



Low temperature effect on impact energy absorption capability of PEEK composites



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ARTICLE INFO

Article history:

Available online 28 August 2015

Keywords:

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

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 $-75\text{ }^{\circ}\text{C}$ to $+25\text{ }^{\circ}\text{C}$ 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.

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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 $-50\text{ }^{\circ}\text{C}$ to $+80\text{ }^{\circ}\text{C}$, 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

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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 $-75\text{ }^{\circ}\text{C}$ to $+25\text{ }^{\circ}\text{C}$ 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 $130 \times 130 \times 3\text{ mm}^3$. 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 $7\text{ }\mu\text{m}$ and $200\text{ }\mu\text{m}$ 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 (SFRTs) by flow molding techniques is that the fibers will tend to become aligned during the flow process, inducing

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

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.

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 INSTRON 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 $30 \pm 2\%$ was calculated for PEEK and $32 \pm 2\%$ 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

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