Manufacture and mechanical characterization of composite material of carbon fiber with matrix of PEEK

Manufactura y caracterización mecánica de material compuesto de fibra de carbon con matriz termoplástica PEEK

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DOI: https://doi.org/10.22517/23447214.24826
Scientific and technological research article

***Abstract*— In this study, a low-cost simple manufacturing process for a composite material of thermoplastic matrix PolyEtherEtherKetone (PEEK) and carbon fiber (CF) was developed. The composite material CF/PEEK was mechanically evaluated by three-point bending and tension tests, obtaining the elastic modulus and the maximum tensile stress respectively. Also, threads were made in the composite material and the strength of threads machined on the composite material was evaluated with tension tests. A comparison of the composite material CF/PEEK and the composite material of carbon fiber and epoxy resin (CF/EP) was performed, the elastic modules, peak tensile stresses and the strength of threads were compared. It was found that it was possible to produce the CF/PEEK composite at low cost by hot molding. The elastic modulus and tensile strength of the CF/PEEK were lower than those obtained in the CF/EP. However, the performance of the thread in tension was better for CF/PEEK compared to CF/EP.**

***Index Terms*— Carbon Fiber, Epoxy Resin Manufacture, PEEK, Mechanical Characterization, Thread.**

***Resumen*—En este estudio se desarrolló un proceso de manufactura sencillo, de bajo costo para producir un material compuesto de matriz termoplástica PolyEtherEtherKetone (PEEK) y fibra de carbón (CF) mediante moldeo en caliente. Se caracterizó mecánicamente el material compuesto CF/PEEK con ensayos de flexión en tres puntos y tensión, se obtuvieron el modulo elástico y la resistencia máxima a tensión, respectivamente. También roscas fueron maquinadas en el material compuestos y se evaluó la resistencia de estas roscas mediante pruebas de tensión. Se realizó una comparación del material compuesto CF/PEEK con el material compuesto de fibra de carbón y resina epoxi (CF/EP), los módulos elásticos, la resistencia máxima a la tensión y la resistencia de las roscas fueron comparadas. Se encontró que fue posible producir a bajo costo mediante moldeo al caliente este compuesto de CF/PEEK. El módulo elástico y la resistencia a la tensión fueron menores que las obtenidas en el CF/EP. Sin embargo, el desempeño del CF/PEEK a tensión en las roscas fue mejor comparado con el compuesto de CF/EP.**

***Palabras claves*— Caracterización mecánica, Fibra de carbón, Manufactura, PEEK, Resina epoxi, Roscado.**

I. INTRODUCTION

FIBER reinforced materials are currently used in several applications as for example, in the aerospace, automotive, and biomedical engineering due to their good mechanical properties combined with a relatively low weight [1]. Fiber reinforced polymers (FPR) as composite materials are a good strengthening technique for a variety of structural applications and have been the focus of research in recent years. FPR were performed as high performance materials because of their advantages such as light weight, fatigue resistance, high tensile strength, corrosion and thermal insulation [2]. These materials are composed of fibers impregnated with a thermoset or thermoplastic polymer matrix. When a thermoplastic polymer is heated, their chemical bonds are broken causing a change of state from solid to high viscosity liquid. In this state this material can be deformed inside a mold where it is subsequently cooled to solidify and obtain a mold-shaped piece [3]. PolyEtherEtherKetone (PEEK) is a thermoplastic polymer with good mechanical properties frequently used as a matrix in composites materials [4]. One of the main characteristics of the PEEK is its high melting temperature until 400°C, which makes it suitable for applications at relatively high temperature. In many countries, the production of parts made of carbon fiber reinforced with PEEK (CF/PEEK) is no completely developed due to low availability of equipment required in the process of autoclave, Automated Tape Laying (ATP) and Laser Automated Tape Laying (LATP) [3]. In developing countries, the situation is even more complex due to the slow process of importing raw materials. However, manufacturing techniques have been designed for these thermoplastic composites with acceptable results. Some techniques such as: hot molding [5][6][7], injection molding [6][8] and fused deposition

This manuscript was sent on June 30, 2021 and accepted on November 21, 2022.
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modeling [9] [10] [11] are manufacturing techniques that have been implemented to obtain composite materials at a low cost. With the development of these techniques, in developing countries composite materials are being manufactured and used in applications such as: in bioengineering with the development of prostheses for amputees [12] and in the development of aircraft parts in Indonesia [13]. While in other countries, such as: Brazil, Nigeria, India and Pakistan use natural fibers to make biocomposites materials [14] [15] [16] [17].

