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Article

Mechanical characterization of polymeric composites reinforced with para-aramid after thermal exposure

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Abstract: In this study, heat treatments were applied to samples of para-aramid fiber-reinforced epoxy at two different temperatures (250 and 500 ℃) for five (5) min. The effects on mass and mechanical properties were investigated by tensile tests according to ASTM-D3039-00 and compared with untreated samples. In addition, the fiber/matrix interface was verified by scanning electron microscopy (SEM). At both exposure temperatures, a mass loss of 2.7% and 8.9% was observed for samples submitted to 250 ℃ and 500 ℃, respectively. However, the mechanical tensile strength in both cases was improved, being more significant in samples exposed to a temperature of 500 ℃, with an increase of more than 30% in tensile strength. Although the presence of delamination in the samples was not verified in the visual analysis, in the micrographs studied, it was observed that the decomposition of the fibers and the matrix causes localized delamination between the weft, the warp, and the matrix of the composites after the test.

Keywords: para-aramid; polymeric composite; thermal exposure; mechanical integrity

1. Introduction

Most materials engineers' real goal is to create new compounds from existing materials. Because of this, substantial research efforts have been made to improve mechanical, electrical, and thermal properties to adapt to a specific application.

The fiber-reinforced polymer, FRP, is the most attractive among composites because it has versatility in applications ranging from mechanical and automotive to structural components. The number of researchers worldwide who develop, improve, and characterize composite materials has grown due to the improvements brought about by using composite materials in the application of these materials [1].

FRP uses fibrous materials to improve the mechanical properties of the polymer matrix. These reinforcements are presented as filaments or reinforcement fibers that have greater strength and rigidity when compared to the matrix, thus making them more resistant but relatively more fragile. The orientation of the fibers in the matrix and the volumetric relationship between the matrix and the reinforcement mainly govern the final mechanical properties. Matrix reinforcement occurs when the final properties of the composite exhibit additional strength or elasticity related to its elasticity and matrix strength [2].

In recent decades, conventionally manufactured fibers have been used as reinforcement in composite materials. Predominantly, carbon, para-aramid, and glass top the list of materials of choice due to high-quality mechanical properties such as

impact resistance and flexibility, corrosion resistance, low density, high modulus, moisture resistance, and ease of fabrication when compared to metal materials [3,4].

Para-aramid is an artificial organic fiber from the family of aromatic polymeric amides. Absolute aromatic polyamides (aramids) stand out from other artificial fibers due to their exceptional qualities and remarkable chemical composition [5,6].

Studies show that para-aramid fiber has a morphological structure of the core-shell type, characterized by a dense and highly oriented core surrounded by a less ordered outer layer. This structural architecture results from the solution-spinning process and the molecular orientation induced during fiber solidification. Such a configuration gives the material superior mechanical properties, such as high tensile strength, high modulus of elasticity, and excellent thermal stability. The interaction between the core and the skin contributes to a more efficient distribution of the applied stresses, promoting greater toughness and impact resistance. Thus, the presence of the core-skin structure represents a determining factor in improving the performance of para-aramid fibers in high-performance engineering applications, such as structural composites and ballistic protection systems [7,8].

In addition, it offers an excellent strength-to-weight ratio, improved impact qualities, thermal stability (160–200 ℃), and wear and stab resistance. Compared with other artificial fibers, it has long elongation and high tensile strength and modulus [9–11]. Due to their molecular structure, they exhibit these enhanced characteristics [12,13] The advantages over other synthetic fibers make it a good candidate for reinforcement in composites.

In this context, polymer matrix composites reinforced with high-performance fibers have been widely studied due to their versatility and high mechanical performance. Among these materials, epoxy matrix-based composites reinforced with aramid fibers stand out for their exceptional impact resistance, high strength-to-weight ratio, and remarkable thermal stability compared to conventional polymers [14]. Para-aramid exhibits 5%–10% higher mechanical properties than other synthetic fibers and maintains these properties at high temperatures, as aramid polymers are excellent heat and flame resistant. These characteristics make them applicable in strategic sectors, such as aerospace, automotive, and defense, in addition to their promising use in personal protective equipment (PPE), especially for professionals exposed to high-temperature environments, such as firefighters and workers in the metallurgical industry [15].

