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Influence of Rigid Brazilian Natural Fiber Arrangements in Polymer Composites: Energy Absorption and Ballistic Efficiency

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Abstract: Since the mid-2000s, several studies were carried out regarding the development of ballistic resistant materials based on polymeric matrix composites reinforced with natural lignocellulosic fibers (NLFs). The results reported so far are promising and are often comparable to commonly used materials such as Kevlar[TM], especially when used as an intermediate layer in a multilayer armor system (MAS). However, the most suitable configuration for these polymer composites reinforced with NLFs when subjected to high strain rates still lacks investigation. This work aimed to evaluate four possible arrangements for epoxy matrix composite reinforced with a stiff Brazilian NLF, piassava fiber, regarding energy absorption, and ballistic efficiency. Performance was evaluated against the ballistic impact of high-energy 7.62 mm ammunition. Obtained results were statistically validated by means of analysis of variance (ANOVA) and Tukey's honest test. Furthermore, the micromechanics associated with the failure of these composites were determined. Energy absorption of the same magnitude as Kevlar[TM] and indentation depth below the limit predicted by NIJ standard were obtained for all conditions.

Keywords: natural fiber composites; ballistic armor; ballistic application; composite design

1. Introduction

The development of military technologies in the past 50 years gave birth to new arms and ammunition associated with great destructiveness power. At the same time, similar efforts were made to substantially improve the materials used as ballistic armor to somehow match the damage caused by those powerful weapons [1]. To be considered an efficient material for application in ballistic protection, the combination of specific characteristics such as low weight, penetration resistance, and high energy absorption is considered mandatory [2,3]. Commercial materials, such as aramid fiber (Kevlar[TM]) and ultra-high high molecular weight polyethylene (Dyneema®), exhibit these characteristics and, therefore, are the mainly used materials for applications in personal ballistic protection against high energy ammunition, such as 7.62 and 5.56 mm [4,5]. However, in recent years, polymeric matrix composites reinforced with natural lignocellulosic fibers (NLFs) were reported as promising materials for application as ballistic-resistant materials [6–13].

In a recent state-of-the-art review on the influence of using various NLFs as armor systems, Nurazzi et al. [8] enlightened about important characteristics of NLFs that caught the attention of researchers around the world to study their application in heavy armor equipment. They include vast availability and low-cost of these NLFs, as well as the

ease of manufacturing such composites. They also highlighted the vast possibility of enhancement to the properties that could lead towards a more reliable use of those NLF composites in ballistic applications. Naveen et al. [10] discussed about different standards and testing that are commonly used to evaluate NLFs reinforcing composites. In addition, they suggested that some strategies such as hybrid composites, i.e., the simultaneous use of two different reinforcements, one synthetic and one NLFs, could unlock superior ballistic properties. Luz et al. [12] compared the ballistic performance of several different NLFs reinforcing polymeric matrix composites when used as intermediate layer in multilayered armor system (MAS). Commonly known fibers such as jute, ramie, and bamboo, as well as less-known fibers such as curaua, mallow, and sugarcane bagasse, were considered in that assessment. All investigated NLFs used as reinforcement were able to meet the standard requirement of indentation depth and, furthermore, in every case, there was complete perforation of the target. Moreover, the results were like Kevlar^{TM} in similar MAS condition, and superior to those where only ceramic or Dyneema$^®$ monolayer protections were taken into consideration. Despite these promising ballistic results discussing the use of NLFs reinforcing polymer composites, most published works consider only aligned fibers or fabrics arrangements. Thus, deeper studies about the influence of the composite design are still needed to determine the condition with the highest possible ballistic efficiency.

The fiber extracted from a Brazilian palm tree, botanically named Attalea Funifera, also known as piassava fiber, is considered a promising candidate for reinforcing polymer matrix composites [14,15]. Indeed, the ballistic assessment of epoxy/piassava composites revealed superior properties of the composites reinforced with 30 vol% of fiber reinforcement [16,17]. However, only unidirectional alignment conditions were taken under consideration. In addition to that, the high stiffness of this fiber does not allow it to be woven as a fabric. In this context, the objective of this study is to assess individual performance and its application in MAS of epoxy matrix composite reinforced with 30 vol% of piassava fibers designed in four different arrangements to determine the influence of it on the ballistic impact energy absorption.