Some authors who used hot molding to manufacture composite materials with a thermoplastic matrix were Batistas et al. [18], In their research, they manufacture sheets of carbon fiber (CF) reinforced with polyphenylene sulfide (PPS), they vary the cooling speed and compare the mechanical properties of these speeds, Kaya et al. [19], Made composite panels in PLA with basalt fibers (BF), they varied the fiber content to find high mechanical properties and Mazur et al. [C], Found in their research that hot molding is a good alternative to making the composite material with a thermoplastic matrix of polyetherketoneketone (PEKK) and carbon fiber with appropriate impregnation.

The biggest challenge in the manufacturing process of thermoplastic matrix composites is to achieve the appropriate stamping temperature, this is the most important factor that affects the quality of processing and the performance of these composites, for example, by increasing temperature can cause porosity, but too high a temperature will cause the matrix to decompose and increase porosity [7]. On the other hand, the cooling speed achieved with hot molding is also another challenge when processing these materials, since it affects the mechanical properties of the final product [6][7].

This paper presents a methodology to produce fiber-reinforced material and thermoplastic matrix at high temperatures, through a low-cost process, where a composite material with acceptable mechanical properties was obtained. The objective of this study was to produce CF/PEEK without conventional and expensive equipment and compare it with carbon fiber composite material and epoxy resin (CF/EP), especially the strength of machined threads for the use of bolted joints.

II. Materials And Methods.

A. *Materials*

Carbon fibers are frequently used as reinforcement material due to their high mechanical properties. Fibers with tension modules ranging from 207 GPa to 1035 GPa are currently available [21]. In this study, for the manufacture of the composite material we used fabric made of carbon fiber 3K reference HexForce 282 (Hexcel Corporation, Stamford, CT, USA), where the fibers follow an orientation pattern in the 0° and 90° directions and a plain weave style. According to manufacturer [22], this fabric has a thickness of 0.26 mm, a tensile strength of 3650 MPa and elastic modulus of 234 GPa. PEEK was chosen as matrix since it offers the possibility of being used at high service temperatures (until 250°C). It has an elastic modulus of about 4 GPa, a yield stress of 100 MPa, and has a higher fracture toughness in comparison with the epoxy resin [23]. Filaments of PEEK with 1.75 mm diameter (Apium Additive TechnologyGmbh, Germany) were used as matrix. While for the composite with thermosetting matrix, epoxy resin 744 was used.

B. *Fabrication process*

Hot compression molding was the selected manufacturing process since it is one of the lowest cost process due to the use of required equipment. This process begins with cutting a rectangular section of fabric. Then, PEEK filaments were added onto the fabric, followed by another layer of fabric laid over the PEEK filaments and so on until eight layers of fabric and eight layers of PEEK filaments were completed.

These layers of PEEK fabric and filaments were stacked and compressed inside a mold made of AISI 1020 steel plates with dimensions 150 mm x 150 mm x 4 mm and an electrical resistance that works at 110 V at the base. The composite material manufacturing process took 90 min. The parameters used during this thermal cycle were: temperature 380 °C, pressure 0.47 MPa and cooling rate 2.2 °C/min. Pressure was applied gradually, while heating the mold, using a Hubbard-field mechanical vise (Hubbard-field, Gainesville, FL, USA) with a 10000-pound capacity. The CF/PEEK pieces obtained were between 4 and 6 mm thick and presented a good surface finish. These pieces were cut with a band saw to obtain 15 mm x 150 mm specimens for bending tests and 25 mm x 150 mm for tensile tests (Fig. 1a). The FC/EP was manufactured by Hand Lay-Up [24].

