a shielding material requires, the carbonaceous material tends to meet the conditions necessary to accomplish the efficiency of an EMI SE, because of its good electrical, mechanical and thermal properties, low density, simple preparation, and low cost. SE is defined as the capacity to protect a system by preventing the transmission of electromagnetic radiation by the effects of reflection and absorption [8–10]. A large range of carbon materials is being used to develop EMI SE, such as carbon fiber, carbon nanofibers, activated

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carbon fiber, graphite, graphene, and so on [2, 11–13]. NGF meets all the required conditions to act as a good EMI SE, and it is widely found in nature in its most common form of microcrystalline graphite in flakes, formed in either metamorphic or igneous geologic environments [2, 14, 15].

Once it is possible to combine dielectric materials with conductive materials, characterizing then in a polymer composite capable of acting as an electromagnetic filter with the function of attenuating the incident electromagnetic radiation, by the mechanisms of shielding from reflection, absorption and multi internal reflections [16, 17]. This work aims to produce and characterize samples of silicone rubber with NGF, and study the dependence on the composition, on its thickness, and on the variation of the sizes of the conductive fillers, with the properties on its SE performance. And finally contribute to the development of the electromagnetic shielding technology in the aeronautical and aerospace environment.

2 Materials and Methods

2.1 Preparation of samples

For this study, samples of silicone rubber and NGF were produced following the measurements of 22.80 mm in length by 10.20 mm in width, and 2.0 mm thickness, with two different concentrations, 95.0 wt.% of silicone rubber plus 5.0 wt.% of NGF and 90.0 wt.% of silicone rubber plus 10.0 wt.% of NGF.

The dielectric matrix (silicone) it is commercially available from ABCOL[®], and the conductive fillers (graphite) by the Nacional de Grafite LTDA in three different groups of particulate size (PS), which will be classified respectively as to “P1” for $PS > 300\mu\text{m}$, “P2” for $250\mu\text{m} > PS > 106\mu\text{m}$, and “P3” for $150\mu\text{m} > PS > 45\mu\text{m}$, as shown in Table 1.

Table 1: Respective classifications for the particle size of NGF.

Particle nomenclature	Granulometry range (μm)
P1	$PS > 300\mu\text{m}$
P2	$250\mu\text{m} > PS > 106\mu\text{m}$
P3	$150\mu\text{m} > PS > 45\mu\text{m}$

2.2 X-Ray Diffraction

The X-ray Diffraction patterns were performed by the PANalytical X-pert Pro, $\text{CuK}\alpha$ X-ray is used, with an accelerating voltage and current of 30 – 50 kV and 15mA, respectively. The diffraction data were collected by step scanning with a step size of $0.02^\circ \theta$ and a scan step time of 1.0 s.

2.3 Raman Spectroscopy

Raman spectra of all carbonaceous materials exhibit characteristic peaks in the region between 1000 and 1800 cm^{-1} for visible excitation energy. The bands found in these ranges are known as bands D (1200 to 1400 cm^{-1}), G (1500 to 1400 cm^{-1}) and D' ($\approx 1620\text{ cm}^{-1}$). The G band can be related to the graphite C-C vibration, on the other hand, the bands D and D' are related to the hexagonal structure disorder of graphite [18, 19]. In general, the Raman technique allows to understand the structural property of carbonaceous materials, in particular, it is used to investigate the defects present in the carbon network. In this work, from the Raman spectroscopy peaks, it was possible, quantitatively, to evaluate the change in the graphite organization for samples of P1, P2, and P3 through fitting by the first-order peak D and G using the method of Sadezky and the smallest square chi. This method consists of a combination of four Lorentzian bands (G, D1, D2, D4) and a Gaussian band (D3) [19].

The Raman Spectroscopy was acquired using a Horiba Scientific LabRAM HR Evolution using the green line of an argon laser $\lambda = 514.5\text{ nm}$. The samples were placed on a glass substrate, the laser intensity was 10 mW at the sample and no filters were used. Extended scans 1200 to 1700 cm^{-1} were performed to obtain the first Raman bands of the samples, with typical exposure times of 30 s.