2. Materials and Methods

Composite plates were produced by compression molding using the epoxy resin diglycidyl ether bisphenol A, DGEBA, with the addition of triethylene tetramine hardener, TETA, in stoichiometric ratio phr 13, both supplied by the Epoxy fiber (Rio de Janeiro, Brazil). Piassava fibers were supplied by a local broom industry, Vassouras Rossi Ltd.a (Rio de Janeiro, Brazil), and used as reinforcement of polymer composites. Prior to the composite production, the fibers were cleaned in an ultrasonic bath with deionizing water for 20 min. Afterwards, they were placed in an air-oven at 80 °C for 24 h to dry and reduce the inherent moisture content of the fiber. This is an important step towards the improvement of the interfacial shear strength between polymeric matrix and NLFs reinforcement [18]. Then, composites with 30 vol% of piassava fibers and 70 vol% of epoxy were prepared by the hand layup process, which is presented in Figure S1. Uniaxial compression molding of 3 MPa at room temperature for 24 h were the parameters used in this process. The arrangement of fibers in composites is described in Table 1. The criterion to determine whether the fiber is long or short was based on pullout tests [19]. If the fiber length is less than 15 mm, it is considered short, and with a length above this value, the fiber is long. Figure S2 shows the macroscopic aspect of the produced composites plates.

Table 1. Nomenclature and configuration of investigated composites.

Nomenclature	Fiber Arrangement
LA2D	Long and aligned; arranged in three layers, each layer rotated 90° from the previous one; cross ply (two directions)
LA1D	Long and aligned fibers (one direction)
LRS	Long and randomly scattered fibers
SRS	Short and randomly scattered fibers

The production of the MAS, total thickness of 25 mm, was made by the union of a 5 mm aluminum alloy plate as third layer, the produced epoxy/piassava composite 10 mm thick as second layer, and a 10 mm Al_2O_3 + Nb_2O_5 ceramic tile as first layer. The 5052 H34 aluminum alloy plate was supplied by Metalak Metais Ltd.a (Rio de Janeiro, Brazil), while the Al_2O_3 + Nb_2O_5 ceramic tile was produced as reported elsewhere [20]. Schematic illustration of the MAS, as well as its front and top view, are shown in Figure S3.

Two different ballistic tests were performed at the Brazilian Army Assessment Center (Rio de Janeiro, Brazil). The used shooting device was a model B290 High Pressure Instrumentation gun barrel placed 15 m away from the target. As a projectile, 7.62 mm M1, 9.7g full-metal jacket, commercial ammunition was used. For the individual performance of the epoxy/piassava composites, the experimental apparatus also included a Doppler radar system, model SL-520P, Weibel (Alleroed, Denmark), to track the velocity of the projectile before and after the ballistic impact. As for the MAS evaluation, the target was placed in front of a modeling clay witness. The clay witness was a Roma Plastilina type, supplied by Corfix (Porto Alegre, Brazil), used to simulate the consistency of the human body. This test is also known as Perforation and Back-face Signature Test, and it is supported by NIJ standard [21]. Figure 1 present the schematic configuration of both ballistic tests, as well as both configuration targets, individual and MAS, before their test.
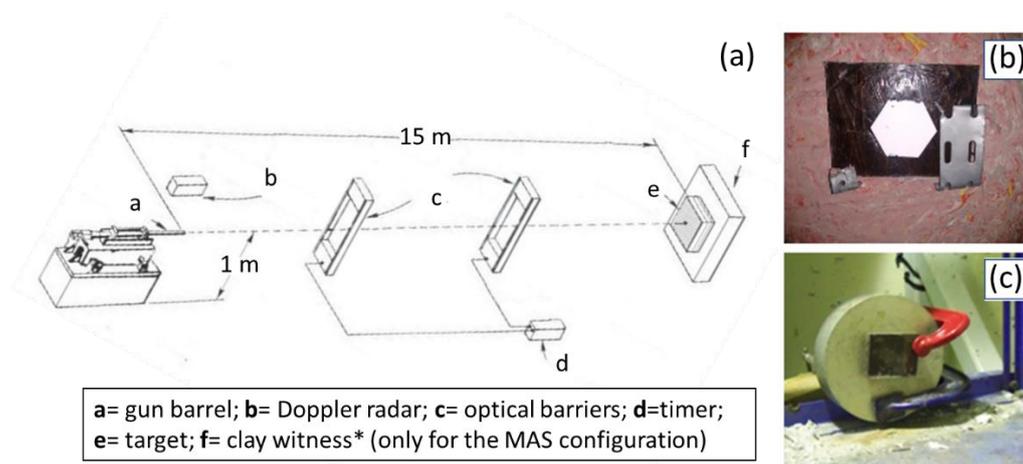
Figure 1. Ballistic test (**a**) schematic illustration; (**b**) MAS target placement, and (**c**) individual performance target placement.

