D. García-González, M. Rodríguez-Millán, A. Rusinek, A. Arias. Low temperature effect on impact energy absorption capability of PEEK composites, in: *Composite Structures* 134 (2015), pp. 440–449
DOI:https://doi.org/10.1016/j.compstruct.2015.08.090

Low temperature effect on impact energy absorption capability of PEEK composites

D. Garcia-Gonzalez [a],*, M. Rodriguez-Millan [b], A. Rusinek [c], A. Arias [a]

[a] Department of Continuum Mechanics and Structural Analysis, University Carlos III of Madrid, Avda. de la Universidad 30, 28911 Leganés, Madrid, Spain
[b] Department of Mechanical Engineering, University Carlos III of Madrid, Avda. de la Universidad 30, 28911 Leganés, Madrid, Spain
[c] Laboratory of Mechanics, Biomechanics, Polymers and Structures, National Engineering School of Metz, 1 route d'Ars Laquenexy, CS 65820 57078 METZ Cedex 3, France

ABSTRACT

This paper describes the results of an experimental investigation which analyses the impact behavior at low temperature of polyether–ether–ketone (PEEK) and its short carbon fiber reinforced composite (SCFR PEEK). These polymer materials are widely employed in aeronautical applications subjected to impact loadings in which the energy absorption capability is an aspect that should be taken into account. The energy absorption capability can drastically decrease if temperatures near to the ductile-to-brittle transition temperature of polymeric matrix are reached. In this work, a set of perforation tests has been conducted covering a testing temperature range from −75 °C to +25 °C and an impact kinetic energy range from 11 J to 175 J, including typical values considered in impact loadings at aeronautical flight speeds. Energy absorption capability, damage extension and failure mechanisms have been quantified and reported. At low temperatures, a ductile-to-brittle transition was found in PEEK unfilled resulting in a suddenly change of its mechanical impact behavior affecting the energy absorption capability. In case of SCFR PEEK composite, a brittle behavior was observed for the whole temperature range considered and its energy absorption capability decreases drastically at lower temperatures. The brittleness of PEEK and SCFR PEEK at low temperature will limit the application of this composite in aeronautical structures exposed to impact.

Keywords:
Short fiber composites
PEEK composites
Low temperature
Impact
Brittle failure
Polyether–ether–ketone

1. Introduction

Thermoplastics and their composites reinforced with short carbon fibers are increasingly employed in many industries due to their attractive mechanical properties, rapid processing by injection molding and relatively low manufacturing cost. In case of aerospace and naval applications, thermoplastics have a special consideration due to their attractiveness in terms of mechanical properties, excellent thermal properties (high melting temperature), recyclability and suitability of being manufactured by modern imaging technology. Short fiber reinforced polymers (SFRPs) were developed to fill the mechanical property gap between the continuous-fiber laminates used as primary structures by the aircraft and aerospace industry and the unreinforced polymers used in non-load-bearing applications [1]. Since nowadays, thermoplastics and their composites are widely used in aircraft applications and civilian aircraft materials usually have to perform their duty in the temperature range from −50 °C to +80 °C, it is essential to know about the thermal properties of these materials.

In this regard, it is known that the behavior of these thermoplastic polymers is rather complex as it is time, strain rate and temperature dependent and couples both viscoelastic and viscoplastic modes of deformation [2]. The mechanical properties are strongly dependent on temperature and strain rate since this process is thermally activated with viscous characteristics [3,4]. So, it is well established that strong coupling exists between the thermal and mechanical behavior of thermoplastic composites.

Considering aeronautical applications which are subjected to dynamic loadings like impact, their structural components must present good energy absorption capability. It seems that strain rate and temperature are the mainly variables controlling the energy absorption efficiency of these thermoplastic materials. The yielding and plastic flow behaviors are affected by strain rate resulting in a continuous hardening and loss in ductility as this variable increases [5,6]. Regarding thermal dependence in the material behavior, if the thermoplastic polymer temperature is below glass transition, there is a sudden change in the amorphous molecule segments and the polymer molecules lack the ability to undergo

---

* Corresponding author. Tel.: +34 916249161.
E-mail address: danigarc@ing.uc3m.es (D. Garcia-Gonzalez).

considerable motion due to insufficient kinetic energy. In case of exposing the thermoplastic to progressively lower temperatures, the material undergoes another transition, known as the ductile-to-brittle transition temperature. During this transition the polymer loses a substantial level of kinetic energy resulting in restricted motion of the chains. This process results in a sudden, sharp loss in ductility [7]. Therefore, in order to prove the validity of using these thermoplastic polymers in aeronautical applications, it is crucial to carry out a study of the impact behavior considering both strain rate and thermal effects on these polymer materials, and how these variables influence the energy absorption capability.

One of the most commonly used thermoplastic for a variety of structural applications is the semi-crystalline thermoplastic polyether–ether–ketone (PEEK) and its composite reinforced with polyacrylonitrile (PAN) short carbon fibers 30% in weight (CF30 PEEK). These materials are currently used in space applications for replacing aluminum because of their superior performance at high temperatures [8]. A few researchers focus on the performance of PEEK composites at room temperature [9]. However, the results about the mechanical and thermal properties obtained from room temperature cannot simply be transferred to the low temperatures. Moreover, the effect of low temperature on the impact behavior of PEEK and short carbon fiber reinforced (SCFR) PEEK composites have not been reported in the scientific literature.

In this work, a set of perforation tests have been conducted covering a testing temperature range from −75 °C to +25 °C and an impact kinetic energy range from 11 J to 175 J, including typical values considered in impact loadings at aeronautical flight speeds. Experimental observations showed that ductile-to-brittle transition is reached in these conditions. The brittleness of PEEK and SCFR PEEK at low temperature has been demonstrated as a combination of thermal and strain rate mechanisms.

2. Material

Commercial plates of PEEK composites reinforced with PAN short carbon fibers 30% in weight, named CF30 PEEK, and unfilled PEEK plates of general purpose grade were purchased measuring 130 × 130 × 3 mm^3. Both materials are produced with injection molding technology. Carbon fiber is currently the most widely used fibrous reinforcing agent for PEEK based composites [10] due to the strong interfacial interaction between short carbon fibers and PEEK matrix. The interfacial strength between short carbon fibers and PEEK matrix is higher than other known combinations of fibers and thermoplastic matrices [11–13], and on average, at least an order of magnitude stronger than that between carbon fibers and ultra-high-molecular-weight polyethylene (UHMWPE) polymers [10,14]. For CF30 PEEK, the diameter and length of PAN carbon fiber were 7 μm and 200 μm respectively. The percentage of 30% carbon fiber in weight (23.5% in volume) of CF30 PEEK provides optimum rigidity and load bearing capability. The mechanical properties of PEEK and CF30 PEEK composite are shown in Table 1 [15–17], supported by data published by authors [17]. Addition of short fiber into PEEK matrix increases the low elastic modulus from 3.6 GPa for neat PEEK to 24 GPa for SCFR PEEK and it doubles the failure strength value. Failure strength in this paper refers to ultimate tensile strength or yield stress, according to which was reached first in tensile testing [12].

