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Graphene Nanoplatelets on Multi-Scale Polymer Composites for Potential Ballistic Shielding

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Continuous development, improvement and innovation of ballistic material systems with superior energy absorption performance has been the subject of numerous works nowadays. Some research has been carried out with the objective of replacing traditional metallic and ceramic materials and composites with high-performance polymeric composites. Due to their excellent properties, multi-scale polymer composites have been used in the most varied segments of the industry, being recently researched in applications such as ballistic materials. This work promotes the combination of high-strength three-dimensional woven aramid fiber fabrics with vinyl ester resin to produce a panel through the compression molding process. The resin was reinforced with graphene nanoplatelets (0.1, 0.2 and 0.3% wt). The impact, tensile and flexural strength were evaluated, along with dynamic-mechanical analysis by DMA and by Hopkinson split bar test, indicating a trend of better performance for the composite containing 0.1% of graphene nanoplatelets.

Keywords: *Graphene nanoplatelets, vinyl ester resin, aramid fiber, composites ballistics, dynamic-mechanical analysis.*

1. Introduction

Military operations including weapons and ammunition are increasingly driven by technology. This demands the development of innovative ballistic material systems, which are expected to be superior in terms of energy absorption, damage resistance, and flexibility, but at a lighter weight. Wearing heavy and inflexible body armour for an extended period could generate excessive heat and reduce mobility[1,2].

The utilization of ballistic shielding such as protective helmets, body armors, and military vehicles has promoted the opening of several fields of studies to be investigated. A body armor panel is comprised of several layers of different materials depending on the threat level to protect the wearer's upper torso[1]. Among the important factors that must be considered when choosing materials used in ballistic shielding structures are weight, volume, resistance and, when used close to the body, they should allow better mobility and comfort to the user[3,4].

It is known that the matrix plays a key role in ballistic performance. For this reason, ceramics are commonly used in combination with other materials for ballistic protection of civilian and military equipment where low weight is a key requirement. Ceramics are attractive materials because they offer low density and high hardness; however, ceramics are highly brittle[5]. Therefore, it is necessary to find solutions to issues in materials selection for ballistic protection.

Aramid fibers – high-strength three-dimensional woven – are used in protective clothing and helmets, bulletproof jackets, and body armor, for example, since the impact resistance of aramid fibers is better than that of other fibers[6,7]. These fibers can also produce a contoured, form-fitting and flexible panel through moulding processes using the three-dimensional woven material. An adhesive resin can be used for wetting the yarns in each sheet without compromising flexibility[1]. In this context, the combination of a three-dimensional woven fabric such as aramid, for example, with thermosetting resins presents a promising class of cutting-edge ballistic materials called multi-scale polymer composites.

The heterogeneous nature of composite materials due to its multiphase characteristics at macroscopic level gives an added advantage, wherein they exhibit the best qualities of their components along with unique properties[8,9]. The significant role that the polymer matrix plays on ballistics is further supported by the fact that delamination has been shown to be an influential factor governing ballistic response and performance[10,11].

Vinyl ester resin, a thermosetting polymer, has been widely used as a matrix in composite materials. They have intermediate properties between those of epoxy and unsaturated polyester resins in terms of chemical, physical, mechanical, and curing characteristics. One of the key shortcomings of this resin is the low impact resistance due to its brittleness. One of the simplest ways to solve such a problem is by incorporating fillers into the vinyl ester resin. Many studies have been performed to increase the impact resistance in these resins[12-14].

It is known that polymers can be reinforced by a number of nanomaterials to enhance its properties and to increase

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their range of applications. With the help of nanoscience and nanotechnology, many nanomaterials have emerged recently, and these can be used as filler materials to make polymer nanocomposites[6].

Graphene-based nanocomposites are rising star materials that greatly open a promising research field in the design of a new generation of body armor systems[1]. Graphene nanoplatelets (GNPs) can offer barrier properties when used in composites and can also improve their mechanical characteristics, including stiffness and tensile strength of different composites due to their strong interfacial interaction with the matrix[15]. This increase in the properties of nanocomposites also depends on the dispersion of the layers of graphene or graphite in the polymeric matrix[16].