Raw data obtained from these ballistic tests were processed with the Windpp software. Ballistic parameters of such as absorbed energy, limit velocity, and indentation depth were measured and calculated. Results were statistically validated by means of the analysis of variance, ANOVA, and Tukey's honest test [22,23].

Finally, macro- and microscopic analysis qualified the physical integrity of the tested materials and verify the failure mechanisms of energy absorption associated with the dynamic impact. Scanning electron microscopy (SEM) was performed in a model Quanta FEG 250, FEI microscope, (Oregon, OR, USA). All samples were metallized with platinum or gold in a model EM ACE600 equipment, LEICA (Wetzlar, Germany).

3. Results

3.1. Individual Performance of Epoxy/Piassava Composites

Figure 2a presents typical raw data of Doppler radar. In Figure 2b, the curve adjustment by means of Windopp software is shown. In this radial velocity fitted curve, one may verify that the projectile is fired with an initial velocity of 863 m/s, and it reaches the target, i.e., epoxy/piassava composite, at a velocity of 850 m/s. The projectile's impact with the target is associated with vertical line at about 0.018 s. The projectile exits the target with a residual velocity of 832 m/s. The shape of the single curve, with continuously decreasing velocity, indicates that the bullet was kept intact after the ballistic impact.
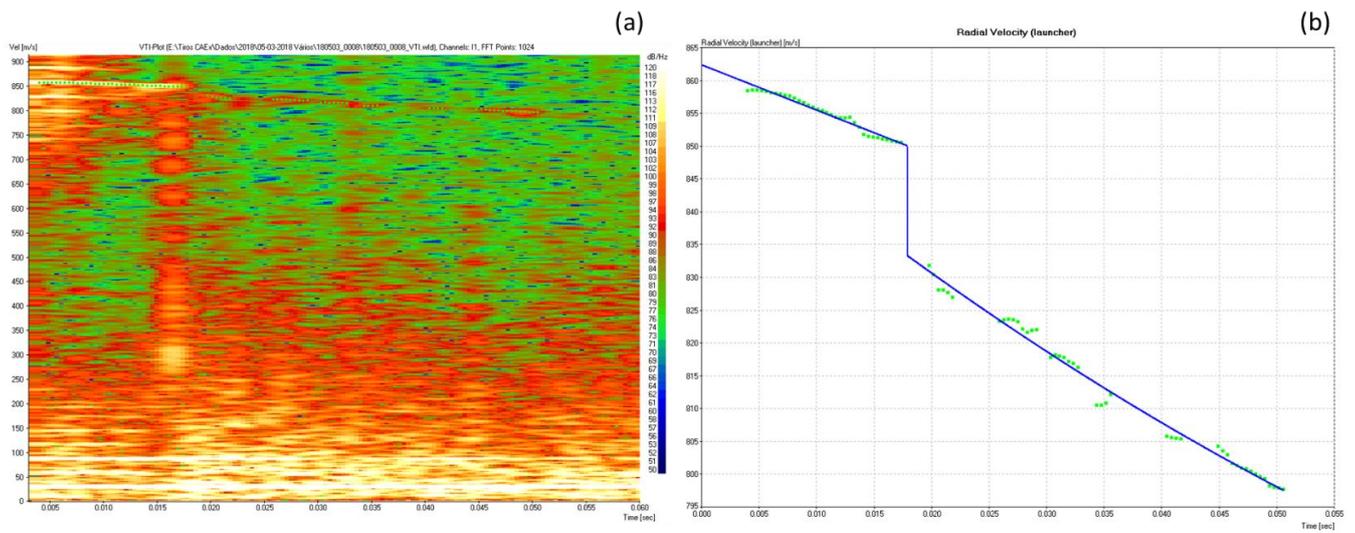
Figure 2. Doppler radar results (**a**) raw and (**b**) processed data.

Measurement of the values of impact and exit velocities allows the estimation of the energy absorbed by the target material [24]. Figure 3 shows the average impact energy absorption and corresponding standard deviation of all tested conditions.
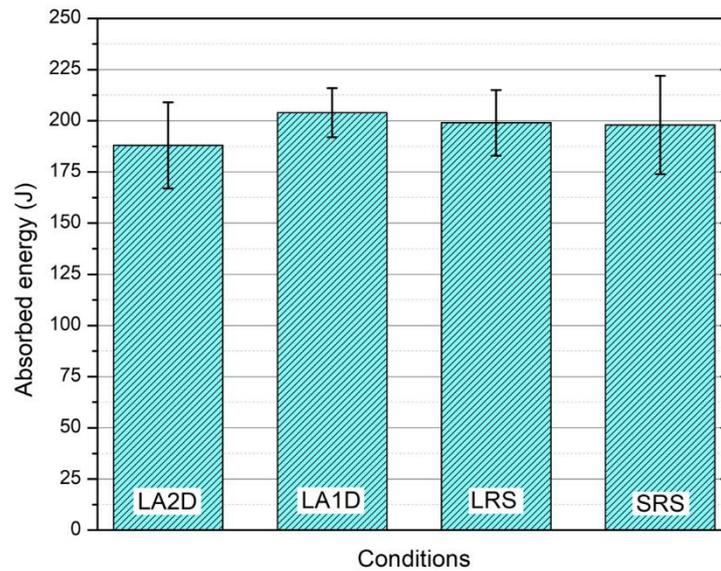