2.1. Mechanical characterization of SCFR PEEK composite

One inherent problem in processing short fiber reinforced thermoplastics (SFRTPs) by flow molding techniques is that the fibers will tend to become aligned during the flow process, inducing anisotropic material properties. In SCFR PEEK composites, the skin-core structure is well document, Fig. 1, [18]. Scanning electron micrographs of the fracture surfaces of SCFR PEEK considered in this work, Fig. 2, correlate well with the above macroscopic considerations. Three layered were observed: a top and bottom skin layer revealed fiber alignment along the melt flow direction, Fig. 2a, whereas in the core they were transversally oriented, Fig. 2b.

Table 1
Mechanical properties of PEEK and CF30 PEEK composite [15–17].

	SCFR PEEK composite (CF30)	Unfilled PEEK
Elastic modulus (*GPa*)	24	3.6
Poisson's ratio	0.385	0.38
Density (kg/m^3)	1400	1300
Yield stress (MPa)	–	107
Tensile strength (MPa)	214	95
Elongation at break (%)	2.0	40.0
Charpy impact strength (kJ/m^2)	6.50	7.0
Glass transition temperature (K)	416	416
Melt transition temperature (K)	610	616
Ductile–brittle transition low temperature (K) [16]	213	208

In order to investigate the effect of orientation on the mechanical behavior, tensile and compressive tests of injection molded specimens were conducted in previous work [17] using a servo-hydraulic testing machine INSTROM 8516 under displacement control at 1 mm min^{-1}. Tensile and compressive samples were machined on the ASTM D-638 recommendations and ASTM D-695. Young's modulus and failure strain and their respective strains were determined as the mean value of at least eight specimens and results are shown in Table 2. Fig. 3 shows the stress–strain curves of tensile and compression tests for CF30 PEEK composite in both injection flow direction (IFD) longitudinal and transverse directions. Longitudinal values are higher for both tensile strength and compressive strength. Short fibers are mainly aligned in the injection flow direction. In addition, the results showed an enhanced behavior under compressive loading than tensile loading (Table 2). Specimens machined in the flow direction showed tensile and compressive strength approximately 40% lower than specimens machined transverse to the flow direction. About the degree of crystallinity, several authors have shown that increasing this property can increase elastic modulus and yield strength while decreasing fracture toughness. From differential scanning calorimetry (DSC) a degree of crystallinity of 30 ± 2% was calculated for PEEK and 32 ± 2% for CF30 PEEK integrating the melt endotherm. The results of DSC testing did not show significant differences in the ability of matrix to crystallize between unreinforced PEEK and CF30 PEEK. This finding is in agreement with data reported by Sarasua and Remiro [17,19].

2.2. Temperature sensitivity

Most thermoplastics and thermoplastic composites are temperature sensitive. This behavior is mainly due to the changes in the matrix properties with temperature. For structural thermoplastic matrix materials the glass transition and the low transition temperature represents useful cut off points. At temperatures above the glass transition, α temperature, the polymer molecules have sufficient kinetic energy to allow considerable motion. Below the glass transition, the molecules lack the ability to undergo this motion. It is important to consider that the glass transition and melting transitions that semi-crystalline polymers undergo, affect the respective amorphous and crystalline phases. However, as an amorphous polymer is exposed to progressively lower temperatures, the material undergoes another transition, known as the

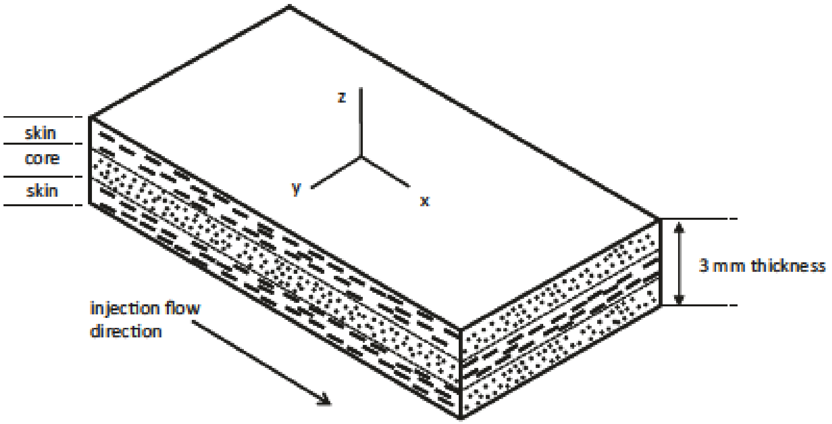
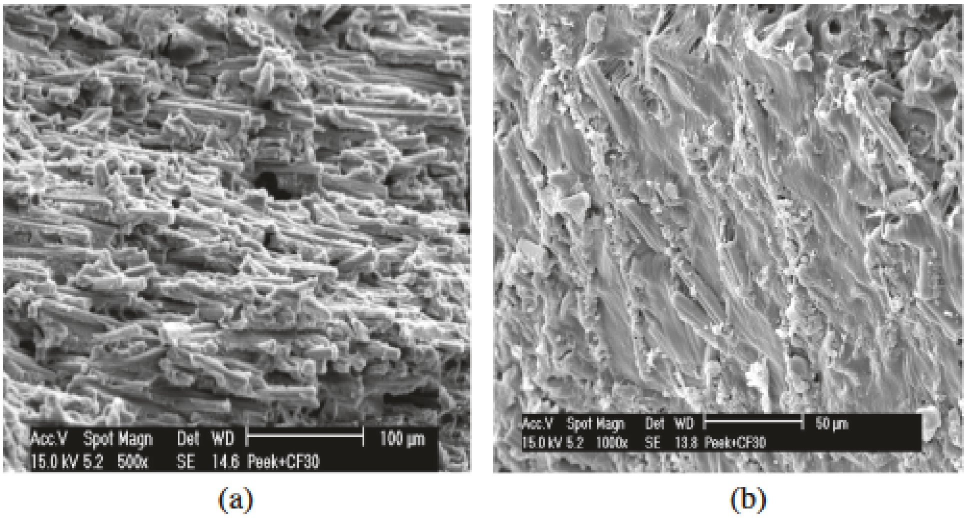


Fig. 1. Skin/core fiber orientations in the short fiber composite plates on this work in agreement with Evans et al. [18].



(a) (b)

Fig. 2. Scanning electron micrograph of skin (a) and core (b) of SCFR PEEK considered in this work.

Table 2
Mechanical properties of SCFR PEEK composite in both IFD longitudinal and transversal material directions [17].

