Study on Compression Moulding of Continuous Long Quartz Fiber-reinforced PEEK Composite Bar

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Abstract. The moulding process of 3D-braided PEEK/LQ prepreg is studied by optical microscope, scanning electron microscope and tensile testing machine in this paper. The mechanical property of composite with different fiber content is analyzed as well. The results show that a better impregnation of moulded PEEK resin to the continuous long quartz fiber is obtainedat the moulding temperature of 380℃.The internal porosity of composite bar decreases as the holding pressure increases. The densest microstructure and no hole is observed inside the composite bar after adding venting inmoulding process.The tensile and shear strength increase at first and then decrease as the quartz fiber content increases from 50% to 70%. The tensile and shear strength are maximum when the quartz fiber content is 60%.

Introduction

Polyether-ether-ketone, or PEEK for short, is linear aromatic macromolecular compound. The rigid benzene ring and flexible ether bond and carbonyl on the macromolecular chain can be seen from the molecular structure,as shown in Fig .1. PEEK is of good mechanical property, heat resistance (the continuous use temperature of 260ºC), corrosion resistance, dimensional stability[1,2]. As thermoplastic resin, PEEK can be formed by the way of extrusion, injection, mould pressing, melt spinning, rotational moulding, etc.

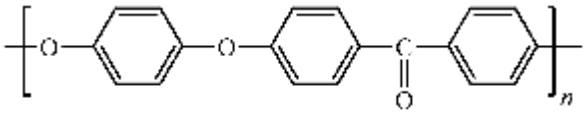

Fig .1 Molecular structure of PEEK

The property of tensile, impact, bending andcroop resistance and the thermal deformation temperature are all improved after PEEK is reinforced by fiberfill. The fiberfill often used in PEEK are carbon fiber, glass fiber and quartz fiber[3].

In recent years, it is becoming a hot point in foreign thermoplastic composite area to develop long fiber-reinforced PEEK, which has a broad application prospect in fields of aerospace, electronic and electrical, medical treatment, energy sources, electric power, machinery, automobile, dope, and so on as its mechanical property is obviously superior to short fiber-reinforced PEEK.

It is difficult to form good impregnation between fiber and resin matrix and make prepregowing to a high melting viscosity of PEEK[4]. In this paper, PEEK fiber and Long-Quartz(short for LQ) are knitted by the way of three-dimension(3D) braid process to preformthat is moulded to bar then, which solves the problem of impregnation between fiber and resin matrix. The effect of compression moulding process on microstructure and mechanical property of continuous long fiber composite material.

Experimental materials and methods

PEEK, Jilin University 021P resin, white granular, melting tempreture of 334ºC;Quartz fiber, Hubei Feilihua Quartz Glass Co., Ltd; Molding press, Y71-100.

PEEK fiber is made from PEEK granular after the processes of drying, heating, melting and spinning. The specification is 0.4mm×18 per single fiber and the linear density is 38.5tex.

The PEEK and quartz fiber are knitted to 3D five-directional braided composites preform with diameter of φ5mm, which is shown in Fig .2. The composite bar with diameter ofφ4mm made from preform is obtained after the processes of heating, compression moulding, heat preservation and cooling.

The transverse and vertical microstructure of φ4×10mm bar cut from the composite bar can be observed under Leica DMI5000M optical microscope after the process of mounting, rough grinding, accurate grinding and polishing. Hitachi S450scanning electron microscope is used to observe the impregnation of fiber and resin matrix after the bar specimen is cooled and brittle fractured in liquid nitrogen. The tensile and shear strength of specimen ofφ4×160mm cut from the composite bar is measured by CMT5504 universal tensile testing machine.