Considering the mechanical and thermal properties of para-aramid fibers in epoxy matrix composites, it is essential to enhance the understanding of the stability of these materials under high-temperature conditions. The primary challenge in developing and applying these composites in personal protective equipment is to ensure that their mechanical properties are maintained even after exposure to short heat waves, a common occurrence in many demanding performance applications, such as firefighting and industrial metallurgical operations. Thus, the mechanical resistance of these composites after thermal exposure is a relevant factor.

The thermal stability of a composite material is defined as its ability to maintain mechanical properties, such as strength and stiffness, within acceptable limits when subjected to a specific temperature range. This characteristic is particularly relevant

in applications where the material is exposed to thermally harsh environments, such as in the aerospace, automotive, and defense sectors. Thermal stability depends heavily on the nature of the constituent phases of the composite, the quality of the matrix-reinforcement interface, and the structural integrity of the system as a whole. An adequately designed, thermally stable composite can withstand significant temperature variations without degrading its physical and mechanical properties. Therefore, accurate evaluation of thermal stability is critical for the selection and development of composite materials in critical applications [16].

The thermal resistance of these composites is a crucial factor for their application in extreme conditions. Previous studies indicate that thermal degradation of reinforced polymeric composites can occur by processes such as oxidative degradation and pyrolysis, leading to the reduction of essential mechanical properties [17–19]. At moderate temperatures (about 250 °C), the epoxy matrix can undergo residual crosslinking reactions, increasing stiffness but compromising toughness. At higher temperatures (above 500 °C), significant degradation of the matrix can occur, exposing the fibers and drastically reducing the structural integrity of the material [20–22]

Recent studies show that the thermal degradation of the epoxy matrix can be partially mitigated by using chemical modifications or ceramic fillers, increasing its resistance to high temperatures [23,24]. However, the overall strength of the composite depends not only on the matrix but also on the thermal stability of the Kevlar fibers. Although aramid fibers have excellent resistance to moderate temperatures, their progressive degradation above 450 °C compromises the mechanical properties of the material, limiting its applicability in prolonged exposures to extreme heat [25].

When a composite specimen is exposed to higher temperatures in a restricted environment, thermogravimetric analysis (TGA) is the most frequently used method to evaluate stability or thermal degradation by loss of mass of the composite specimen [26].

Naveen et al. [7] evaluated the thermal degradation of epoxy matrix hybrid composites reinforced with different types of structural fibers, and it was observed that thermal stability tends to decrease with the introduction of additional fibrous phases, regardless of their origin. However, there was an improvement in the damping factor with the increase in fiber content, indicating a greater capacity of the material to dissipate mechanical energy. On the other hand, both the loss modulus and the storage modulus showed a reduction proportional to the increase in the volumetric fraction of added fibers, a behavior attributed to the change in the overall stiffness of the composite and to the modification of interfacial interactions [27].

Despite these changes, it was found that the inclusion of hybrid reinforcements can, in certain formulations, raise the temperature at the beginning of thermal deterioration, which shows a synergistic effect between the different types of fibers on the thermal resistance of the system as a whole. At the same time, this combination of reinforcements can reduce the degree of crystallization of the polymeric matrix, possibly due to the interference in the molecular arrangement promoted by the presence of fibers, which directly impacts the final morphology of the composite. These effects must be carefully considered when designing advanced

composite materials since they influence thermal stability and mechanical performance [27].

While there is a growing need for high-temperature-resistant materials, a notable dearth of studies exists on the thermal degradation of polymeric composites reinforced with para-aramid fibers. This gap limits the complete understanding of the behavior of these materials in extreme heat environments, highlighting the urgency of more research in this area.

Given this, this study investigates the behavior of epoxy matrix composites reinforced with Kevlar fibers subjected to thermal stresses of 250 °C and 500 °C, comparing their mechanical and structural properties with those of samples not exposed to heat. The primary objective was to investigate the impact of thermal exposure on these properties, utilizing tensile tests for mechanical evaluation, TGA and DSC analyses for material degradation, and scanning electron microscopy (SEM) to assess the integrity of the fiber/matrix interfaces. The evaluation of these thermal effects is crucial for understanding the degradation mechanisms and contributing to the development of more effective and safer personal protective equipment (PPE). This evaluation enables the proposal of strategies to optimize the thermal performance of these composites and expand their applicability in high-thermal-risk environments.