Mechanical properties	SCFR PEEK composite (CF30)	
	Transversal	Longitudinal
Tensile strength (MPa)	148	214
Compressive strength (MPa)	174	239
Tensile elastic modulus (GPa)	12.6	24
Compressive elastic modulus (GPa)	15	44
Poisson's ratio (–)	0.38	0.38
Elongation at break (%)	1.9	2.0

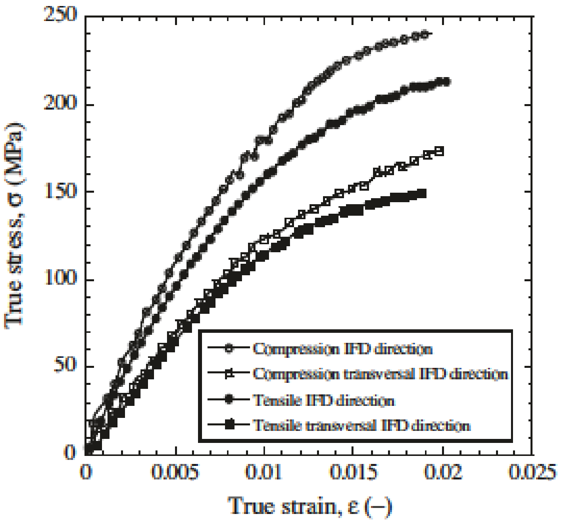


Fig. 3. Mechanical behavior of CF30 PEEK composite under compression and tension for specimen machined in the IFD longitudinal direction and transverse direction [17].

ductile-to-brittle transition, β temperature. This temperature represents passage through a lower order transition. During this transition, the polymer loses a substantial level of kinetic energy, which results in restricted motion of the chains. This transition results in a sudden, sharp loss in ductility. The beta transition occurs below the glass transition temperature for many polymers at −100 °C to 100 °C. It is caused by the rotation of segment of the polymer chains. In case of PEEK matrix, the beta transition occurs at −60 °C at quasi-static conditions [16]. In this regard, PEEK matrix has been tested by Rae et al. [6] for different initial temperatures and strain rates. In this study, positive strain rate sensitivity was observed with the mechanical properties increasing with the strain rate. Concerning the temperature effect, Fig. 4, a loss of ductility was observed when the initial temperature was lower than the room temperature $T_0 = 300$ K. Nonetheless, the

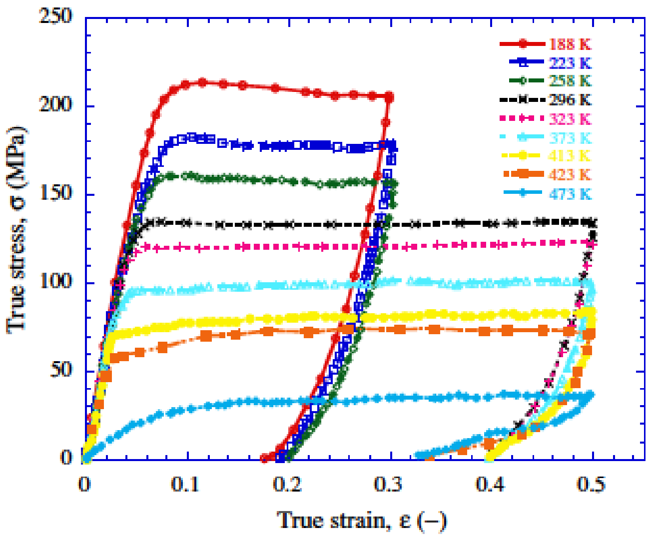


Fig. 4. Mechanical behavior of PEEK under compression for different initial temperatures at 0.001 1/s [6].

ductility was retained with a strain level larger than $\varepsilon_{low\ temperature} > 0.2$. The value for temperature sensitivity is $v = \partial\sigma/\partial T \approx -0.63\ \mathrm{MPa\ K^{-1}}$ [9]. In dynamic loading, for a strain rate close to $\dot{\varepsilon} \approx 3000\ \mathrm{s^{-1}}$, the temperature sensitivity is equal to $v = \partial\sigma/\partial T \approx -0.71\ \mathrm{MPa\ K^{-1}}$.

3. Low velocity perforation of PEEK and SCFR-PEEK plates at different temperatures

In order to conduct the low impact tests at different temperatures a drop weight tower has been used. The drop weight tower has a climatic chamber allowing a range of initial temperatures during the tests from −75 °C to 25 °C. A thermocouple was connected to a temperature controller regulating the opening of an electrovalve, which allowed a controlled volume of liquid nitrogen to enter the chamber, Fig. 5. Thus, the testing temperature could be accurately defined by the operator. Before testing at low temperature the PEEK specimens (clamped and screwed), they were subjected to the initial temperature for 15 min. This period of time is suitable to reach thermal equilibrium material-target/testing-temperature. More details of drop weight tower can be found in the work developed by Gómez-del Río et al. [20]. This analysis considers impact energy (controlling both impact velocity and striker mass), deformation mode, evolution of the impact force versus striker displacement and testing temperature.

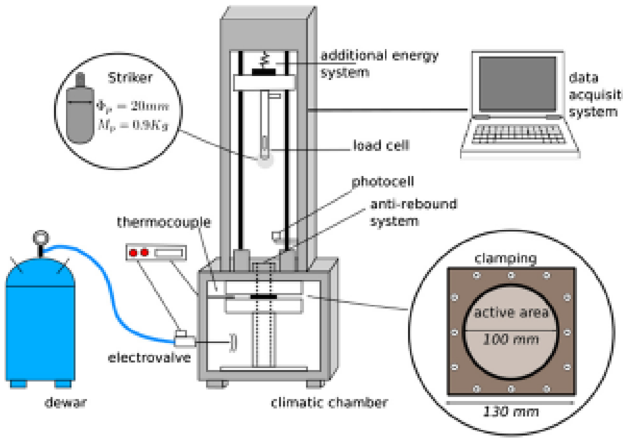


Fig. 5. Experimental setup for drop tower test at low temperature.