Figure 3. Energy absorption of studied configurations.

The highest energy absorption was calculated as 222 J for the SRS condition, while the lowest value of 167 J was obtained for the LA2D condition. Considering these results, the average values associated with the standard deviations suggests that the absorbed energy of all conditions was virtually the same. Further statistical analysis is required to validate this data. Table 2 presents the ANOVA parameters.

Table 2. Analysis of variance parameters.

| Variation Causes	DF	SS	MS	F	Fc
Treatments	3	537.7	179.2	0.51	3.49
Residue	12	4219.3	351.6	-	-
Total	15	4756.9	-	-	-

DF = degree of freedom; SS = sum square; MS = mean square.

Comparing calculated F with the tabulated one, Fc, one may verify that F is lowest than Fc. This indicates, with 95% confidence, that energy absorption is statistically the same for all investigated condition. Additionally, Tukey's honest test was performed to compare the mean values quantitatively two-by-two. Calculated honest difference was 39.4 J, and differences above that should be considered significant. Table 3 summarizes the Tukey's analysis.

Table 3. Tukey's honest test result.

Condition	LA2D	LA1D	LRS	SRS
LA2D	0	16	11	10
LA1D	16	0	5	6
LRS	11	5	0	1
SRS	10	6	1	0

As none of the differences were found to be higher than the calculated honest difference, it is further confirmed that the target material arrangement does not influence the energy absorption of the ballistic impact. On the other hand, when the physical or structural integrity of the composites plates is taken into consideration, there is a clear difference, as shown in Figure 4a–d.
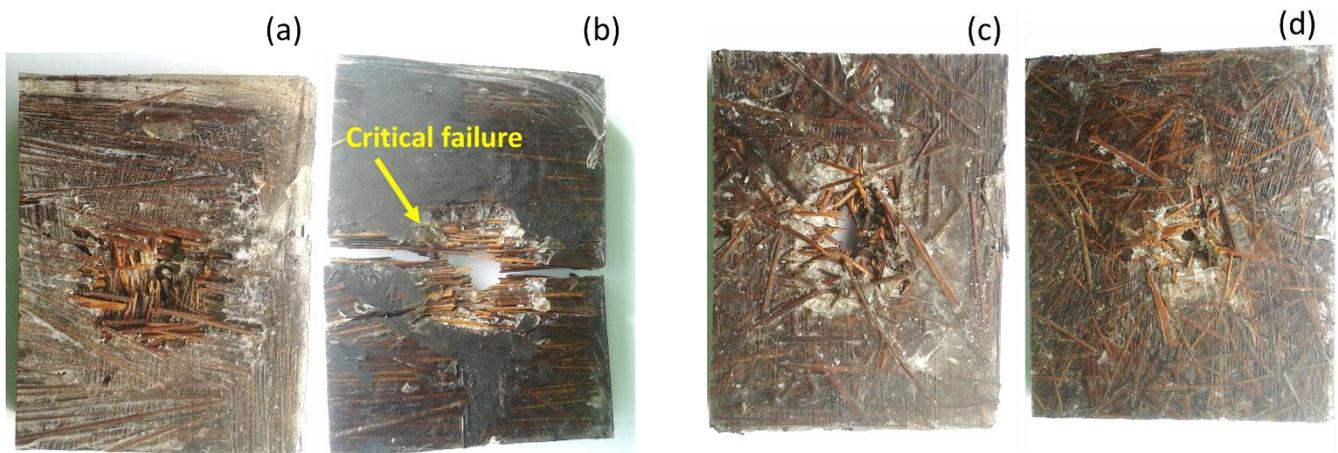
Figure 4. Macroscopic aspect of failure after ballistic impact (**a**) LA2D; (**b**) LA1D; (**c**) LRS, and (**d**) SRS.