2.4 Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) images were obtained using an ultra-high-resolution field emission microscope (TESCAN, Mira 3) equipped with an in less detection system and operated at an acceleration voltage of 1.0 kV. A working distance of between 2.0 and 3.0 mm was used.

2.5 Measurements of Electromagnetic Properties

The results of the measurement of the scattering parameters (S Parameters (S_{11} ; S_{21} ; S_{22} ; S_{12})) of the device un-

der test (DUT) was performed by a rectangular waveguide method using a Vector Network Analyzer (VNA) model N5235A. The DUT was submitted to an electromagnetic pulse (V^+) in the frequency range of 8.2 to 12.4 GHz and measured the magnitude and phase of the reflected signal (V^-) [20]. Figure 1, illustrates the reflection (S_{11} ; S_{22}) and transmission (S_{21} ; S_{12}) processes of the DUTs quantified by $R = |S_{11}|^2 = |S_{22}|^2$, and $T = |S_{21}|^2 = |S_{12}|^2$, respectively. The amount of absorbed radiation A is obtained indirectly by the $A = 1 - (T + R)$, where “ R ” represents the portion of the electromagnetic radiation that has undergone reflectivity phenomenon, “ T ” represents transmitted radiation and “ A ” represents the portion of the radiation that was absorbed by the material medium [21, 22].

Figure 1: The schematization of the signals emitted by a Vector Network Analyzer.

Therefore, the total amount of electromagnetic shielding effectiveness (SE_T) of a material can be quantified as the sum of the contribution from the shielding from reflection (SE_R), shielding from absorption (SE_A) and the shielding from multiple internal reflections (SE_{MR}) [23, 24].

$$SE_R (dB) = 10 \log_{10} \left(\frac{1}{1 - R} \right) \quad (1)$$

$$SE_A (dB) = 10 \log_{10} \left(\frac{1 - R}{T} \right) \quad (2)$$

$$SE_{MR} (dB) = 20 \log_{10} \left| \left(1 - R^2 10^{\left(-\frac{SE_A}{10} \right)} \right) \right| \quad (3)$$

$$SE_T (dB) = SE_R + SE_A + SE_{MR} \quad (4)$$

3 Results and Discussion

3.1 Morphological and Structural characterization

The morphology of the NGF was analyzed from a Scanning Electron Microscope (SEM). Figure 2 presents the SEM images of the NGF, with lattices in the form of flakes with

Figure 2: SEM of the NGF, (A) $PS > 300\mu m$, (B) $250\mu m > PS > 106\mu m$, (C) $150\mu m > PS > 45\mu m$.

Figure 3: X-ray diffraction patterns of the NGF.

a rough surface and evidence the difference between the sizes of the particulates. Each group of granulometry may have distinct behaviors in the interaction with the electromagnetic radiation, depending on the frequency of the incident radiation [25]. The composites containing larger particle sizes may reflect a larger portion of the incident electromagnetic radiation when compared with the smaller particles, once you have a larger surface area for the incident electromagnetic wave to reach. Where on the other hand, with the composites containing smaller particles sizes, the absorption phenomena may occur more evident, whereas due to the reduction of the surface area of the particles, the electromagnetic wave can be transmitted more easily by the material medium and create then a path for the electromagnetic wave to be reflected by the particles of the medium [2]. According to the study of Wei Xie *et al.*, once an electromagnetic wave reflects many times in the NGF, thus can be absorbed many times by the polymer composite.