The specimens with dimensions of 130 × 130 × 3 mm^3 were clamped in a modular tool by screws all around a circular active area of 100 mm of diameter. The screws were symmetrically fixed in order to avoid any disturbance during the test. The steel striker used has a hemispherical shape. Its larger diameter is $\varphi_p = 20$ mm and its mass is $M_p = 0.90$ kg. The projectiles were machined using maraging steel, which exhibits higher yield stress – $\sigma_y \approx 1$ GPa – than that of the materials tested under dynamic conditions of deformation. In addition, the projectiles underwent a heat treatment to increase their hardness. The striker was attached to the instrumented bar attached to a metal frame, whose mass is $M_f = 2.9$ kg. Additional mass was added to the setup in order to increase the effective mass (M_{total}) from 3.8 kg until 28.8 kg. After the impact, an anti-rebound system held the striker in order to avoid multi-hits on the specimen if no perforation of the plate occurred. A local cell placed on the striker calculates the time dependent displacement $\delta_s(t)$ of the striker during the impact process by integration of the impact force versus time curve $F(t)$:

$$\delta_s(t) = \int_0^t \left[V_0 \int_0^\tau \frac{F(\theta) \cdot M_{total} \cdot g}{M_{total}} d\theta \right] d\tau \qquad (1)$$

where t is the time from the instant at which the striker bar hits the specimen, M_{total} is the striker bar mass and V_0 the striker initial velocity at the beginning of the experiment ($t = 0$): During the test, the energy transmitted to the specimen E at any time may be approached

$$E(t) = \int_0^t F\, d\delta = \int_0^t F(\tau) \left[V_0 \int_0^\tau \frac{F(\theta) \cdot M_{total} \cdot g}{M_{total}} d\theta \right] d\tau \qquad (2)$$

4. Results and discussion

The ductile/brittle behavior of polymer thermoplastic composites depends on many factors and most composites can exhibit the two responses depending upon environmental conditions. The failure of a brittle or ductile material is fundamentally different: brittle fracture occurs fast and is often characterized by instability. On the other hand, ductile failure is more progressive. The conditions influencing the ductile/brittle aspect of composites can be grouped into two categories. The first category includes environmental parameters such as temperature (ductility is increased by raising the temperature), strain rate (ductility is increased by decreasing the strain rate) and solvents. The second category influencing the brittleness of the composite includes parameters intrinsic to the material: the nature of the polymer matrix, fillers and fibers.

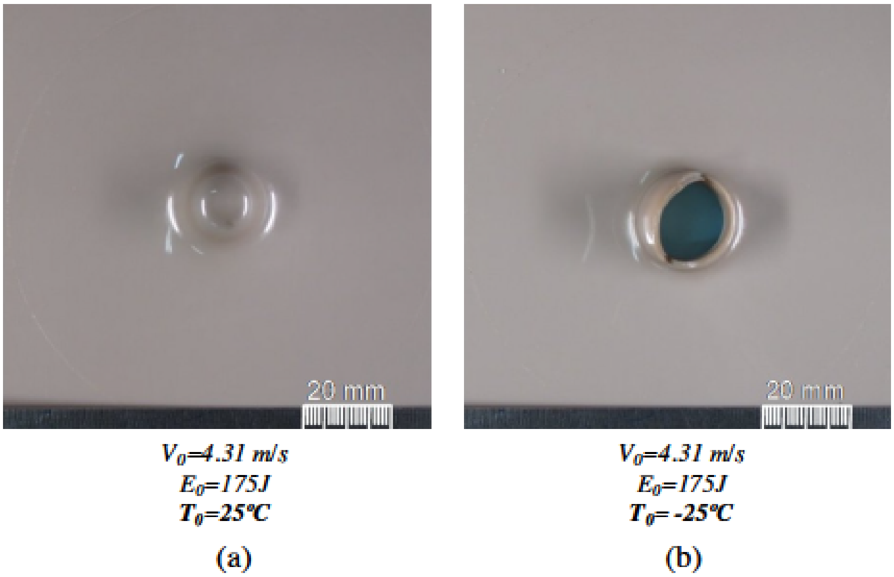
4.1. Failure mode of PEEK unfilled and SCFR PEEK composite

Figs. 6 and 7 illustrate the final stage of the impact process for different initial temperatures of PEEK. Figs. 8 and 9 show the final stage of the impact process for different initial temperatures of SCFR PEEK composite. The failure mode of SCFR PEEK composite is different from that observed in unfilled PEEK. While SCFR PEEK shows a brittle behavior for all testing conditions considered, unfilled PEEK shows a transition of ductile/brittle behavior depending on the initial temperature.

For PEEK, Fig. 6a, according to experimental observations in terms of failure mode, it was observed that when the specimen is tested at room temperature (≈25 °C), it fails following a hole enlargement mechanism. Moreover, this ductile behavior is still remained until −25 °C, Fig. 6b. However, in case of the specimens tested below −25 °C, all of them have shown a completely brittle behavior, as it can be observed in Fig. 7a and b. Regarding the specimens tested using −25 °C as initial temperature, it was found cases in which PEEK specimen behaves in a ductile manner and others in which it behaves in a brittle one. Therefore, it has been assumed the ductile-to-brittle change in impact behavior at −25 °C, founding the initial temperature as a key variable in the material behavior of PEEK. This change in the impact behavior is influenced by the ductile-to-brittle transition temperature. This value is defined as the temperature at which brittle and ductile response of the material is equally likely [21]. Although it is known that ductile-to-brittle transition temperature for PEEK, the beta transition, occurs at −60 °C at quasi-static conditions [16], other authors have observed that this transition is dependent on both temperature and strain rate. So there is a coupling between ductile-to-brittle transition temperature and strain rate depending on tests conditions, the higher strain rate, the higher ductile-to-brittle transition temperature [7].

Regarding all the specimens tested below the transition temperature, −50 °C, Fig. 7a and −75 °C, Fig. 7b, they have shown a failure based on the propagation of radial cracks from the impact zone. In all these cases below the transition temperature the failure was found completely brittle without any evidence of local bending in the impact zone. These observations are in agreement with the study reported in the work of Karger and Friedich [22], where a trend to brittle fracture with decreasing temperature and/or increasing strain rate was found, while a more ductile failure mode was found in the opposite direction.

For SCFR PEEK composite, Figs. 8 and 9, the experimental observations allow a thorough study of the damage extension showing a characteristic mode of failure based on the propagation of some cracks from the impact zone which cover the whole plate for impact energies near to the perforation limit. In case of increasing the impact energy, there is a reduction in the damaged area and also another failure mechanism based on an elision shape hole.



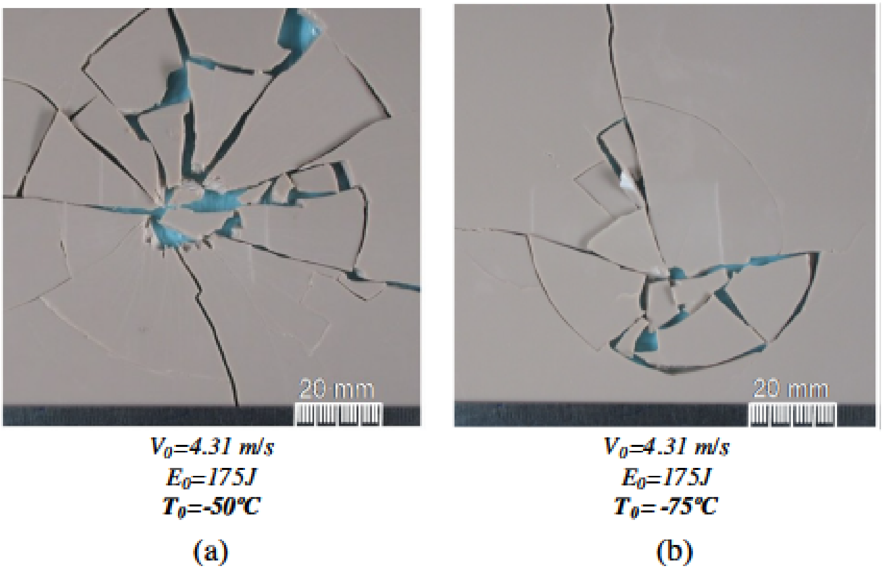
V_0=4.31 m/s
E_0=175J
T_0=25°C

(a)

V_0=4.31 m/s
E_0=175J
T_0= -25°C

(b)

Fig. 6. Final stage of ductile failure after the impact process of unfilled PEEK at initial temperature: (a) T_0 = 25 °C; (b) T_0 = −25 °C.