For the LA1D condition, Figure 4b, a critical crack propagates from the impact point towards the edge of the composite following the same axial direction of the fiber arrangement. No other condition, Figure 4a,c,d, displayed a similar failure. For these conditions, the fiber reinforcement arrangement was able to block the path of the crack, preventing a catastrophic failure of the system. This analysis suggests that fiber reinforcement arrangement in an aligned and axial configuration may not be suitable for ballistic applications. None of the conditions were able to stop the projectile impact, they only absorbed part of its kinetic energy, as discussed in Figure 2b. To investigate a scenario where the projectile would be stopped by the target, the limit velocity was calculated for each condition [24]. Figure 5 presents these results.

The limit velocity of all investigated conditions was in the same magnitude, around 200 m/s. A behavior like that was expected once the amount of impact energy absorbed by these composites was shown to be statistically the same, as seen in Table 2. Furthermore, this ballistic parameter was previously determined as 212 ± 23 m/s for Kevlar^{TM}, style 745, produced with fill and warp yarns of Kevlar$^{®}$ K-29 3000 denier, with the same thickness of the investigated piassava/epoxy composites [25]. This is an important result that

helps validate the epoxy/piassava composite as a ballistic impact resistance material once Kevlar^{TM} is commonly praised as efficient against high energy ammunition.
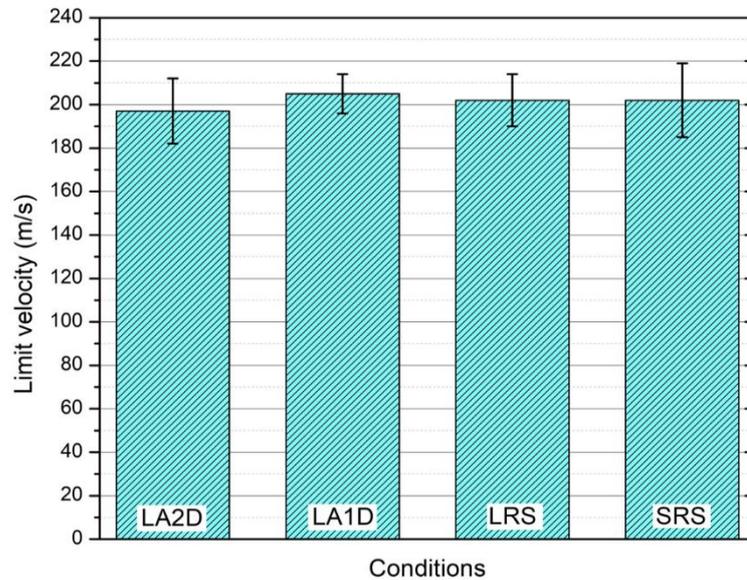

Figure 5. Limit velocity of different investigated conditions.

3.2. MAS Application of Epoxy/Piassava Composites

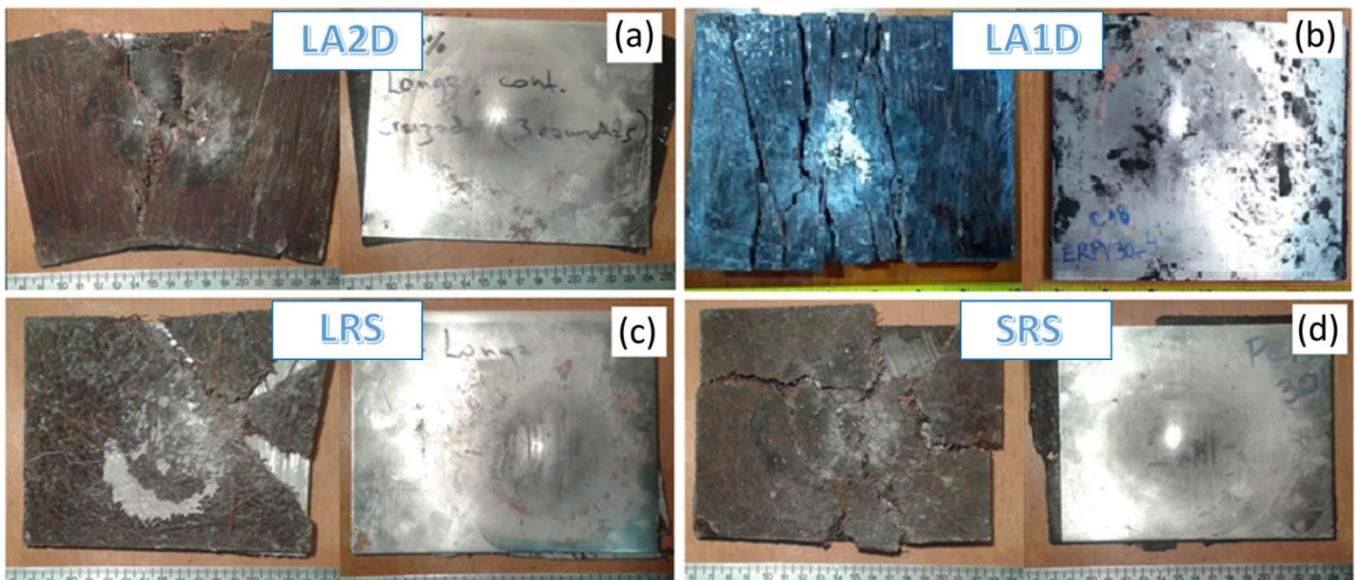Figure 6a–d shows the front and rear face of each of the conditions analyzed.