Fig .2 PEEK/LQ 3D-braided prepreg

Results and discussions

Effect of moulding temperature

The bar surface blackened after compression moulding at temperature of 400ºC, as shown in Fig .3(a). The reason analyzed was some degree of the resin thermal degradation caused by too high temperature of moulding, which was over melting points 56ºC. A large number of dispersed quartz fiber was found inside the bar on the scanning electron micrograph, asshown in Fig .4(a), which showed bad impregnation between PEEK and quartz fiber at 400ºC. There were two reasons analyzed. On the one hand, it was difficult for the PEEK resin to clad the quartz fiber as resin decomposed at high temperature and lost its original features. On the other hand, volatilizable gas generatedby the resin thermal degradation at the temperature of 400ºC could not vent in the process of moulding. The composite bar surface didn't blacked and was pale yellow at the moulding temperature of 380ºC, as shown in Fig .3(b), it was uniform and compact in microstructure, there were a lot of PEEK adhering to fiber, which showed that the combination and interface between the fiber and peek were good and high. The reason was that the melting PEEK resin flowed sufficiently without thermal degradation and cladded the quartz fiber well. The microstructure of composite bar moulded at 380ºCwas shown in Fig .4(b).

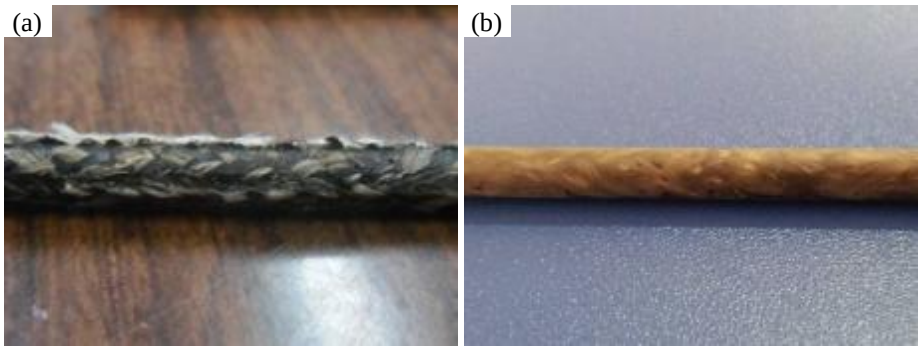

Fig .3 PEEK/LQ bar with different moulding temperatures
(a) 400ºC (b) 380ºC

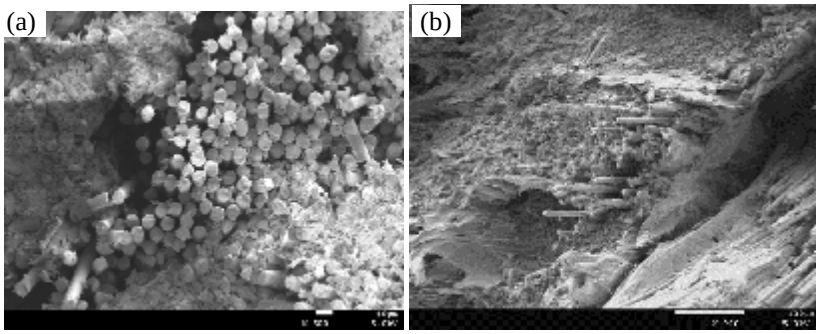

Fig .4 Microstructure of PEEK/LQ bar with different moulding temperatures
(a) 400ºC (b) 380ºC

Effect of mouldingpressure

Pressure-holding in moulding process can improve the microstructure compactibility of composites and the mechanical property of bars. The transverse microstructure of bar produced under pressure of 5MPa, 10MPa and 15MPa at the moulding temperature of 380ºC is shown in Fig .5, from which it is known that hole defect and dimension decrease as the pressure increases. In Fig. 5(a), Melting PEEK resin cannot dip quartz fiber well as a result of high viscosity and insufficient flow under pressure of 5MPa, It is easy to cause loose of PEEK composited products under low pressure, the material is not quite compact, this affects its mechanical performace. The hot-melt flowability of resin and the impregnation get better when pressure-held at 10MPa in Fig. (b). Though best impregnation is obtained under pressure of 15MPa, a few of holes are observed, which are observed in Fig. 5(c).