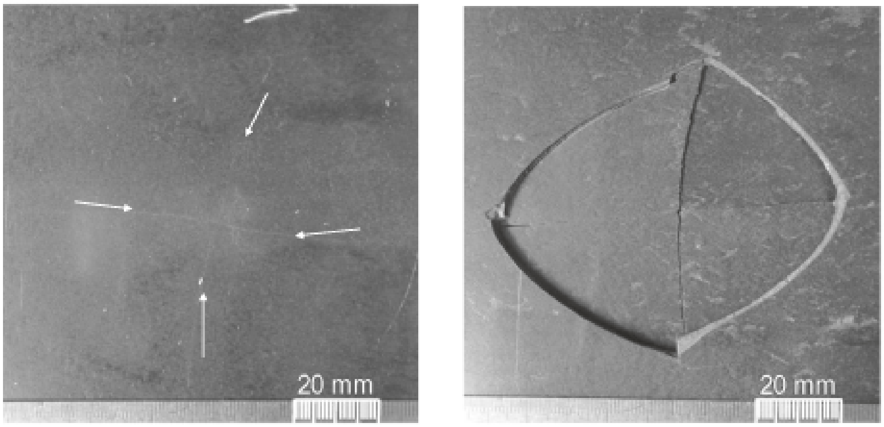
V_0=4.31 m/s
E_0=175J
T_0=-50°C

(a)

V_0=4.31 m/s
E_0=175J
T_0= -75°C

(b)

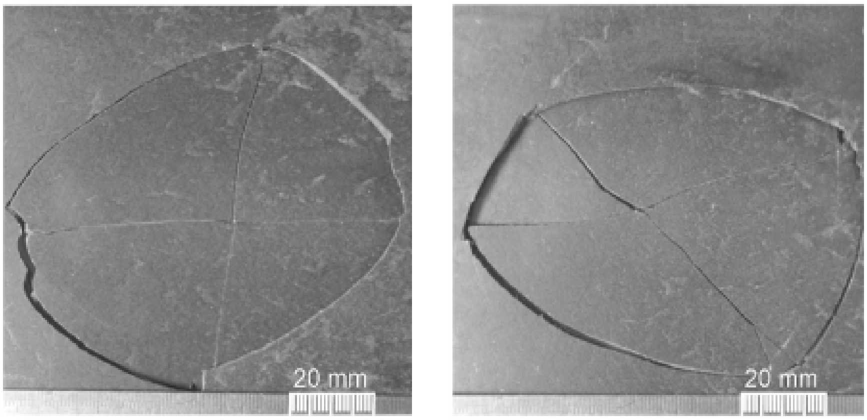
Fig. 7. Final stage of brittle failure after the impact process of unfilled PEEK at initial temperature: (a) T_0 = 25 °C; (b) T_0 = −25 °C.



V_0=2.43m/s
E_0=11.5J
T_0=25°C
(a)

V_0=4.31m/s
E_0=175J
T_0=-25°C
(b)

Fig. 8. Final stage of brittle failure of the impact process of SCFR PEEK at initial temperature: (a) $T_0 = 25$ °C; (b) $T_0 = -25$ °C.



V_0=4.31m/s
E_0=175J
T_0=-50°C
(a)

V_0=4.31m/s
E_0=175J
T_0=-75°C
(b)

Fig. 9. Final stage of brittle failure of the impact process of SCFR PEEK at initial temperature: (a) $T_0 = -50$; (b) $T_0 = -75$ °C.

The failure sequence of generation of cracks for SCFR PEEK composites with skin-core structure, Fig. 1, has been reported in the work of Jeng and Chen [10], Fig. 10. The failure mechanism observed in the SCFR PEEK specimens tested in this work is in agreement with the observation reported by Solomon et al. [23] and for drop weight impact tests on injection molded short fiber reinforced polymer composites. This failure is based on the propagation of two fissures along the IFD and the perpendicular one. A brief explanation of these preferential directions in the failure and the effects of fiber orientation can be obtained from the work of Mortazavian and Fatemi [24]. These authors that strain at tensile strength, as a measure of ductility, presents the lowest values in specimens with fibers aligned in longitudinal and transverse direction. In case of specimens presenting longitudinal direction of fiber alignment, the low ductility of the composite can be explained by the low capacity of fibers in straining. Regarding specimens presenting transverse direction of fiber alignment, the low ductility can be explained by the limited space for yielded material to transport. Regarding temperature effects, it was observed a higher damage

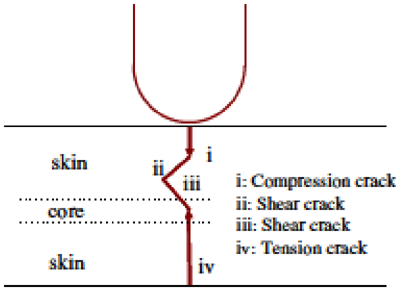


Fig. 10. Failure sequence of generation of cracks in SCF/PEEK composites subjected to impact load [10].