2. Materials and methods

The laminate was made with 10 layers of para-aramid plain fabric (K29 Kevlar® DuPont, 200 g/m^2) impregnated with 2001 Epoxy Resin and 3154 Hardener (Redelease) with viscosities of 100–140 P and 2 P, respectively. Consistency in material properties was maintained by using the same resin with a content of 40% in all laminates. The impregnation was performed manually with a roller in a metal apparatus covered with release agents. The stack was heated to 100 °C at a rate of 5 °C/min for 5 h in a parallel plate autoclave.

A preliminary TGA test was performed with a NETZSCH STA 449F3 Jupiter analyzer in the range of 25 °C to 550 °C at a 10 °C/min rate under an N_2 atmosphere. It is made of laminated para-aramid fibers and composites.

The specimens laminated for the treatment and subsequent mechanical test were cut in the warp direction, with a length of 250 mm, a width of 25 mm, and a thickness of 2.5 mm [28].

The study lots were separated according to the thermal exposure temperatures, thus being lot 25 °C, lot 250 °C and lot 500 °C, each composed of 15 specimens obtained from plates laminated under the conditions previously specified.

The exposure protocol followed the EDG 1800 muffle heating routine. After stabilizing the exposure temperature, the specimens were inserted into the heating chamber and kept at a constant temperature for 5 min.

The samples were measured and weighed before and after heat treatment to assess dimensional and mass stability during exposure to high temperatures. Each batch was subjected to mechanical tests performed on an Instron universal testing machine model 5900-5594 following the protocol described in the ASTM-D3039-00 standard [28].

The tensile tests were performed using Body-over-Wedge grips, the loading was controlled at a rate of 2 mm/min, and the end of the test was determined with the reduction of tension to levels below 40% of the maximum tension reached in the test.

3. Results and discussion

The results of the preliminary analysis performed by thermogravimetry on the fibers and the laminate, **Figure 1**, made it possible to delimit the critical temperatures of thermal exposure of the laminates.