According to a review by Abtew et al.[1], a great effort has been dedicated to assessing the potential applications of graphene and its oxide in the developments of body armors, Besides, graphene quickly absorbs and dissipate its energy effectively to halt the projectile due to its higher sound wave propagation performance, and higher strength, stiffness, and structural anisotropy compared to steel. However, most of this research deals only with impact tests on nanometric graphene sheets or even with the prediction of results by numerical and analytical modeling[1].

Studies on resin polymeric composites and nanocomposites with graphene and high-strength fabrics (aramid woven) have been carried out by many researchers, due their inherent processing ease and low density[17-19].

Given this perspective, the aim of this work is to study the influence of the addition of different concentrations of GNPs into vinyl ester resin when used in multi-scale composites with aramid fibers. Their impact, tensile and flexural strength were evaluated, along with the dynamic-mechanical properties by dynamic-mechanical analysis, and dynamic properties obtained from split Hopkinson pressure bar testing.

2. Materials and Methods

2.1. Materials

The vinyl ester resin Derakane (grade 411-350, brand Ashland) was acquired from Disfibra (RS, Brazil). Cobalt octalate 6% (Liosec HC, brand Miracma Nuodex) was used as a curing promoter, and was obtained from Disfibra (RS, Brazil). Initiator methyl ethyl ketone peroxide (Butanox M-50, brand Akzo Nobel), was obtained from Disfibra (RS, Brazil). The graphene nanoplatelets (GNP) were obtained from Strem Chemicals (grade 06-0220, 6-8 nm thick, 25 μm wide). Aramid fiber fabrics from Barrday, description A314 KV K129 2820D 1x1, flat, were donated by the Center of Technology of the Brazilian Army (CTEx – Centro Tecnológico do Exército).

2.2. Dispersion of GNPs and assembly of composites

GNP were added to the vinyl ester resin in concentrations of 0.1%, 0.2% and 0.3% (wt%). After quick manual homogenization, the mixture was sonicated in a Sonics Vibra-Cell VCX-500 equipment, with an amplitude of 40%, net power 500 W, during 30 minutes. The mixture was kept in an ice bath to lower the temperature of the system. After cooling to room temperature, the initiator and curing promoter were added, in a proportion of 1.0% each (wt%), followed by complete manual homogenization. Using a spatula, the first layer of vinyl ester resin mixture with GNPs was applied to the mold, followed by a layer of aramid fabric. Subsequently, another layer of vinyl ester resin was applied to the fabric, followed by another layer of aramid, and so on until 10 fabrics were used. A stainless-steel mold with male/female halves and internal dimensions of 170 x 170 mm was used, with a 5 mm mold cavity. A pressure of 5 bar was applied to the mold for 18 ± 2 minutes at a temperature of 80 ± 5 °C. After opening the mold, the composite was removed and placed in a forced circulation oven for 4 hours at 80 °C and 2 hours at 120 °C to perform the post curing process.

2.3. Characterization of composites

The viscoelastic properties (storage modulus and tan delta) of the composites were determined using dynamic-mechanical analysis (DMA). The analyzes were performed using a TA Instruments Q800 AT equipment. A dual cantilever clamp was used, and the tests were performed in a non-isothermal method, in a temperature range of 30 to 160 °C with a heating rate of 3 °C.min^{-1}, frequency of 1 Hz and deformation amplitude of 0.1%.

The Izod impact strength tests were performed using a maximum pendulum energy of 294 J. The tests were performed according to the ASTM D256-10 standard with an adaptation of the ASTM E23-12C standard. The tensile strength tests were carried out in an EMIC DL – 3000 universal testing with a load cell of 200 kN. The tests were performed at a speed of 5 mm.min^{-1}, according to ASTM D3039-14 standard with adaptation of ASTM A370. The flexural strength tests were performed in a universal mechanical testing machine EMIC DL – 3000. A 200 kN load cell with a speed of 1.8 mm.min^{-1} was used, according to the ASTM D7264M-15 standard.