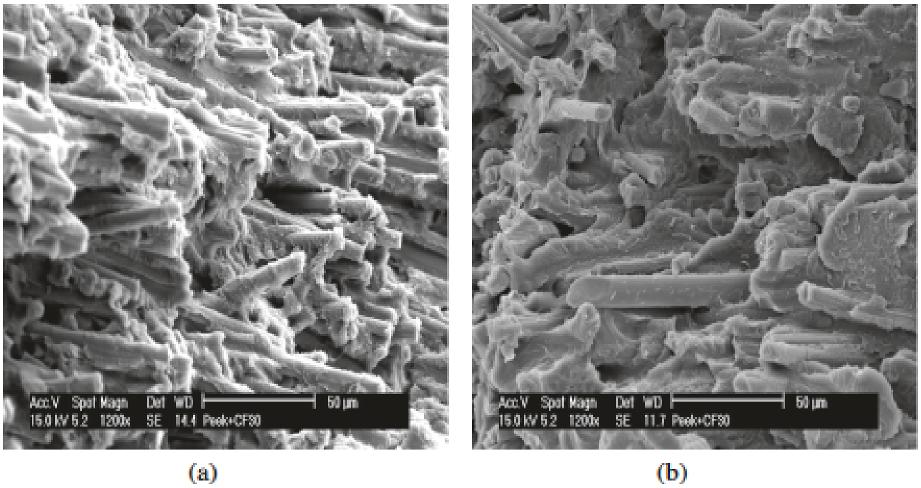
extension as the testing temperature decreases, Figs. 8 and 9. While there is evidence of temperature influence on the damage extension, the way all specimens fail does not vary. A failure mode based on an elision shape hole compound by four pieces controls the SCFR PEEK failure under impact loading, presenting a more localized damage extension for higher impact energies and a higher damage extension for lower testing temperatures. Scanning electron micrographs of the fracture surfaces of specimens tested at −75 °C and 25 °C are shown in Fig. 11. Observations of fracture surfaces revealed the existence of a high amount of matrix adhered to fiber surfaces, characteristic of the high interfacial strength between short carbon fibers and PEEK polymer [17]. At low temperature, micrograph correlates well with the above macroscopic observations, showing a reduction of matrix plastic deformation and adhesion at −75 °C.

4.2. Low impact velocity response of PEEK unfilled and SCFR-PEEK at room temperature

The analysis of the experimental force-striker displacement curves at room temperature, Fig. 12, shows a strong dependent of the material considered on the amount of kinetic energy of the striker converted into plastic work. During perforation, the much greater ductility of PEEK unfilled results on a greater strain leading to target failure. Consequently, a completely different material behavior is denoted and suggests different failure mechanisms for both materials tested. Despite the higher flow stress level of SCFR PEEK, the slope of the force-striker displacement curve is quite similar for both PEEK materials. The gap in the striker displacement at failure is much greater in case of PEEK unfilled according to what can be expected from tensile tests, Figs. 3 and 4. Following with the observations from the curves represented in Fig. 12, it can be noticed higher values of force to perforate PEEK unfilled. The positive strain rate sensitivity with the mechanical properties observed in PEEK unfilled [9,25], has a direct influence on the maximum force reached under dynamic conditions. Regarding SCFR PEEK, early onset fracture initiated on the tension side implies the very brittle behavior of the composite limiting the maximum force that this material can be suffer without perforation. Both higher values of maximum force reached and maximum striker displacement at perforation presented by PEEK unfilled, make it more competitive than SCFR PEEK in terms of energy absorption capability. Despite the increase in mechanical properties when short fibers are added, the loss in ductility results in an energy absorption capability reduction, represented by the decrease of area under the force-striker displacement curve. Therefore, in order to confirm the validity of employing SCFR PEEK in applications that can be subjected to dynamic conditions, a specific consideration in terms of energy absorption capability must be taken into account due to the loss in ductility caused by adding short fiber reinforcement.

4.3. Temperature influence on the impact behavior of unfilled PEEK

It was carried out an analysis of the temperature influence on the PEEK experimental force-striker displacement curves, Fig. 13a. These results show a strong dependence of the PEEK impact behavior on the testing temperature. While at room conditions it was found a clearly ductile behavior of PEEK material according to previous experiments [9], it was observed a change in the failure mode when a temperature is reached leading to a change in the ductile/brittle behavior of the polymer, −25 °C. As it can be expected from the stress–strain curves, Fig. 4, as the temperature is decreased, a material hardening is appreciated but also a strong reduction in the material ductility. This loss in ductility due to low temperatures directly affects to the energy absorption capability, represented by the area under the force–displacement curve Fig. 13b. In Fig. 13a it has been compared the impact behavior of PEEK specimens for different initial testing temperatures, fixing an impact kinetic energy equal to 175 J in each test. No perforation and a very ductile behavior are observed in case of conducting the test at room temperature. The transition behavior from ductile to brittle starts to be considerable if the temperature is decreased until −25 °C. Moreover, it can be noticed an increase in stiffness associated to a hardening due to low temperature effects and also a first loss in ductility which results in a completed perforation of the specimen. These observations are in agreement with the work presented by Pettarin et al. [26] in which a set of thermoplastic polymers are analyzed in terms of low temperature impact fracture data. Regarding the tests conducted for testing temperatures lower that −25 °C, it can be observed that the loss in ductility results in a strong change in the failure mode to brittle. This mode of failure is determined by which of these critical stresses, the fracture stress σ_b or the yield stress σ_y, is first exceeded in a sample. Below the transition temperature, brittle behavior is encouraged, because σ_b is smaller than σ_y; yielding occurs above the transition temperature [21]. The embrittlement due to the reach of the σ_b before the σ_y leading a loss in ductility, directly



(a) (b)

Fig. 11. Scanning electron micrograph of the fracture surface of impacted SCRF PEEK composite: (a) room temperature $T_0 = 23$ °C m/s; (b) low temperature $T_0 = -75$ °C.