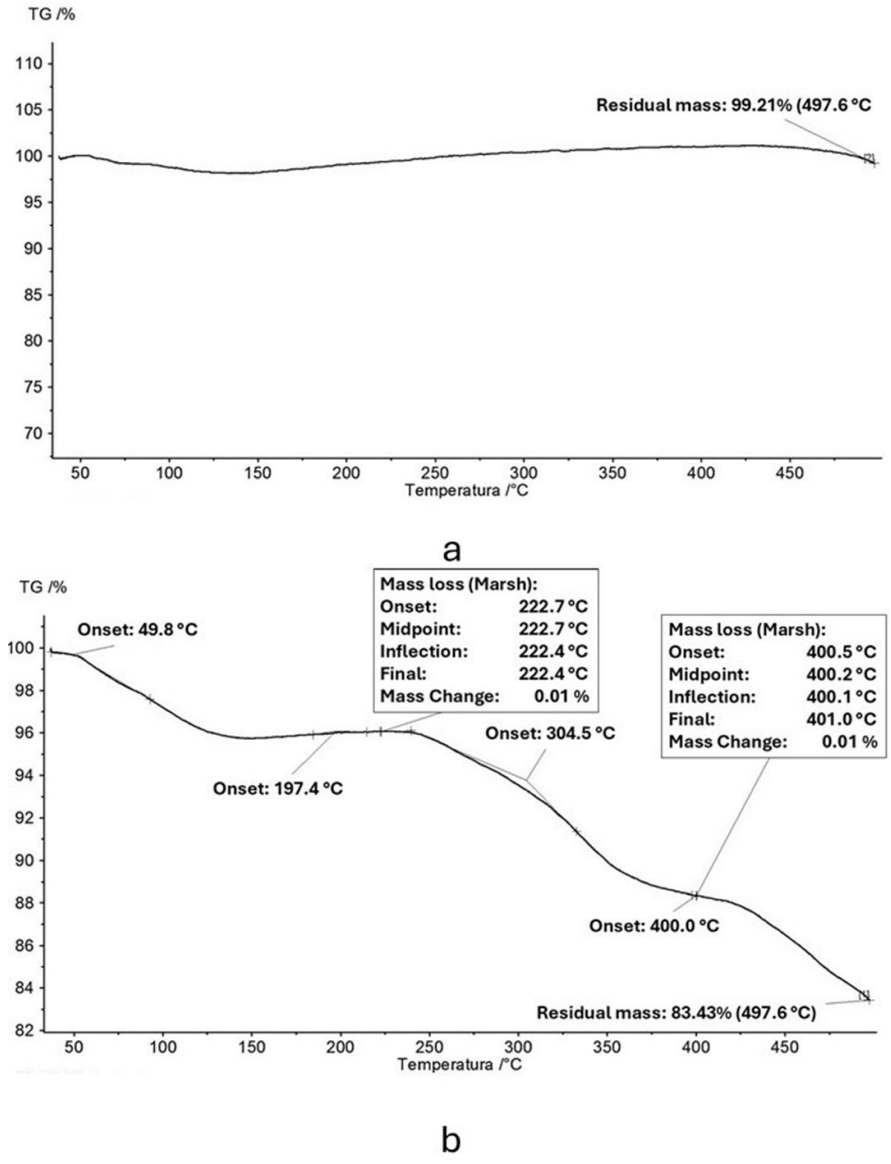

Figure 1. Thermogravimetric analysis of **(a)** para-aramid fibers and **(b)** laminated composite.

It is observed in **Figure 1a** that the para-aramid fibers do not suffer significant degradation when exposed to the study temperatures, with a loss of less than 1%, which proves that the limiting factor for the maintenance of the properties of the polymeric composites reinforced by para-aramid fibers is the resistance of the polymeric matrices [29].

This fact was proven by the assay carried out on the sample laminated with epoxy resin, **Figure 1b**; it was observed that at milder temperatures, around 100 ℃, the release of the water molecules adsorbed on the para-aramid fibers occurs, and the weak intermolecular bonds of the polymer break. According to the literature, two events occur at temperatures between 250 ℃ and 350 ℃, which lead to a reduction in mass, successively due to the evolution of water through vaporization and the degradation of the crosslinking bonds of the epoxy matrix [7,30]. At temperatures of 400 ℃ mass loss is observed due to the release of volatile compounds resulting from the degradation of the resin [30] in sequence; with the reach of temperatures above 500 ℃, the degradation of para-aramid fibers begins, which is believed to be the breakdown of the main polymer chains [7].

After controlled thermal exposure, a visual analysis of the samples was performed (**Figure 2**), and it was verified that with this procedure, the samples remained intact and free of delaminations, which indicates that there was no sudden release of gases from the degradation of the resin and para-aramid. The water is released up to 900 ℃, with the maximum release of water vapor at temperatures of 200 ℃ and 500 ℃ [30].

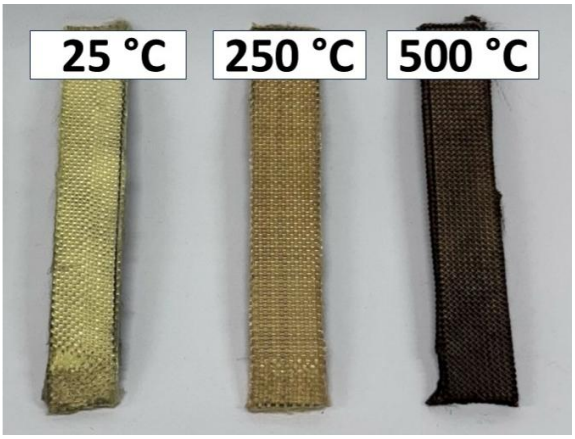

Figure 2. Image of the samples after thermal exposure.

Thermal degradation of a para-aramid composite initially occurs between 100 and 200 ℃ due to physically weaker water molecules on the surface of the composite and dehydration induced by the group of secondary alcohols. From 300 ℃, the second stage of thermal degradation is mainly due to the degradation of the epoxy matrix of the composite [7]. Volatile substances from resin heating, such as methane, carbon monoxide, and hydrogen, are released at 400 ℃ [30]. Finally, the last stage takes place at 500 ℃, in which the hydrogen bonds of the material break, leading to the complete decomposition of the para-aramid fibers; from this temperature, the carbonization of the fibers also occurs [7]. The thermal exposure time did not allow water vapors and organic volatiles from the interior of the laminates to be formed and released abruptly, thus ensuring the integrity of the samples for mechanical testing.