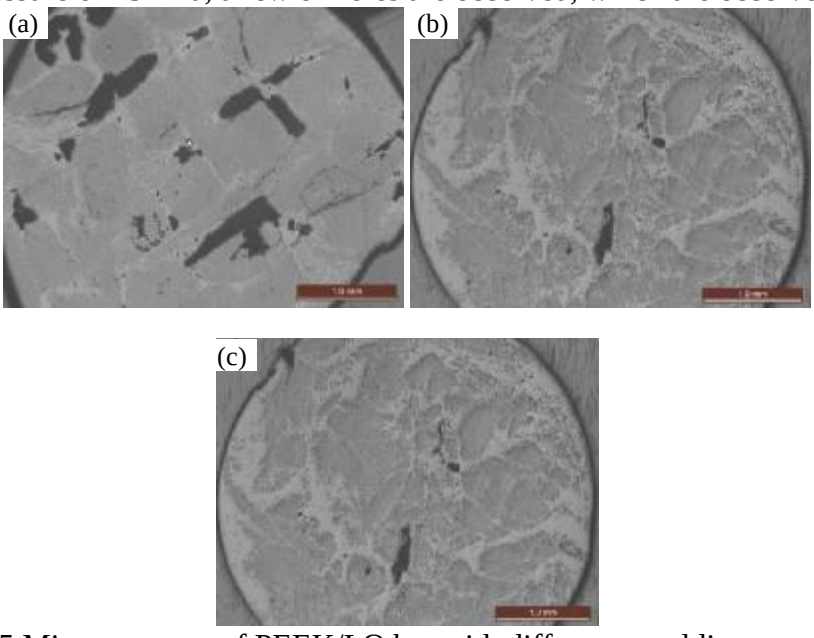

Fig .5 Microstructure of PEEK/LQ bar with different moulding pressures
(a) 5MPa (b) 10MPa (c) 15MP

Effect of venting

Internal air vent around as the upper and lower dies go down and up separately when moulding flat plate. However, moulding of composite bar is different. Venting process needs to be involved as the mould of bar surround the prepreg on every side. The surface resin of bar melts first on account of its higher temperature than inner resin, which makes it difficult for the air inside the cylinder to vent. Remained air inside the bar finally forms a hole. As is shown in Fig .5(c), a few of inner holes appear even if the pressure is 15Mpa.

When adding venting process before moulding, the microstructure of bar produced under the moulding temperature of 380℃ and the pressure of 15MPa is shown in Fig .6(a), from which compact transverse microstructure and no hole defect can be observed. Fig .6(b) and Fig .6(c) show the magnified transverse and vertical microstructures, in which every dark punctiform quartz fiber strand arranges closely and a mass of light PEEK resin fills among thembecause of long distance between every fiber strand. However, it is difficult to homogenize absolutely as determined by the specialty of 3D-braid process.It is observed that the impregnation is good through further electron microscopy of bar cross-section, as shown in Fig .6(d). The quartz fibers solidify as a whole by gluing of PEEK resin, which improves the mechanical property of bar.