The dynamic mechanical compression tests on Hopkinson split bar were performed in a Rel Inc® equipment with a Sure Pulse® software (REL SURE PULSE® 2014), a Pico Scope 5000 Series® oscilloscope from Pico Technology ™ and a Vishay Micro Measurements ™ 2300 System® signal amplification system. In each test, the following were used: a 203.20 mm long impactor, and 2 m long cylindrical incidence and transmission bars of aluminum alloy 7075-T6, both with a 19.05 mm diameter. Table 1 shows the parameters used in the experiment. The nomenclature adopted for composites is the acronym GNP followed by the numbers that indicate the percentage of nanofiller (GNP0, GNP01, GNP02 e GNP03).

Table 1. Split Hopkinson pressure bar parameters: impact velocity (v_{SB}), deformation (ε), sample thickness (L_A), sample diameter (D_A).

Sample	v_{SB} (m/s)	$\dot{\varepsilon}$ (s^{-1})	Sample	
			L_A (mm)	D_A (mm)
GNP0	12.93	1608.47	4.76	10.36
GNP01	13.35	1631.56	5.12	10.41
GNP02	13.70	1676.67	5.00	10.63
GNP03	11.92	1760.03	4.46	10.55

3. Results and Discussion

3.1. Dynamic mechanical analysis

Figure 1 shows the storage modulus (E') and tan delta curves of the GNP0, GNP01, GNP02 and GNP03 samples. The highest storage modulus in the glassy region – at around 30 °C - was presented by the sample GNP02 (29.0 GPa), followed by samples GNP01 (27.9 GPa) and GNP03 (27.1 GPa). Assuming a deviation of 10% in E', there is no significant difference between the E' values. However, a general tendency towards the increase of E' up to 2% GNP content was observed and could be attributed to the agglomeration of the nanofiller in the polymer matrix[20].

The storage modulus is associated with material stiffness, that is, it reflects the elastic properties of the materials[21]. As the temperature increases, E' decreases for all composites, and this can be attributed to the increased molecular mobility of the polymer chains[22]. The tendency towards an increase in the storage modulus in both the glassy and rubbery regions could possibly indicate increased stiffness due to the addition of GNP. Almajid et al.[23] the same behavior in their studies using vinyl-ester resin nanocomposites and carbon nanotubes (CNT) – 0.01 to 0.3% (m/m) or graphene (0.5 to 5% wt) – and, even at low concentrations, the nanofillers promoted an increase in the E' of the nanocomposites at room temperature in relation to the pure resin. The authors attributed this increase to the good dispersion of the nanofillers. Adequate dispersion, even with low levels of GNPs, can create a strong interaction with the vinyl ester matrix, resulting in effective stress transfer at the interface, which contributes to the increase in E'. Surnova et al.[21], reports that well dispersed nanofillers increase the contact surface area with the polymeric matrix and provides better matrix/nanofiller interaction.

The glass transition temperature (T_g) is related to the degree of molecular mobility of the materials and it was taken at the maximum intensity of the damping curve (tan δ). The T_g values found for the samples were 113 °C, 114 °C, 113 °C and 111 °C for GNP0, GNP01, GNP02 and GNP03, respectively. The slight reduction in the T_g found for the composite with 0.3% GNP could be explained by a possible agglomeration of the nanofillers. For nanocomposites, when a concentration threshold is reached, a decrease in properties is observed due to agglomeration[20,24].

A decrease in the tan delta peak height was observed with the addition of GNP when compared to the GNP0 sample. The tan delta peak height of the composites decreased with the addition of GNP. The height and position of the tan delta peak are related to the degree of mobility of the polymer chains[25,26]. According to Ornaghi et al.[22] the damping or adhesion factor tends to decrease whenever there is greater interaction between the components. This could probably be due to the rigid nature of GNP and, as such, by introducing a rigid phase, the molecular mobility is hindered[20].