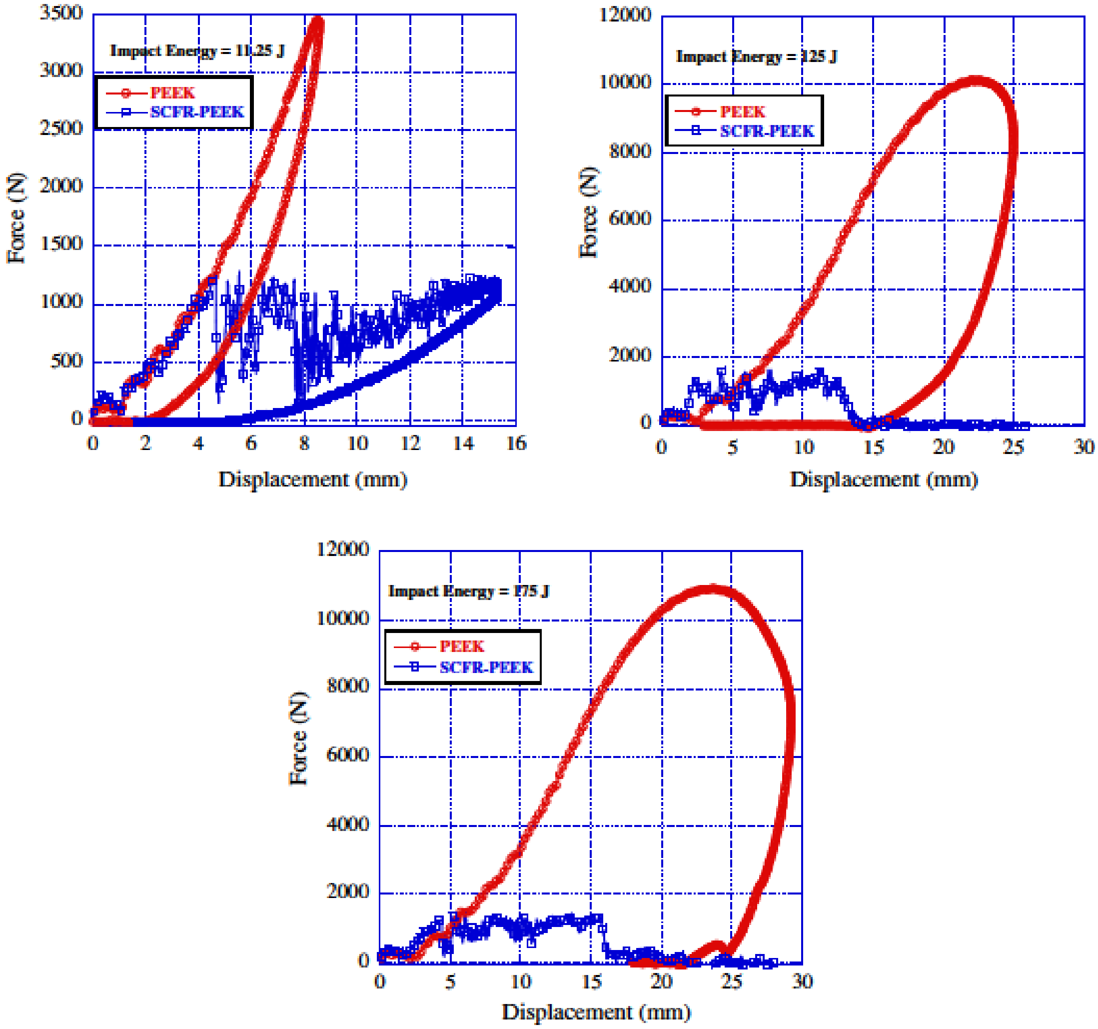
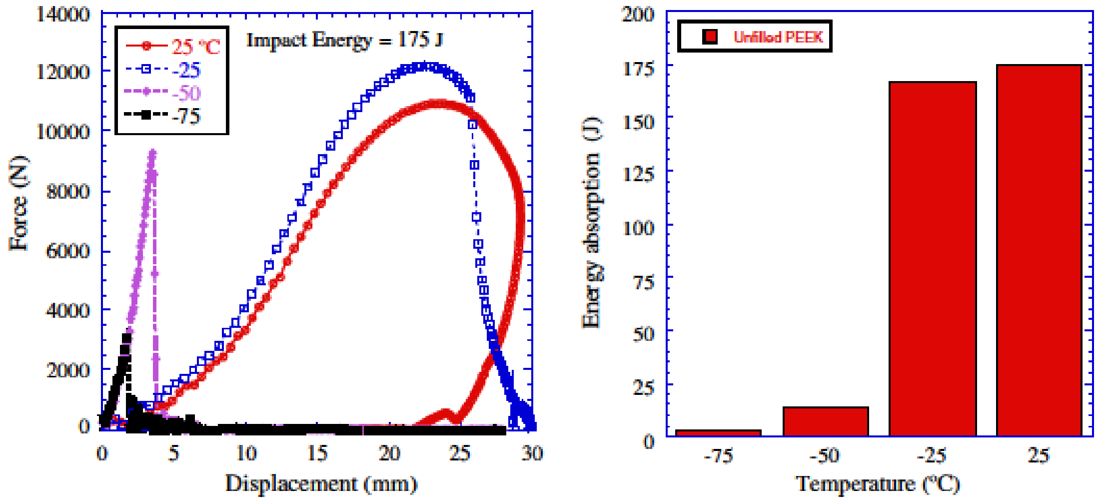
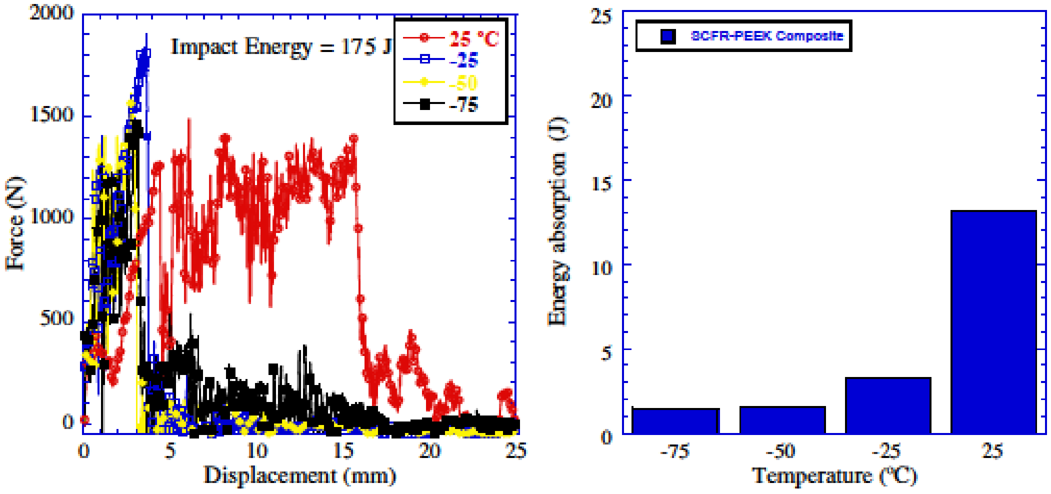


Fig. 12. Force–displacement curves of unfilled PEEK and SCFR PEEK composite for low velocity impact tests at initial room temperature, 25 °C.



(a) (b)

Fig. 13. (a) Force–displacement curves of unfilled PEEK for low velocity impact tests at different initial temperature; (b) energy absorption of unfilled PEEK versus different initial temperature.



(a)

(b)

Fig. 14. (a) Force–displacement curves of SCFR PEEK composite for low velocity impact tests at different initial temperature; (b) energy absorption of SCFR PEEK composite versus different initial temperature.

affects the energy absorption capability making this material less competitive in impact applications in which the environmental temperature reaches levels that induce a change in the ductile/brittle behavior.

To a better understanding of the loss in energy absorption capability, the observations from the study reported by Karger-Kocsis and Fiedrich [22] have been taken under consideration. These authors studied the effects of temperature and strain-rate on the fracture toughness of PEEK and its short fiber reinforced composite. On the one hand, they observed a linear decrease with temperature in terms of elastic tensile modulus. This fact would theoretically suppose higher energy absorption capability for lower temperatures. However, they also found a second grade relation between fracture energy and temperature with a maximum peak around 0 °C. For temperatures above the maximum peak of fracture energy, the continuous decrease in energy needed to fracture can be explained by the thermal softening which affects both elastic modulus and yield stress [6]. In case of the reduction of fracture energy for temperatures below the maximum peak of fracture energy, it may be explained by the strong reduction in ductility which limits the deformation until failure. Besides, this reduction in the failure strain is more pronounced in case of high strain rates like the ones applied in impact conditions [22].