C. *Fiber weight fraction*

The fiber weight fraction in the composite material resulting from compaction and heating process was calculated by using (1) [25].

$$W_f = (W_t / W_t + W_m) \cdot 100 \quad (1)$$

Where w_t and w_m are the weights of the fabric used and matrix respectively. For CF/PEEK the following procedure was performed to determine the weights mentioned above. First, the carbon fiber fabric and PEEK filaments needed for the composite material were weighed. Then, the compaction and heating process take place, and then the residual PEEK from the hot compression process was removed which was filtered from

the mold. Finally, the final product resulting from the CF/PEEK composite manufacturing process was weighted. While for the CF/EP composite, the weight fraction of the fiber was calculated with the initial weight of the fabric introduced into the mold, and compared with the final weight of the piece impregnated with the cured resin.
Total fiber volume fraction is calculated by using (2) following as [25] [26]:

$$V_f = \frac{w_t/\rho_t}{w_t/\rho_t + w_m/\rho_m} \quad (2)$$

Here, ρ_t and ρ_m are the densities of the fabric used and matrix respectively.

D. *Mechanical tests*

Three-point bending tests and tensile tests were performed on samples of the composite materials to determine their elastic modulus and ultimate strength. Also the strength of a thread machined on the composite material was evaluated by tensile tests as explained below. Three specimens were evaluated for each kind of test.
Based on the ASTM D7264 standard [27], this standard recommended a strain range of 0.002 with a start point 0.001 and end point 0.003, therefore, only the valid elastic region of stress-strain curve was registered. The specimens were subjected to three-point bending tests as shown in Fig. 1b. The two endpoints were simply supported while the load was applied at the midpoint. The tests were performed on a testing machine reference Lloyd LF plus instruments (AMETEK TCI, FL, USA). A displacement of two millimeters was applied to characterize the linear elastic zone of the material avoiding any damage to the specimens. Displacement was measured with a (1) LVDT reference LD620-7.5 (Omega Engineering Inc, Norwalk, CT). The force was measured with a tension-compression load cell type S NTEP (2). Data were registered with a DC 204R data acquisition system (TML, Tokyo Sokki Kenkyujo Co., Ltd., JP) with a sampling rate of 100 Hz.
According to ASTM D7264 the stress (σ) in the middle of the specimen is calculated with the force by using (3):

$$\sigma = 3PL/2bh^2 \quad (3)$$

Where P is the applied force, L is the distance between supports, b is the width of the specimen and h is the thickness. In addition, the strain (ε) in the center was calculated by using (4):

$$\varepsilon = 6\delta h/L^2 \quad (4)$$

Where δ is the deflection at the midpoint.

The elastic modulus was determined as the slope of the linear zone of the stress-strain curve.
Tensile tests were performed under the ASTM D3039 standard [28]. The tests were carried out in a universal Tinius Olsen H50KS test machine (Fig. 1c), to characterize the material ultimate tensile stress.

According to ASTM D3039 standard [28], the ultimate tensile stress (F^{tu}) recorded at the failure of the specimen is calculated by using (5):

$$F^{tu} = P^{max}/A \quad (5)$$

Where P^{max} is the maximum force reached just before the fracture and A is the cross-sectional area of the specimen.
The strength of a machined thread on the composite material was also evaluated. To generate the thread, an $13/64$ in hole was drilled in the composite material and subsequently an $1/4$ in tap with ordinary thread was introduced. An $1/4$ in bolt was inserted into this thread where a tensile test was performed on the Tinius Olsen H50KS universal testing machine based on ASTM F549-17 [29]. For the tests, an assembly was constructed (Fig. 1d) in which a fixed base (1) maintains the threaded area of the composite material (2) confined while the cover (3) top supports the test piece with bolts. With the screw (4) the force is applied.

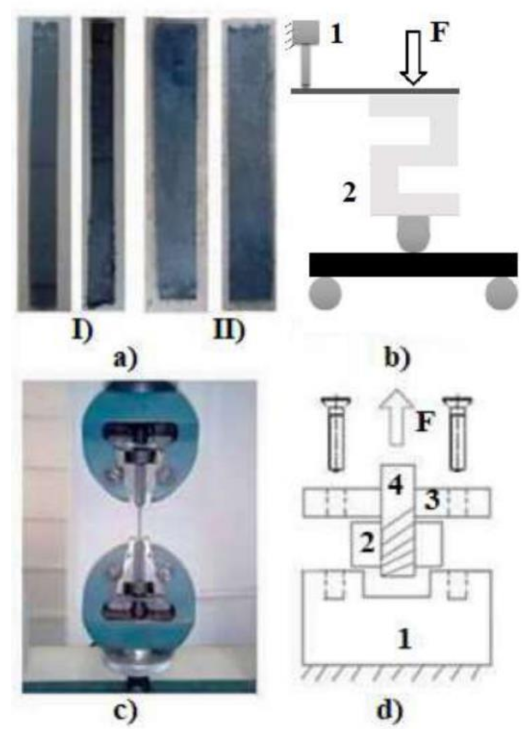

Fig.1. a) Specimens for mechanical tests I) Bending test, II) Tensile test. b) Three-point bending tests. c) Tensile test. d) Tensile testing scheme in composite thread