Figure 6. Front and back face of MASs (**a**) LA2D; (**b**) LA1D; (**c**) LRS, and (**d**) SRS.

In the front face, the hexagonal ceramic tile first layer was destroyed, despite some white fragments remaining attached to the composite second layer. On the other hand, in the back face, none of the conditions were completed perforated. Instead, a protuberance associated with the plastic deformation of the aluminum alloy caused by the impact is observable. As for the composite intermediate layer, the expected physical integrity, especially for the multiaxial reinforcement arranged composites presented in Figure 4, did not maintain. However, it is clear there was a shift in fracture mode comparing the LA1D, where several cracks in the same direction of the fiber alignment are observed, to

the other three conditions. The difficulty of the composite in maintaining its integrity may be associated with the configuration of the MAS itself. The system, as assembled, aims to "trap" the shock wave generated in the ballistic impact [26]. To minimize its amplitude, various transmission and reflection surfaces are required in the MAS. As the composite has a lower shock impedance than ceramic, this wave is reflected, returning with tensile nature, which results in the spalling of the first layer. Analogously, the third layer has a greater shock impedance than the composite. This will make the nature of the reflected wave remain compressive, trapping the shock wave in the intermediate layer [27].

Figure 7 presents the indentation depth of the investigated MAS. Perforation and Back-face signature test, in accordance with the NIJ standard [21], states that for a material to be considered effective against high energy ammunition, it must not be perforated nor display a back-face indentation depth greater than 44 mm.
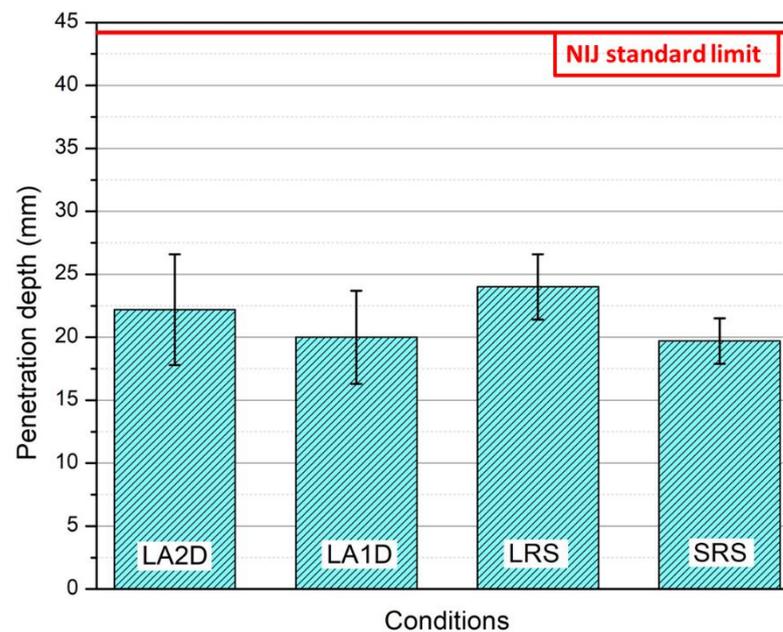

Figure 7. Penetration depth measurements for investigated MASs.

The results obtained are way below the limit required by the NIJ standard. Ultimately, all investigated conditions are characterized as efficient ballistic protection, although the issue regarding the structural integrity of the intermediate layer still needs to be addressed.