3.2. Mechanical testing

Figure 2 shows the impact strength of the composites. A decrease in the impact strength was observed for the GNP01 sample. For 0.2% and 0.3% GNP content, there is no statistical difference between the samples in relation to the neat resin. The reason for the behavior herein found could lie on several factors ranging from graphene dispersion in the vinyl-ester matrix to factors influencing the composites' manufacturing. When fabricating the composites by compression molding using liquid resins, it is important that the resin is well-distributed between the fabric layers before applying pressure and temperature. When closing the mold, the resin must be able to flow through the fabrics. If the resin does not flow, any places left with no resin become sites with no fiber/matrix interface. When there is no adhesion in the interface, stress transfer is not possible, and the mechanical properties of the composites are affected.

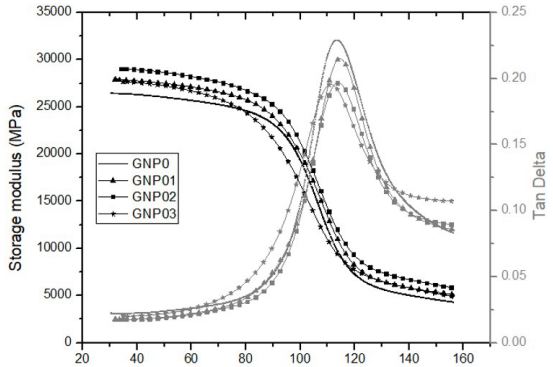

Figure 1. Storage modulus (E') and tan delta curves by DMA analysis.

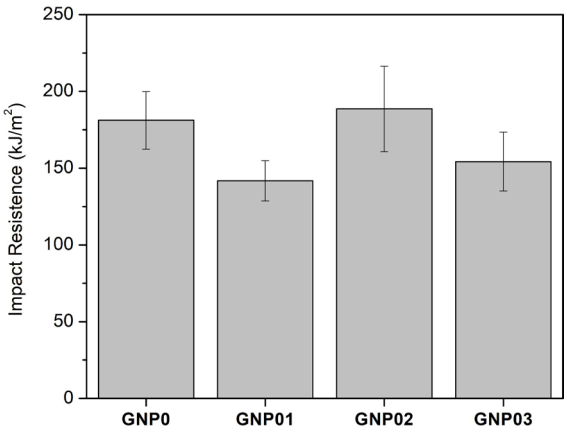

Figure 2. Impact strength of the samples studied.

Singh et al.[27], observed that epoxy composites reinforced with multi-wall carbon nanotubes (MWCNT) had higher impact strength in comparison to the composites reinforced with GNP. Moreover, some agglomeration was observed for epoxy/GNP when compared to epoxy/MWCNT. The results found by these authors are in line with the observations of this study. Figure 3 shows the tensile strength and tensile modulus of the samples GNP0, GNP01, GNP02 and GNP03. The elastic modulus of the composites increased for all GNP contents, although no particular trend was observed. The introduction of a rigid filler such as graphene derived nanofillers could induce an increase in the stiffness of the composites. This trend was observed by several authors when