Figure 3 and Figure 4 present the structural characterization of the NGF in those three groups of granulometry used in this work. The X-ray diffraction patterns in Figure 3 show that all samples analyzed, obtained the three most relevant peaks of the crystalline structure of carbon, with no presence of other peaks characteristics of impurities. The planes 002, 004 and 112 at 26°, 55°, and 87°, respectively, follows the characteristic values of the graphite which consists in a succession of layers of graphene parallel to the basal plane of hexagonally linked carbon atoms [26]. The peak of the plane (002), of greater intensity, represents the c axial plane, perpendicular to the hexagonal planes of graphite [27, 28].

Figure 4: Raman spectra fitting of the (a) P1, (b) P2, and (c) P3 of NGF.

Figure 4 shows the first order G and D bands relating to sp^2 carbon networks and the bands' curve fitting technique for different particles size of NGF. Although the fitting method of Sadezky *et al.* used in this paper it was not

Table 2: Fitting parameters of the G, D1, and D2, bands for the NFG with different particles size.

Band	Line Shape	P1				P2				P3			
		Intensity	Area	Center	FWHM	Intensity	Area	Center	FWHM	Intensity	Area	Center	FWHM
D1	Lorentz	67	906	1324	70	115	4001	1332	46	92	2165	1335	44
G	Lorentz	146	2071	1580	15	219	4184	261	16	219	5363	1583	17
D2	Lorentz	-	-	-	-	72	365	1621	18	70	173	1622	12

necessarily used by the D4 and D3 bands, due to the lack of bands around 1250 and 1450 cm^{-1} , respectively. It is well known that the D3 band is associated with amorphous carbon, in other words, the NGF is not amorphous [29]. However, the D2 band around 1620 cm^{-1} , was used only for samples with particles size P2 and P3, this occurred due to the appearance of the peak near G band. According to the literature, D1 and D2 bands have been proposed to originate from surface graphene defects [29] which allow the processes of single and double scattering of the photoelectron by the domain edge, respectively [30]. Therefore, the probability for this double scattering event to occur decreases as the domain size increases, with a limit for crystallite size equal to the length associated with the mean free path of the photoelectron involved in the double resonance process which is about 5 nm [30]. Therefore, this explains why the D2 band is inconsiderable for larger particle sizes (P1) than for smaller particulates (P2 and P3).

In the literature, it has been accepted that the relationship between band intensities is proportional to the degree of graphite organization [18, 29]. Thus, the ratio of D1 with $G = \left(\frac{I_{D1}}{I_G}\right)$ and D2 with $G = \left(\frac{I_{D2}}{I_G}\right)$ band intensities are used to quantify this property for the NGF with different particle sizes. The parameters used for fitting the Raman of NGL are shown in Table 2.

Figure 5 demonstrates the ratios of disordered band (D1 and D2) intensities to the intensity of the G band. The I_{D1}/I_G ratio. According to the result of Figure 5, was observed that the NGL with particulates P1 had I_{D2}/I_G equal to 0.0, while P2 and P3 had a value around to 0.31. As for I_{D1}/I_G , the values for particulates P1, P2 and P3 were 0.46, 0.52 and 0.42, respectively. Consequently, the NGL with particles P1 possesses more double resonance processes than P2 and P3, even though all NGL samples present graphene defects.

3.2 Electromagnetic Characterization and Shielding Effectiveness

The electromagnetic characterization of the DUTs is given by the S Parameters measured from the VNA, and with the values obtained from reflection (S_{11} ; S_{22}) and trans-

Figure 5: Intensity ratios involving D1, D2 and G bands as function of particle size of NGF.

mission (S_{21} ; S_{12}) processes, the SE can be determined by applying equations 1, 2, 3, and 4. The addition of NGF to the polymer matrix of silicone rubber is 5.0 wt.% and 10.0 wt.%, with a thickness of 2.0 mm and a variation of one group of granulometry for each sample as shown in Figure 6, and a combination of two groups of granulometry for each sample as presented in Figure 7. In order to compare the effect of the conductive fillers with the polymer dielectric, an analysis was made with samples produced of only silicone rubber and classified as (P0), that is 100.0 wt.% of silicone rubber and 0.0 wt.% of NGF.