3.3. Mechanisms of Energy Absorption

Several failure mechanisms are commonly reported for fiber reinforced composite materials [28–31]. Effects such as pullout, fiber bridging, delamination, and rupture of fiber are some of the mechanisms presented to describe the failure of these materials. Figure 8 exhibits some of the micromechanisms observed in the piassava/epoxy composites after ballistic impact in both MAS and individual performance conditions.
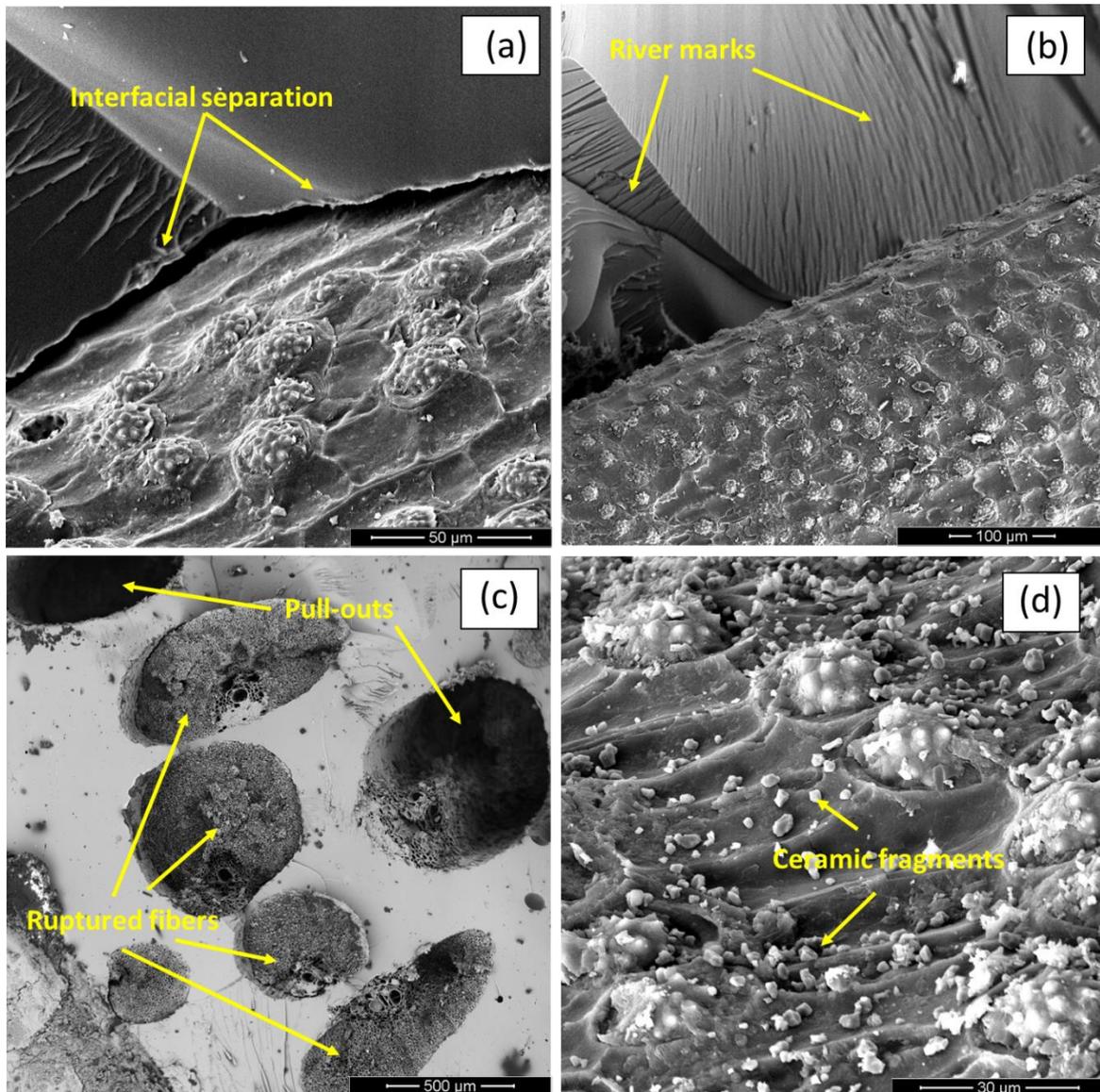
Figure 8. SEM of failure micro mechanisms associated with ballistic impact (**a**) fiber/matrix separation; (**b**) brittle characteristic of epoxy matrix; (**c**) pullout and fiber breakage, and (**d**) ceramic incrustation on surface of piassava fiber.

In Figure 8a, the interfacial separation between fiber and matrix is shown. In this figure, an arrow highlights the crack that propagates through the interfacial separation between the epoxy matrix (above) and piassava fiber (below). In Figure 8b, the fragile characteristic of the matrix is evident by the presence of "river marks", which are associated with the brittleness of the epoxy. In Figure 8c, one may notice both the pullout and rupture of fibers. All these mechanisms were observed and disclosed as responsible for absorbing part of the energy of the ballistic impact in both ballistic configuration (MAS and individual performance). Finally, in Figure 8d the mechanical encrustation of ceramic fragments in the piassava fiber surface is visible. This phenomenon, observed only for the MAS test configuration, is associated with electrostatic and Van der Waals attraction forces [32]. This characteristic is essential for an intermediate layer in MAS, as the cloud of ceramic fragments generated after the ballistic impact could be considered as harmful as the projectile itself.