During thermal exposure at 500 ℃, smoke and a strong burning smell were released, which was not observed in samples treated at 250 ℃. According to Chinnasamy et al. [1], from 300 ℃ onwards, the compound shows thermal

degradation and total mass loss, primarily due to the resin between the layers. While between temperatures of 550 and 650 °C, the mass loss of the material is greater.

Like any polymer composite, para-aramid carbonization is associated with mass loss. However, the time of exposure to high temperatures is a more important factor than the increase in temperature [31].

When analyzing the mass variation of the samples exposed to 250 °C and 500 °C (**Figure 3**), it was possible to verify a mass loss of 2.7% and 8.985%, respectively. These variations were low due to the short period of exposure of the sample to the study temperature.

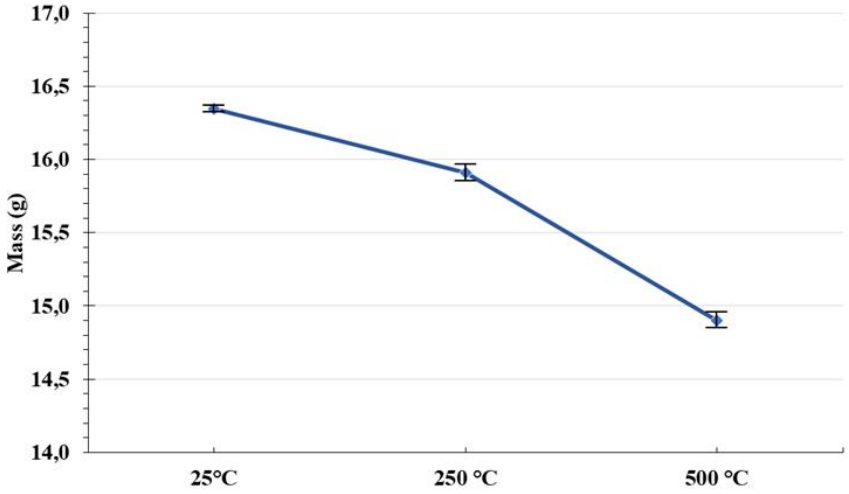

Figure 3. Relationship between mass loss and exposure temperature.

Samples treated at 500 °C after cooling showed a darker color than samples at room temperature. Traces of sulfuric acid from the manufacture of the fiber accelerate the hydrolytic and oxidative degradation of the para-aramid fibers and cause the chains to break, resulting in early carbonization, and with this, the fibers will darken at temperatures above 75 °C [32].

According to Chinnasamy et al. [1], polymeric composite laminates reinforced with para-aramid subjected to controlled heating have better properties and higher glass transition temperature, maintaining the material's thermal stability. According to Yue et al. [25], when heating para-aramid fibers for periods of two, four, six, and eight (2, 4, 6, and 8) h at temperatures of 100, 200, and 300 °C, the increase in heating temperature causes a reduction in mechanical strength. However, Young's modulus remained constant in the different tests. As already discussed, the exposure time is more relevant than the temperature, which explains the increased resistance in the para-aramid composites studied.

This is also pointed out in the studies of Hindeleh and Abdo [32], who reported the improvement in the mechanical properties of the composite after exposure to 400 °C for 15 min. This is correlated with increased crystallinity and increased distribution of microparacrystallites in the para-aramid material.

The 5-min thermal exposure applied in this study proved insufficient to compromise the integrity of the para-aramid fibers, which were primarily responsible for maintaining the mechanical strength of the composites. Although the fiber-matrix interface and the polymer resin suffered visible damage, the para-aramid fibers

remained intact, ensuring a notable increase in the stress supported by the composites. However, even with the improved tensile mechanical properties, the physical integrity and shape of the material were not maintained, which makes their use in structural applications unfeasible. Despite this, these composites show promise for personal protective equipment, where the ability to absorb and dissipate energy is crucial, even in the presence of deformation. This finding is crucial because, in contrast to other studies that report degradation of the strength of aramid-polymer composites under prolonged exposure to high temperatures, our results highlight the intrinsic resistance of para-aramid fibers to short heat spikes, highlighting the relevance of this research for the development of heat-resistant materials.