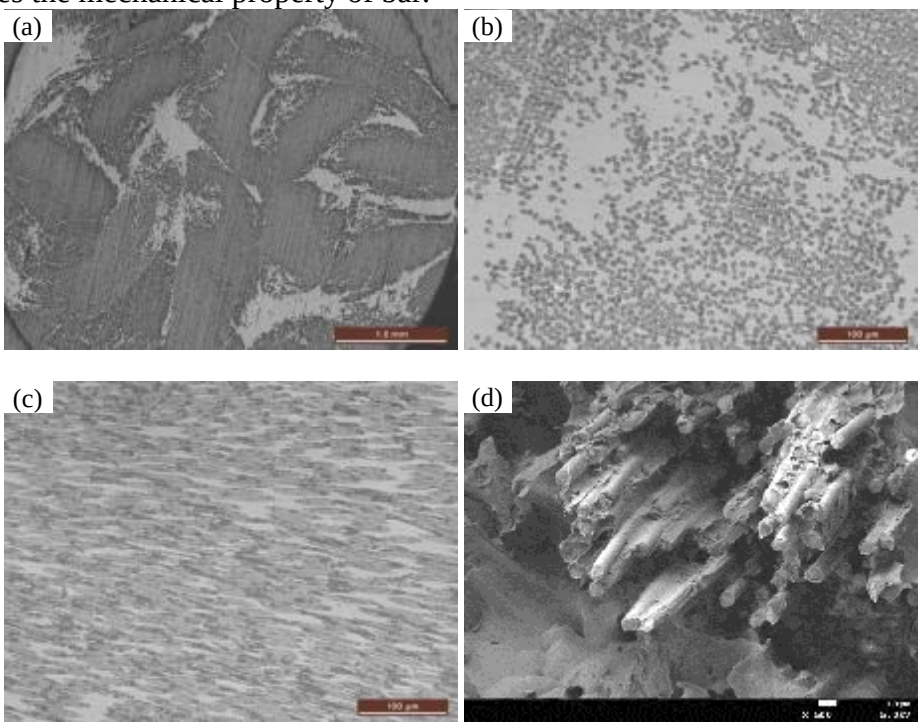

Fig .6 Microstructure of compostieafter adding venting process
(a) Section of OM (b) Crosswise of OM (c) Lengthways of OM (d) Fractograph of SEM

Effect of different fiber content to mechanical property

The tensile and shear strength of PEEK/LQ bar with different fiber content are listed in Table 1. The maximum tensile and shear strength is up to 628MPa and 306MPa separately while the strength of extension of short fiber-reinforced 30% C/PEEK composite bar is 130MPa[5]. The 3D braided composites is a whole fabric body, which have a high strength, It overcomes the disadvantage of low interlaminar shear strength and also slove the problem for thermoplastic resin impregnant, the level of shear strength is up to alumimum alloy. The strength of PEEK resin improves greatly through continuous long quartz fiber. The tensile and shear strength increase at first and then decrease as the quartz fiber content increases. The effect of fiber bearing capability and strength enhancing get better as the quartz content increases less than or equal to 60%. When the fiber content is over than 60%, reduced resin content results in worse adhesion to quartz fiber and mechanical property of composite bar. In conclusion, the optimum fiber content is 60%.

Table 1 Mechanical property of PEEK/LQ bar withdifferent fiber content

Number	Braiding technology	Quartz fiber content [wt.%]	Shear strength [MPa]	Tensile strength [MPa]
1	3D five-directional	50	252	483
2		60	306	628
3		70	272	537

Conclusions

1. The PEEK/LQ bar surface blackens after compression moulding at temperature of 400ºC as a result of resin thermal degradation and bad impregnation to quartz fiber. The composite bar surface is pale yellow and PEEK resin clad the quartz fiber wellat the moulding temperature of 380ºC.

2. The internal hole dimension and porosity of composite bar decrease as the holding pressure increases at moulding temperature of 380ºC. Though the densest microstructure is obtained under pressure of 15Mpa, a few of holes are observed.

3. Full impregnation from resin to quartz fiber leads to further decease of internal porosity of bar by adding venting inmoulding process. It is concluded that the optimum moulding process of PEEK/LQ composite bar is at the moulding temperature of 380ºC, pressure of 15MPa and involving venting.

4. The maximum tensile and shear strength of PEEK/LQ after braiding and moulding process is up to 628MPa and 306MPa separately.The tensile and shear strength increase at first and then decrease as the quartz fiber content increases to 50%, 60% and 70%. The strength is maximum when the quartz fiber content is 60%.

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