Figures 6 and Figure 7 show the SE of the samples produced for this study. The graphics are plotted by the results of the calculation for shielding from reflection (SE_R), shielding from absorption (SE_A), shielding from multi reflection (SE_{MR}) and the resultant (SE_T) of all three mechanisms of SE calculated by equations 1, 2, 3, and 4, respectively.

The graphs in Figure 6 shows that with an increase of 5.0 wt.% to 10.0 wt.% in the concentration, there is a slight variation in its SE values, though the DUT containing the smaller particle size (P3), the transmission portion gets higher with an increase in the concentration and frequency. When comparing the DUTs without NGF (P0) the transmission process is considerably stronger than those

Figure 6: SE of the polymer composites in 5.0 and 10.0 wt% of NGF. (a) SE_R , (b) SE_A , (c) SE_{MR} , and (d) SE_T .

with filler particles. Owing to the fact that the silicone rubber practically does not interact with the electromagnetic field due to its low conductivity. In Figure 7 the DUTs with a combination of two groups of granulometry did not present much change on their reflection and absorption coefficients, but the addition of bigger particle size to the sample containing the smaller ones contributed to normalizing the bandwidth results, linearizing the response that occurred in Figure 6.

The DUT with particles P3 and 10.0 wt.% (Figure 6), showed a decrease in its values with the increase of frequency, meanwhile, when combining P3 with P2 or with P1 (Figure 7), a stabilization on the coefficients of SE values along the X-band can be obtained. Consequently, the results demonstrate that when smaller particles (P3) is added an absorption index different from that shown if us-

ing only one particle size distribution (P1 or P2) can be obtained.

As foretold in section 3.1, this effect is associated with the increase of the specific surface area of the polymer composite, in which, its specific area extension allows to generate a high index of atoms with sigma bonds, and thus, promoting an increase in its dipole moments and polarization [2]. Such a physical phenomenon explains the increase in the electrical permittivity and magnetic permeability of the material, and finally resulting in an improvement of the interaction between the electromagnetic wave and the composite material, and also contributing to the SE results.

In Figure 6 it can be seen that the results of the SE_R (a) of the DUTs filled with the conductive material (NGF) presented a considerably higher value than the sample with only silicone rubber (P0), starting from ~6.6 dB to ~3.5 dB

Figure 7: SE of the polymer composites with combinations of granulometry in 5.0 and 10.0 wt% of NGF. (a) SE_R , (b) SE_A , (c) SE_{MR} , and (d) SE_T .

for (P1) with 5.0 wt.%, ~ 8.0 dB to ~ 2.6 dB for (P1) with 10.0 wt.%, and an average value of ~ 1.5 dB for P0. For SE_A , (b) the presence of NGF did not show great influence, in which the average value remained close to ~ 1.0 dB. These results of SE_R and SE_A refer to the electromagnetic properties of the NGF that contribute directly to the parameters of absorption and reflection of the incident electromagnetic waves [5, 31].

Both plots of shielding from reflection and absorption are in a proportional dependence with frequency. As the frequency increases its value the reflection loss decreases, and the absorption loss gets higher, as stated by Wei Xie *et al.* [2] this phenomenon is associated with particle size, as discussed earlier. This can be explained as the principle for highly conducting materials, where only conductivity (σ) and magnetic permeability (μ) are important, such that

the SE_R is dependent upon their ratio (*i.e.* σ/μ) while the SE_A is a function of their product ($\sigma\mu$) [31].