The mechanically tested samples were not brought to total failure, and the maximum peak resistance was considered the limit of the study of the material. After the removal of the samples from the test equipment, the formation of strains in the midline and the delamination of the outermost layers of fibers of the samples submitted to 500 ℃ were observed, which was not observed in the samples treated at room temperature and 250 ℃. Indicating the increased fragility of the treated samples at this temperature, evidenced by the increase in Young's modulus, **Figure 4**.

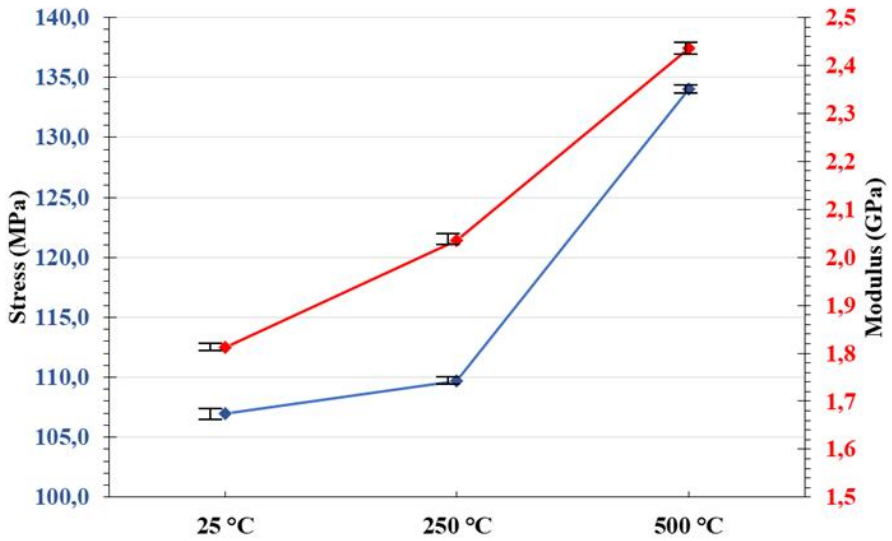

Figure 4. Relationship between stress (blue curve), Young's modulus (red curve), and exposure temperature.

It is not possible to verify a clear relationship of proportionality between the elasticity and the stress analyzed in the samples. This is due to the fact that the material presents a nonlinear and orthogonal behavior in its elasticity curve.

The darkened coloration and increased mechanical strength of the composite indicate that the heating of para-aramid leads to the carbonization of the polymer fibers, resulting in higher modulus and specific final strengths since there is an increase in properties and a reduction in mass. It was possible to notice that the higher the exposure temperature of the samples, the higher the resistance and Young's modulus, especially in the samples at 500 ℃, which showed an increase of

25.3% in mechanical strength and 34.4% in Young's modulus. This addition may be associated with the charring of more widespread para-aramid fibers in these samples, resulting in changes in material properties such as increased material stiffness.

Figure 5 shows the cross-sectional incidences of the samples tested in traction. It is observed that in the samples tested at room temperature (a) and after exposure to 250 °C (b), there are interfaces between weft, warp, and matrix with good adhesion absent of localized detachments, while in the sample tested after exposure to 500 °C, the detachment between the fibers of the weft and the warp is observed.

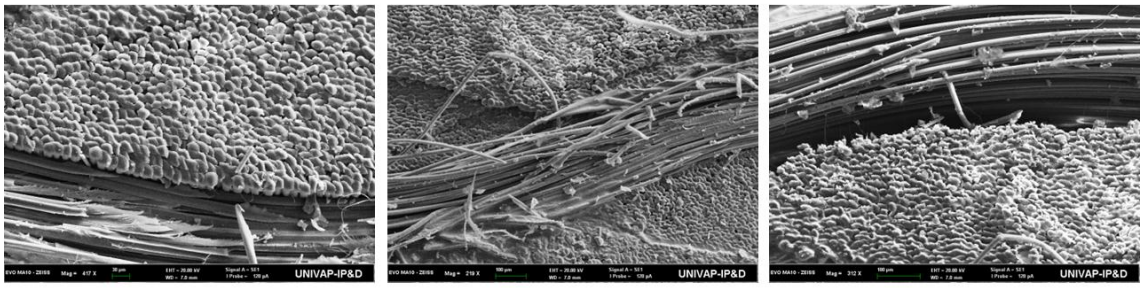

Figure 5. Micrograph of the cross-sectional views of samples tested at room temperature **(a)** after exposure to 250 °C **(b)** and 500 °C **(c)**.