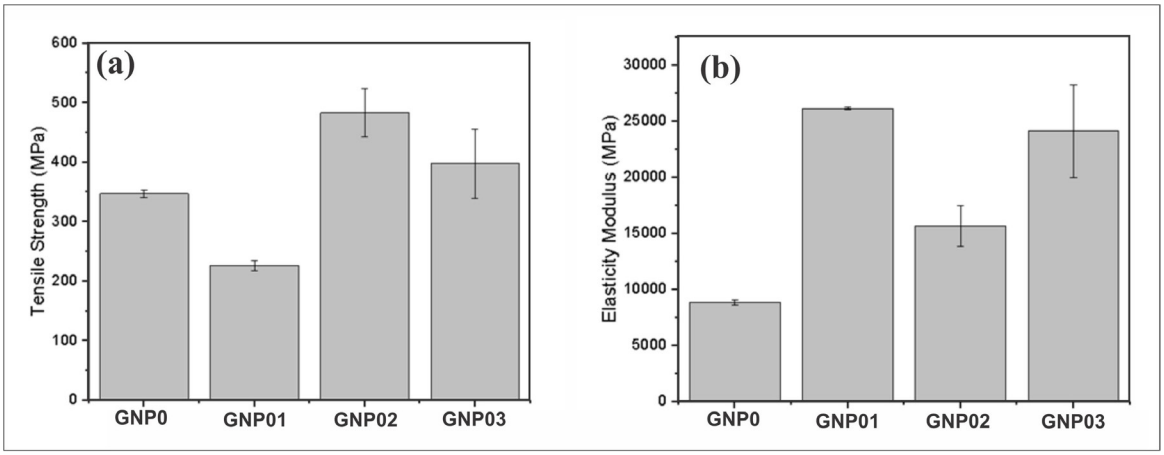

Figure 3. (a) Tensile strength and (b) Elasticity modulus from tensile testing of the samples studied.

incorporating GNP in thermosetting resins[20,28]. Zhu et al.[29], investigated the reinforcement of Kevlar® nanofibers with carbon nanotubes (CNT) to obtain strong and rigid nanocomposites. In their work29, CNT/Kevlar® nanofibers composite films were prepared by vacuum-assisted flocculation and vacuum-assisted layer-by-layer assembly and showed high final strength (up to 383 MPa) and Young's modulus (up to 35 GPa), with lower strength and modulus slightly superior to those obtained in this study for sample GNP01, in which we use vinyl ester resin as a matrix. According to the authors, the degree of dispersion could play a role in the composites properties and could explain the gap between the expected theoretical results and the practical results[29].

The tensile strength of the composites decreased with 0.1% GNP and increased with larger GNP content. According to O'Masta et al.[30] the addition of graphene can provide an increase in tensile strength and elastic modulus and stiffness – corroborating the DMA results – although it also causes a reduction in ductility[30]. Jofre-Reche et al.[16], in their studies on the effect of the addition of GNPs (0.1, 1 and 5% (w/w)) on the structural, thermal, mechanical, and viscoelastic properties of nanocomposites based on vinyl ester resin, observed that the addition of GNPs increased the tensile strength of the nanocomposite. The addition of GNPs restricts the deformation of the polymeric matrix, and so the tensile strength of the composite increases as the addition of GNPs in the composite increases. Some of the composites studied by the authors underwent a curing process at 100 °C, which provided almost completely cross-linked composites, consequently, with lower deformation capacity under load. Since the composites in this study were cured at 80 °C to 120 °C, the explanation may apply[16].

The flexural strength and flexural modulus results are presented in Figure 4. By analyzing the results of flexural strength, it is possible to verify that the results did not significantly differ from GNP0 considering the standard deviation. A similar behavior was observed by Almajid et al.[23] when using carbon nanotubes in vinyl ester resin. They verified that the flexural strength and the deformation did not improve with increasing carbon nanotube content, which could imply higher fragility of the composites as verified by the impact strength results[23].

The results for the flexural modulus corroborate that the level of dispersion is differentiated in the sample GNP01. In Figure 4b, the largest modulus is that of the sample GNP01 (25726 ± 1106 MPa). The GNP02 and GNP03 samples have values close to the GNP0 sample (18245 ± 825.7 MPa). A decrease when using higher concentration of GNP could be related to either poorer dispersion or higher void content, both due to agglomeration. Agglomerates act as stress concentrators and, as such, instead of distributing the stress evenly at the interface, the concentration may act by hindering the stress-transfer, therefore impairing the mechanical properties[20]. In this study, the flexural modulus results were found to be in line with the elastic modulus from tensile testing – that is, the addition of GNP increased the flexural modulus for all concentrations, especially for GNP01. Wang et al.[20] attributed this to the high modulus of GNPs, regardless of the particle size[20]. Overall, GNP01 obtained satisfactory mechanical and dynamic-mechanical behavior.