4. Discussion

The amount of energy absorbed and limit velocity, Figures 3 and 5 respectively, were shown to be independent of the fiber arrangement in the piassava/epoxy composite. The statistical analysis presented in Tables 2 and 3 validate these results. In addition to that, micro mechanisms associated with the failure of these composites revealed that similar phenomena occur in all investigated conditions, which corroborates with the assumption of composite design independence for energy absorption under dynamic conditions. Interfacial separation, brittleness of the matrix, and rupture of fibers as well as pullout of fibers, Figure 8a–c, were observed in all composites designed for this investigation. Opposite behavior is observable when the volume fraction of fiber reinforcement is varied [33–35]. A higher amount of reinforcement ultimately leads to a higher efficiency in energy absorption. However, an interesting behavior is found when comparing the macroscopic aspect of failure after ballistic impact, Figure 4. The uniaxial configuration, LA1D, exhibited a critical failure where there was a crack propagation from the impact point towards the edge of the composite following the same direction of fiber reinforcement. Multiaxial configurations, i.e., LA2D, LRS, and SRS, did not behave like that. There was a complete perforation of the target; however, the physical integrity of the system was maintained. Therefore, uniaxial fiber reinforcement configuration may not be suitable for ballistic impact application despite several studies that considered this kind of configuration [5,12].

For the MAS investigation, the areal density can be considered an important parameter to appraise any layered armor system [36]. The areal density of the proposed MAS system can be estimated by the equation:

$$Areal\ density\ \left(\text{kg/m}^2\right) = \sum_{i=1}^{n} t_i \rho_i \tag{1}$$

where t and ρ are thickness and density of the individual layered structures. The calculated value of 58.66 kg/m^2 can be compared with the value of 62.06 kg/m^2 obtained for a MAS structure containing Kevlar$^{\text{TM}}$ as intermediate layer [37]. As light weight armor is considered essential for saving energy and increasing mobility, the lowest possible areal density that can resist the threat is desirable in any armor design studies [38]. This suggests a superior performance of the epoxy/composite against the Kevlar$^{\text{TM}}$ as intermediate layer in MAS. Indeed, the areal density of the epoxy/piassava MAS configuration is lower than several steel and laminated composites for armor applications, which exhibit values in the range of 51–92 kg/m^2 [39]. Indeed, any of investigated conditions were perforated, and all of them displayed an indentation depth lower than 44 mm, Figures 6 and 7, respectively. Therefore, all of them can be considered efficient materials against high-energy ballistic impact. In addition to that, SEM (Figure 8d) revealed the composite capability of absorbing ceramic fragments generated in the ballistic impact. Electrostatic and Van der Waals attraction forces are claimed as driven force of this phenomenon [32]. Nevertheless, the physical integrity of the MAS system still requires investigation, unlike that observed for the individual performance of composites (Figure 4), in which all conditions exhibited fractures that could be seen as catastrophic in a multiple shot condition.

5. Conclusions

Four different fiber reinforcement arrangements of piassava natural fibers were used in epoxy matrix composite. Axial and biaxial reinforcement configuration as well as short and long scattered fibers were considered. These composites were analyzed under ballistic impact conditions to measure individual composite performance and MAS efficiency.

- For individual performance of the composites, the energy absorbed by them ranged from 167 to 222 J. Furthermore, it was statistically proven that the fiber arrangement did not influence the energy absorption. However, in terms of structural integrity, conditions that provided reinforcement in more than one direction rise as most promising. The limit velocity parameter was calculated for all conditions and presented values of the same magnitude as traditional materials.

- For the efficiency of MAS, all conditions were considered efficient according to NIJ standard as none of the systems were perforated and indentation depths were below the limit of 44 mm. On the other hand, contrary to what was expected, the structural integrity was an issue for all conditions. This was associated with the design of MAS itself, alternating materials with high shock impedance with ones with low impedance.
- Failure of micromechanisms, such as fiber breakage, fiber/matrix delamination, and pullout, were verified as the main failure mechanisms in these materials. Furthermore, it was verified that the absorption of the cloud of fragments was generated in the ballistic impact by the piassava fiber through mechanical incrustation.

Conflicts of Interest: The authors declare no conflict of interest.

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