4.4. Temperature influence on the impact behavior of SCFR-PEEK composite

An analysis of the temperature influence on the SCFR PEEK impact behavior was also developed in terms of experimental force-striker displacement curves, Fig. 14a. Although no change in terms of failure mode due to low temperature effects is observed, a loss in ductility has been found as the temperature is decreased. This fact results in a loss of the energy absorption capability understood as the area under the force–displacement curve, Fig. 14b. It is due to a strong dependence of the mechanical properties of PEEK matrix with temperature.

5. Conclusions

In this work, a set of perforation tests has been conducted at low temperatures covering a testing temperature range from −75 °C to +25 °C and an impact kinetic energy range from 11 J to 175 J, including typical values considered in impact loadings at aeronautical flight speeds. In the full range of temperatures considered, SCFR PEEK composites showed a brittle failure in line with the experimental observations studied by other authors in terms of thermal and strain rate mechanisms. SEM inspection tests and macroscopic observations on impacted specimens show that the failure of SCFR PEEK at low temperature is dependent on material directions, derived of anisotropic material properties due to flow molding manufacturing. Experimental observations showed a change in the PEEK impact behavior due to the reach of the ductile-to-brittle transition. This change directly affects the energy absorption capability limiting the application of PEEK composites in aeronautical components subjected to impact loadings at low temperatures. In conclusion, the absorption energy capability of SCFR PEEK decreases drastically in comparison with unfilled PEEK. The brittleness of PEEK composites at low temperatures will limit the application of this composite in aeronautical components.

Acknowledgments

The researchers of the University Carlos III of Madrid are indebted to the Ministerio de Ciencia e Innovación de España (Project DPI/2011-24068) and to the Ministerio de Economía y Competitividad de España (Project DPI/2014-57989-P) for financial support towards part of this work. The researchers are indebted to LATI Company for PEEK material supplied. The authors express their thanks to Mr. Iago Funes, Mr. Sergio Puerta and Mr. David Pedroche for their technical support.

References

[1] Rezaei R, Yunus R, Ibrahim N. Effect of fiber length on thermomechanical properties of short carbon fiber reinforced polypropylene composites. Mater Des 2009;30:260–3.
[2] Krairi A, Doghri I. A thermodynamically-based constitutive model for thermoplastic polymers coupling viscoelasticity, viscoplasticity and ductile damage. Int J Plast 2014;60:163–81.
[3] Ashby M, Jones DRH. An introduction to micro-structures, processing and design. In: Engineering materials. Pergamon Press; 2001.
[4] McCrum NG, Buckley CP, Bucknall CB. Viscoelasticity. In: Principles of polymer engineering. Oxford Science; 2003.
[5] El-Qoubaa Z, Othman R. Characterization and modeling of the strain rate sensitivity of polyetheretherketone's compressive yield stress. Mater Des 2015;66:336–45.
[6] Rae P, Brown E, Order E. The mechanical properties of poly(ether-ether-ketone) (PEEK) with emphasis on the large compressive strain response. Polymer 2007;48:598–615.

[7] Gaymans R, Hamber M, Inberg J. The brittle–ductile transition temperature of polycarbonate as function of test speed. Polym Eng Sci 2000;40:256–62.
[8] Davim J, Reis P, Lapa V, António C. Machinability study on polyetheretherketone (PEEK) unreinforced and reinforced (GF30) for applications in structural components. Compos Struct 2003;62(1):67–73.
[9] Garcia-Gonzalez D, Rusinek A, Jankowiak T, Arias A. Mechanical impact behavior of polyether–ether–ketone (PEEK). Compos Struct 2015;124:88–99.
[10] Jeng C, Chen M. Flexural failure mechanisms in injection-moulded carbon fibre/PEEK composites. Compos Sci Technol 2000;60:1863–72.
[11] Green S. Chapter 3 – compounds and composite materials. In: Andrew William, editor. PEEK biomaterials handbook. Elsevier; 2012. p. 23–48.
[12] Ramsteiner F, Theysohn R. Tensile and impact strengths of unidirectional, short fiber-reinforced thermoplastics. Composites 1979;10:111–9.
[13] Chukov D, Stepashkin A, Maksimkin A, Tcherdyntsev V, Kaloshkin S, Kuskov K, et al. Investigation of structure, mechanical and tribological properties of short carbon fiber reinforced UHMWPE-matrix composites. Compos Part B 2015;76:79–88.
[14] Hamdan S, Swallowe G. Crystallinity in PEEK and PEK after mechanical testing and its dependence on strain rate and temperature. J Polym Sci Pol Phys 1996;34:699–705.
[15] LATI High Performance Thermoplastic. Polyether–ether–ketone material properties, <http://www.lati.com>;2015.
[16] Adams R, Gaitonde J. Low temperature flexural dynamic measurement on PEEK, HTA and some of their carbon fibre composites. Compos Sci Tech 1993;47:271–87.
[17] Garcia-Gonzalez D, Rodriguez-Millan M, Rusinek A, Arias A. Investigation of mechanical impact behavior of short carbon-fiber-reinforced PEEK composites. Compos Struct 2015;133:1116–26.
[18] Evans WJ, Isaac DH, Saib KS. The effect of short carbon fibre reinforcement on fatigue crack growth in PEEK. Compos Part A 1996;27A:547–54.
[19] Sarausa J, Remiro P. The mechanical behaviour of PEEK short fiber composites. J Mater Sci 1995;30:3501–8.
[20] Gómez-del Río T, Zaera R, Barbero E, Navarro C. Damage in CFRPs due to low velocity impact at low temperature. Compos Part B 2005;36:41–50.
[21] Motz H, Schultz JM. The solidification of PEEK. Part I: morphology. J Thermoplast Compos 1989;2:248–66.
[22] Karger J, Friedrich K. Temperature and strain-rate effects on the fracture toughness of poly (ether ether ketone) and its short glass-fiber reinforced composite. Polymer 1989;27:1753–60.
[23] Solomon S, Bakar A, Mohd Z. Drop weight impact properties of (CO) injection molded short glass fiber/short carbon fiber/polyamide 6 hybrid composites. J Reinf Plast Compos 2007;26:405–18.
[24] Mortazavian Fatemi A. Effects of fiber orientation and anisotropy on tensile strength and elastic modulus of short fiber reinforced polymer composites. Compos Part B 2015;72:116–29.
[25] Garcia-Gonzalez D, Rodríguez-Millan M, Vaz-Romero A, Arias A. High impact velocity on multi-layered composite of polyether ether ketone and aluminium. Compos Interface 2015. http://dx.doi.org/10.1080/09276440.2015.1051421.
[26] Pettarin V, Elic GE, Frontini PM, Leskovics K, Lenkey GYB, Czigany T. Analysis of low temperature impact fracture data of thermoplastic polymers making use of an inverse methodology. Eng Fract Mech 2006;73:738–49.