3.3. Split Hopkinson pressure bar (SHPB) dynamic testing

The purpose of adding fibers and fillers - or even nanofillers - to ballistic composites is to increase their strength and stiffness, and above all, to increase their ability to absorb and distribute kinetic energy laterally.

The specific ballistic response of a ballistic composite is determined from a set of conditions, such as armor composition, area density of the armor, mechanical boundary conditions and projectile characteristics (Manero II, 2015). Therefore, the intention of adding NPG to the ballistic composite is to test its influence on this system, considering that the addition of this nanofiller directly interferes in all conditions of the set.

Therefore, the Figure 5 shows the results of the average stress behavior as a function of the deformation of the GNP0, GNP01, GNP02 and GNP03 samples, obtained by Split Hopkinson pressure bar dynamic testing. Initially, it is possible to observe that the addition of GNP promoted an increased in the true stress for the composites with 0,1% and 0,2% of GNP (GNP01 and GNP02 samples) in comparison

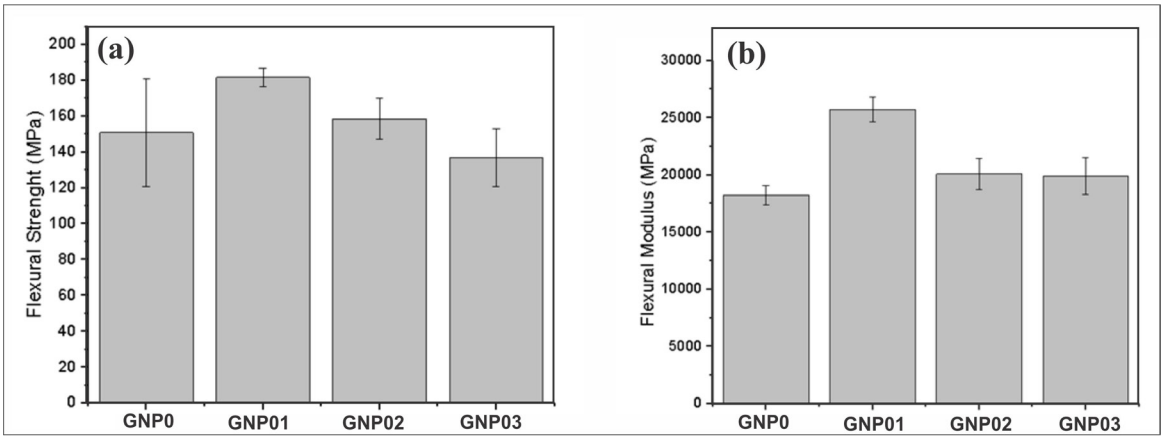

Figure 4. (a) Flexural strength and (b) Elasticity modulus from flexural testing of the samples studied.

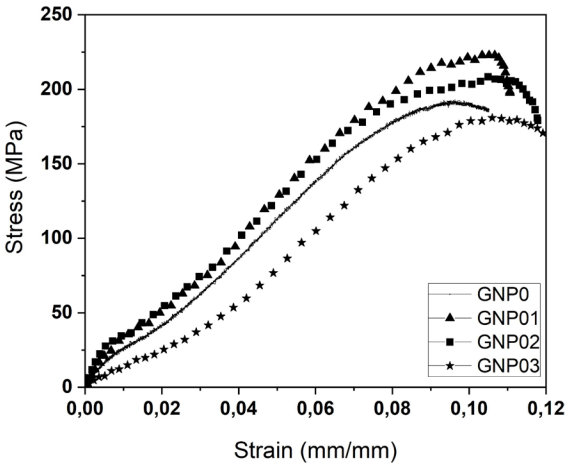

Figure 5. Stress vs strain curves obtained from split Hopkinson pressure bar dynamic testing.

to the neat resin. The highest values were found for the GNP01 sample, with a maximum stress 18% higher than the GNP0 composite. Table 2 presents the values of the dynamic properties of the samples obtained in the SHPB testing, being εt the total strain, σmax the mean maximum stress, εmax the strain at the maximum stress and Umax the maximum tenacity.