III. EXPERIMENTAL DESIGN

A completely randomized experimental design was used for one factor: the composite material, with two levels: CF/PEEK and CF/EP, using three replications per treatment. Six specimens for each composite material were mechanically evaluated: three using bending test and three using tensile test. Variable responses were the elastic modulus and the ultimate tensile stress.

Statistical analyses of variance were conducted for the elastic modulus and ultimate tensile stress with confidence level of 95%.

IV. RESULTS

To validate the analyses of variance, Ryan-Joyner (similar to Shapiro-Wilks) test was performed with p-values greater than 0.1 for the two analyses performed. In addition, we performed Levene test with p-values of 0.264 for the analyses of the elastic modulus and p-values of 0.386 for the analyses of the tensile ultimate strength results. The independence was guaranteed by randomize the sequence of the tests.

The fiber weight fraction achieved for the CF/PEEK composite was 44.3%, while for the CF/EP was 65.4%. Both CF/PEEK and CF/EP specimens showed an elastic linear behavior (Fig. 2) under the test conditions (only in the elastic region). For the CF/PEEK an average elastic modulus of 8.35 GPa was found with a standard deviation of 0.82, this value is within the elastic modulus of the matrix and the reinforcement, while the CF/EP material presented an average elastic modulus of 20.7 GPa with a standard deviation of 8.54.

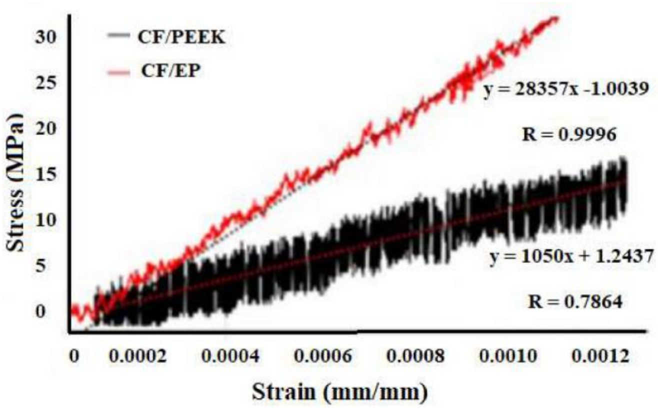

Fig. 2. Typical stress vs. strain curve obtained in FC/PEEK and CF/EP specimens tests

An analysis of variance (Anova) (Table I) showed that the difference between those average values was statistically no significant (p-value >0.05), probably due to the low number of specimens used for the tests, three for treatment.

TABLE I.
ANOVA ELASTIC MODULUS

Source	SS	df	Ms	F	p
Composite Material	232.9	1	232.88	6.32	0.066
Error	147.4	4	36.85		
Total	380.3	5			

The average ultimate tensile strength of the CF/PEEK composite materials was 256.4 MPa with a standard deviation of 117.8 MPa, this high standard deviation could be due to the manufacture of the composite material, the bond between PEEK layers and carbon fiber cloth, and for the CF/EP the average was 365.4 MPa with a standard deviation of 21.2 MPa. An analysis of variance (Table II) showed that the difference between those average values was statistically no significant (p-value = 0.308). The tensile tests were conducted only to obtain the tensile strength of the material; therefore, the strain was not registered during these tests and the corresponding stress-strain diagrams were not obtained.