The influence on the concentration and on the sizes of the particulates is also detected on the properties of the SE_T . There is a pattern on the SE results where as the concentration and the size of the particulates in the DUTs is increased, the total amount of SE_T is also increased. For the DUTs with 5.0 wt.% and containing P3 particles the SE_T obtained was (~ 0.7 to 1.0 dB), for the P2 particles a SE_T of (~ 0.7 to 1.1 dB), and for the P1 particles a SE_T of (~ 0.4 to 1.2 dB). For the DUTs with 10.0 wt.% and P3 particles a SE_T of (~ 1.6 to 1.5 dB), for the P2 particles a SE_T of (~ 1.0 to 1.3 dB) and for the P1 particles a SE_T of (~ 2.1 to 2.7 dB). Increasing the concentration from 5.0 to 10.0 wt.% caused to the variation of the particulate size to be more evident, for instance in SE_R , the values of P2 and P3 are

practically the same to 5.0 wt.%. However, when the concentration is 10.0 wt.%, a variation in the SE_R of each size occurs, showing that the reflection is more effective as the particle size increases, considering that P1 had the highest SE_R . In terms of absorption, the variation of the particulate size was not significant, but the increase of the concentration generates a slight increase in SE_A , corresponding to that there is more carbon in the system.

The negative values show a predominance of the effect of multi reflections on the behavior of electromagnetic shielding. This effect is the result of the low percentage of SE_A (described in Eq. 3) in which the electromagnetic wave remains reflected inside the material through its boundaries. Eq. 3 shows that the lesser of SE_T values, the greater the amount of energy transmitted T , as a function of the amount of attenuated energy in the material medium $\left(-10^{\left(\frac{-SE_A}{10}\right)}\right)$.

The results demonstrate that the addition of NGF contributes to the reduction of the transmitted energy, considering that the SE_T has a positive value in dB scale. This effect is most emphasized for the DUT with 10.0 wt.% and particulates P1. By analyzing the results shown in Figure 6 it is possible to conclude that the reflection phenomenon is what contributes most to the reduction of T . Confirming that the characteristics of the conductive fillers are of great significance to the properties of SE_T , as anticipated in section 3.2.

As seen in Figure 7 the results of the DUTs with combinations of particle sizes did not show influence on the SE_A , though it shows that there is a combination of effects for the SE_R values, considering the fact that all combinations generated values close to each other. For the reflection phenomenon, the use of particulates with combined sizes provided the filling of the empty space between the particulates with only one group of granulometry, expanding the range of distribution within the dielectric matrix and enabling an improvement on the shielding performance. Where, as the SE varies with frequency, geometry, angle of incidence and so on [1, 4, 23, 32]. The expanse of the granulometric distribution showed a possible parameter to provide a constant SE_R results even with the increasing of frequency. Pointing that for the results of SE_T , the combination of two groups of particulate sizes allows increasing the shielding potential, where the SE_R presents a bigger portion on the mechanisms of SE . Although it is clear that the quantitative effect of multi reflections is predominant, which is mostly due to the specific large interface area of the composite material containing the filler particles. This specif interface area generates a high index of reflection of the electromagnetic wave among the surfaces

of the filler particles. Though contributing negatively to the results of SE_T .

4 Conclusions

The polymer composite based on silicone rubber and NGF presented an interesting performance in the analysis of the electromagnetic shielding effectiveness on the X-band frequency range (8.2 to 12.4 GHz). The concentration, variation, and combination of the particles sizes of the conductive fillers (graphite) showed a considerable behavior on its electromagnetic properties. Increasing the concentration of the composites from 5.0 wt% to 10.0 wt% resulted in higher values of SE . Composites produced with larger particle sizes “P1” showed slightly better performance on the SE_T when compared to the composites with the smaller ones. The mechanism from SE_R contributed with the higher percentage to the SE_T , due to its higher specific surface area. Although when two groups of granulometry were combined in one composite, the results became linear around the X-band frequency range, which contributed to increasing the bandwidth SE_T . Therefore, the variation and combination of particle sizes showed a promising property on the material shielding efficiency, which allows manipulating the SE_T performance over the 8.2-12.4GHz microwave frequency range.

Acknowledgement: This work was financially supported by the National Council for Scientific and Technological Development funding agency [136196/2017-9], and to the fellow collaborators that aided to the development of this work.

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