It is worth mentioning that this test is a dynamic loading of short duration. The efficiency of impact energy absorption is related to the type of structure material, loading mode (axial, transverse, combined, etc.), strain rate, among others. At higher strain rates, as in the SHPB test, fractures associated with in-plane wave propagation, such as delamination, starts to take place.

The addition of GNP in concentrations of 0.1 and 0.2% (GNP01 and GNP02) appears to be effective in absorbing energy during impact. This result was more expressive in sample GNP01, where the increase in maximum tenacity was around 12%, when comparing the GNP0 sample. This indicates that the GNP01 sample absorbs more energy before breaking than the others, due active deformation mechanisms. It is known that an armoring system must provide for the

Table 2. Split Hopkinson pressure bar dynamic results: mean deformation (ε_t), maximum average stress (σ_{max}), deformation at maximum stress (ε_{max}), maximum tenacity (U_{max}).

Sample	ε_t (mm/mm)	σ_{max} (MPa)	ε_{max} (mm/mm)	U_{max} (J/m^3)
GNP0	0.104 ± 0.01	191 ± 7	0.095 ± 0.01	16.68 ± 0.5
GNP01	0.111 ± 0.01	224 ± 8	0.107 ± 0.01	18.66 ± 0.7
GNP02	0.118 ± 0.01	209 ± 6	0.111 ± 0.01	17.92 ± 0.4
GNP03	0.119 ± 0.01	180 ± 4	0.109 ± 0.02	16.56 ± 0.4

spread of impact energy over the largest possible area, thus reducing the unit of energy per area of the armor to levels below the failure point of the armoring material.

The ballistic testing by Split Hopkinson pressure bar showed specific weight fractions of graphene nanoplatelets to be effective of raising the ballistic resistance performance, and showed that others were not. The decline in performance of the sample with 0,3% of GNP can be associated to agglomerations and difficulty transferring loading throughout the matrix and for nanofillers.

Materials can display favorable, or sometimes unfavorable behaviors when they are subjected to high-strain rates or high-pressure shocks. The effect of the deformation rate can be associated with the molecular movement of the polymer chains, which, when subjected to high deformation rates, have restricted molecular movement. In this case, there is less time for the polymer chains to reorganize due to the high velocity nature of the SHPB testing – contrary to what happens in tests in which the deformation occurs slowly and/or with an increase in the temperature e.g., tensile and dynamic-mechanical testing. Since the fundamental process in the obtention of amorphous polymers consists in the movement of the macromolecules segments from one point of equilibrium to another, when subjected to a higher deformation rate, there is greater molecular resistance to the movements, and as such higher stress[31].

4. Conclusions

In this study, it was observed appreciable changes on the properties of the ballistic composites with the addition

of graphene nanoplatelets. The lowest concentration of GNP, referring to sample GNP01, presented a superior dynamic-mechanical behavior retained the tenacity while displaying superior maximum stress in comparison to the remaining samples.

The impact strength test results showed that the samples containing GNP had a slight change in their resistance. It is worth mentioning that this test is a dynamic loading of short duration. The efficiency of impact energy absorption is related to the type of structure material, loading mode (axial, transverse, combined, etc.), strain rate, among others. At higher strain rates, as in the SHPB test, fractures associated with in-plane wave propagation, such as delamination, starts to take place. This mechanical behavior explains the increase in the energy absorbing capacity and the higher impact resistance of the material. Hence, for samples GNP01 and GNP02, as the strain rate increased, the material responded substantial increasing on Umax and σmax.

Then it is concluded that an increase in ballistic resistance with the addition of negligible weight of nano-particles can potentially affords either a reduction in composite weight to maintain the same performance or provides a marked improvement in ballistic resistance performance. For future consideration, all GNP concentrations should be implemented to the baseline composite to test for further increases in ballistic performance, as the mechanisms appear to be unique, non-linear, and could be optimized for high performance gains.

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