TABLE II.
ANOVA ULTIMATE TENSILE STRENGTH

Source	SS	df	Ms	F	p
Composite Material	14153	1	14153	1.50	0.308
Error	28260	4	9429		
Total	42413	5			

For comparison purposes, Table III presents the results of elastic modulus and ultimate strength obtained for the CF/PEEK material together with other recent investigations. The elastic modulus and ultimate strength obtained was smaller than most of the values reported by other studies. However, the cost of the manufacturing process proposed in this study is low and this composite material could be carried out successfully. Low values of mechanical strength and ultimate tensile can be explained by the fiber volume low fraction and the adhesion between the layers of fabric and matrix obtained with the process presented in this paper in comparison with those reported in the literature. Chunrui et al. [30] obtained greater elastic modulus and tensile strength, probably because they manufactured the composite material CF/PEEK starting from layers of fabric made from yarns of PEEK and carbon fibers, which have good adhesion of peek in the carbon fiber, improved by the applied pressures of 0.5 MPa. Elwathing et al. [31], also obtained a material with greater elastic modulus than the obtained in the present study, probably due to chemical activation process in the carbon fiber with acid, combined with a manufacturing process using pressures and cooling rate of 2.5 MPa and 20 °C/min respectively. Shang-Lin [32] and Sharma et al. [33], manufactured composite material of CF/PEEK using carbon fiber and powder of PEEK. While Shang-Lin [32] obtained a material with relatively low elastic modulus, Sharma et al. [33] obtained a material with better mechanical properties probably explained by the thermal cycle and the compression using a pressure of 0.7 MPa.

TABLE III.
COMPARISON OF RESULTS CF/ PEEK COMPOSITE

Researcher	% Fiber Volume Fraction	% Fiber Weight Fraction	E (GPa)	σ_{max} (MPa)	Year
In this paper	36.8	44.33	8.35	256.4	2019
Lu et al. [30]	40.3	-	48.9	480.7	2018
Elwathing et al. [31]	40	-	23.5	-	2018
Shang-Lin et al. [32]	61	-	4.5	140.2	2000
Sharma et al. [33]	-	68	51	576	2011

The maximum force reached in the tensile tests on the threads made in the CF/PEEK composite material reaches an average of 5.6 KN with a standard deviation of 0.75 and for the CF/EP the average was 2.6 KN with a standard deviation of 0.79. The CF/PEEK composite was significantly better than the CF/EP for this application (p-value = 0.011) as shown in table IV.

TABLE IV.
ANOVA TENSILE ON THE THREADS

Source	SS	df	Ms	F	p
Composite Material	11358544	1	11358544	20.42	0.011
Error	2225118	4	556279		
Total	13583662	5			

The CF/PEEK composite material showed a lower modulus of elasticity and a lower ultimate tensile strength, compared with the CF/EP composite as shown in table V. This is probably due to the fact that in the CF/PEEK composite it was not possible to obtain a fiber weight fraction as high as was achieved in the CF/EP composite. The performance of the CF/PEEK material was greater in the maximum force reached in the threads, which may be due to a greater shear resistance of the PEEK with respect to the EP. This makes the CF/PEEK material a promising material in the manufacture of threaded joints.

TABLE V.
COMPARISON OF RESULTS CF/ PEEK AND CF/EP COMPOSITES

Composite	% Fiber Weight Fraction	E (GPa)	σ_{max} (MPa)	Fs_{max} (kN) on the threads
CF/PEEK	44.33	8.35(0.82)	256.4(117.8)	5.6(0.7)
CF/PEEK	65.4	20.7(8.5)	365.4(21.2)	2.6(0.8)

Data in parentheses are standard deviations

V. CONCLUSIONS

It was possible the manufacture of composite material of CF/PEEK without the conventional machines and at a low cost, additionally, this composite material presented mechanical properties similar to those reported by other authors. A linear behavior was evident between the stress and the strain in all the bending tests, for the evaluated range up to strain of 0.0012 (only in the elastic region) on the two composite materials. Regarding the effective elastic modulus and ultimate tensile strength, the CF/PEEK composite material was no better than the CF/EP composite material, a 60% lower effective elastic modulus and an ultimate tension 1.37 times lower for the PEEK compound compared to the EP compound. However, there are still opportunities to improve properties for the CF/PEEK composite material since by optimizing the manufacturing process, for example, by improving the adhesion of the PEEK in the carbon fiber, layering prepreg first, and increasing the weight fraction of carbon fiber.

The machining process of holes and threads in the composite material CF/PEEK was carried out with good results. The thread strength of the CF/PEEK reached values 2.15-fold higher than in the CF/EP composite. Therefore, the PEEK allows to use bolted joints more resistant in the composite material. Therefore, we can conclude that using this composite material in parts and geometries that require bolted joints guarantees better resistance than in composite material CF/EP.

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