In addition, as the treatment temperature is increased, the fiber's fracture characteristics become ductile to brittle, which corroborates the data already discussed from Young's modulus.

4. Conclusion

The research presented investigated the impact of thermal exposure, for a limited period of 5 min, on para-aramid fiber and epoxy matrix composites subjected to temperatures of 250 °C and 500 °C. The results revealed that, despite the degradation of the polymer matrix and the fiber-matrix interface, the para-aramid fibers demonstrated remarkable resilience to short-term thermal exposure. The thermogravimetric analysis (TGA) confirmed that the para-aramid fibers did not suffer significant degradation at the temperatures studied, with a mass loss of less than 1%. This finding is crucial as it solidifies the understanding that the limitation of the strength of para-aramid-reinforced composites under thermal stress lies primarily in the fragility of the polymer matrix.

Despite the mass loss observed in the composites (2.7% at 250 °C and 8.9% at 500 °C), attributed to resin degradation and the release of volatiles, the mechanical tensile properties were significantly improved, especially in the samples exposed to 500 °C, where there was an increase of more than 30% in tensile strength and 34.4% in Young's modulus. This increase in strength and stiffness is attributed to the carbonization of para-aramid fibers, a phenomenon that alters the material's structure while enhancing its ability to withstand tension. The carbonization of the fibers, evidenced by the darkening of the treated samples at 500 °C, suggests a molecular restructuring that contributes to the increase of mechanical strength.

Although the material exhibited improved mechanical tensile properties,

exposure to 500 °C led to a loss of physical integrity and shape of the samples, characterized by the formation of deformations in the midline and delamination of the outermost fiber layers following mechanical tests. This suggests that while para-aramid fibers maintain their structural integrity and contribute to increased strength, degradation of the matrix and fiber-matrix interface compromises the composite's ability to maintain its shape and structural integrity under thermal stress. The loss of formal integrity renders these composites unsuitable for use as structural materials in applications that require the preservation of geometry and dimensional stability.

In contrast to the structural application, the results indicate that these composites are promising for personal protective equipment (PPE) applications. The ability to absorb and dissipate energy under high-temperature peaks, even with some deformation, is a valuable feature of PPE, especially for professionals exposed to high thermal risk environments, such as firefighters and workers in the metallurgical industry. The improved tensile strength and toughness of para-aramid fibers, even after thermal exposure, make them suitable for protecting against impacts and high stresses in emergency scenarios.

It is crucial to note that the duration of thermal exposure is a more significant factor than the magnitude of the temperature itself, as evidenced by the maintenance of mechanical properties in this short-term study. Previous work has shown that prolonged exposure to elevated temperatures results in a significant loss or reduction of mechanical strength due to the progressive degradation of aramid fibers above 450 °C and matrix degradation. This underscores the importance of this study in demonstrating the material's resistance to short-lived heat waves, a relevant scenario in several risk situations.

Despite the importance of the theme, the number of studies focused on the degradation of polymeric composites reinforced with para-aramid fibers by thermal exposure is scarce. This research gap limits the development of more efficient and safer materials for applications in high-temperature environments. A detailed understanding of the mechanisms of thermal degradation and the interactions between fibers and the matrix under various conditions is essential for optimizing the performance of these materials.

In summary, this study makes a significant contribution to the understanding of the behavior of para-aramid composites under conditions of short-term thermal exposure. Although the formal integrity of the material is compromised, the maintenance and increase of mechanical tensile strength, driven by the resilience of para-aramid fibers, opens up new perspectives for its application in personal protective equipment. Continued research in this area is crucial for filling the existing gap and advancing the development of composite materials with improved thermal performance for various critical applications.

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have read and agreed to the published version of the manuscript.

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