

On Studies of Tensile Properties in Injection Molded Short Carbon Fiber Reinforced PEEK Composite

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Both elastic modulus and strength of injection molded carbon fiber filled poly-ether-ether-ketone (PEEK) composite are studied under tension. The measured moduli and ultimate strengths of injection molded carbon fiber reinforced PEEK have been compared with the model predicted values. For injection molded PEEK composite, the experimentally obtained values of tensile modulus show a fair agreement but those of the tensile strength show a poor agreement with the theoretically predicted values. Many processing factors seem to be more critical issue for the strength than the stiffness of short-fiber reinforced composites. Considering the service performance of composites depends on three interactions - material, design and processing, monitoring the processing can be critical to have a best performance of composite. Processing factors have been discussed in cases of short carbon fiber reinforced PEEK composite based on the comparison between experimental and theoretically predicted data to obtain the best composite material.

Key Words: Short Carbon Fiber Reinforced PEEK Composite, Injection Molding, Tensile Strength, Processing Parameters

1. Introduction

Recent development in high-performance polymer composites has made it possible to offer advanced composites with superior weight-to-strength ratios compared to the conventional metal alloys. Especially carbon fiber reinforced plastics are being used increasingly in load bearing components such as aircraft components, pressure vessels and pipes. Therefore, the growing acceptance of polymer composites as effective load bearing and weight saving structural materials have brought about a need for development of technologies for efficient design and cost effective fabrication methods. The short-fiber composites, not as strong or as stiff as continuous fiber composites, do have several attractive characteristics that make them worthy of consideration for other applications such as automotive parts. Those are the possibilities of fabricating compo-

nents having complex geometrical contours with great versatility, fast and inexpensive processing methods for high-volume applications and isotropic behavior of parts. Nevertheless, the existing short-fiber composites have not been fully developed to yield optimum performance due to no clear understanding of processing parameters on the injection molded parts and no standard evaluation methods.

PEEK is currently the most studied semicrystalline, thermoplastic material of the kind. It is also one of the most selected as a matrix material for advanced composite materials with rich field for various product forms. The reasons for its popularity stem from its good thermal stability, excellent toughness, outstanding chemical and solvent resistance, and low flammability (Cogswell, 1992). Therefore, PEEK has been selected for this study since the properties of matrix resin are critical to each stage in the life of composites. Furthermore, the properties of polymer composites are strongly influenced by the properties of reinforcing materials, their types, their distribu-

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tion and interaction between the matrix and reinforcing materials. Among these factors affecting the properties of composites, one of the key elements having profound effects on the properties is the geometry of the reinforcing materials. The geometry of reinforcing material can be described as shape, size, aspect ratio and distributions (Kelly, 1973).

The properties of simple test specimens are often superior to those of actual molded parts. The main reason may be the molding conditions are different although the mold temperature and injection conditions appear to be same. Therefore, the effects of processing conditions should be understood in order to develop the materials with maximum performance. The properties of injection molded parts depend especially on factors such as flow pattern during molding, locations of weldlines, fiber orientations, packing density, thermal history and morphology. To obtain the maximum performance with optimum microstructure of short-fiber composites, the effects of processing conditions and the strength mechanisms should be understood. The first step is not only organizing the proper evaluation methods but also readjusting or setting a standard model that is currently not clear to apply as evaluation techniques. Several approaches have been proposed to develop theoretical models predicting elastic modulus and ultimate tensile strength of short-fiber composites in the literature. These approaches range from empirical modeling based on the experimental observations to sophisticated analytical treatments based on a microscopic point of view.

The objective of this study is to investigate the various theories of the elastic modulus and the strength of randomly oriented fiber reinforced composites, and to relate these models to the injection molded PEEK/carbon fiber composites. Also, a simple and reliable computational procedure is suggested to estimate composite properties and select a standard model for this composite system. Furthermore, it tries to understand the effects of processing conditions and microstructure on the mechanical properties of injection molded PEEK composites.

2. Experimental Procedures

The PEEK/carbon fiber composites specimens were made with 10, 20, 30 and 40 weight % fiber with fiber length of 1.0 mm manufactured by RTP Corp., Winone, Mn. U.S.A. for the present study. Young's modulus, tensile strength and Poisson's ratio were 3.6 GPa, 92.0 MPa, 0.4 for PEEK and 230 GPa, 3650 MPa, 0.20 for fiber respectively. The ASTM D638 tensile specimens with 2 mm thickness were used to measure the tensile properties of short-fiber composites. Strain gages were chosen to measure specimen modulus. The open-faced general purpose strain gage and M-bond 200 kit from Micromeritics Group Inc. were used in his study. The 2100 multichannel system by Measurement Group Inc. were used to measure strain with a bridge voltage of 1 volt. For flexural and shear properties, the bar specimens (1.26 cm \times 0.65 cm \times 12.7 cm) were used. Both moduli were measured using strain gage mounted in tension side for flexure and mounted ± 45 degree on the modified Iosipescu sample for shear modulus (Lee et al., 1995).

Both types of specimens were molded on an ARBURG 221E/150 injection molding machine. Molding conditions were :

Temperature Settings,	
At barrel-rear	354°C
-middle	371°C
nozzle	377°C
mold	191°C
pressure Setting,	
Injection pressure	68 MPa
Time Settings,	
Injection	5 sec
Clamp	15 sec
Total Cycle	18 sec

Since the mechanical properties of the semicrystalline matrix material depend on microstructural details such as spherulites size, orientation, degree of crystallinity, and degree of transcrystallinity that depend on the processing conditions, the same molding conditions were kept for all sam-

ples.

All tests were done using an Instron universal testing machine in room temperature and relative humidity of 50%. A testing speed was 1.25 mm/min as a recommended testing speed for many polymeric materials. Typically, five specimens were used for a single evaluation.

3. Results and Discussion

3.1 Models for elastic modulus of short-fiber composite

Among the many models predicting elastic modulus of short-fiber composites from empirical modeling based on the experimental observations to sophisticated analytical treatments based on a microscopic point of view, some of them will be discussed. The emphasis will be on the two-dimensional case in this study.

The simplest models are those which make use of the rule of mixtures (combining rules). Voigt assumed that each component was subject to the same strain (isostrain), giving,

$$E_c = E_f V_f + E_m V_m$$

Alternately, Reuss assumed that each phase was subject to the same stress (isostress), giving,

$$E_c = E_m E_f / (E_m V_f + E_f V_m)$$

where E denotes modulus and V volume fraction, and the subscripts c , m and f represent composite, matrix resin and fiber, respectively.

Hori and Onogi (1951) proposed the following:

$$E_c = (E_{11} E_{22})^{1/2}$$

where E_{11} and E_{22} are the moduli in the machine and cross-directions for machine-made paper. For short-fiber composite, one can use $E_{11} = E_c$ from Voigt model and $E_{22} = E_c$ from Reuss model.

Cox (1952) who used a shear lag formulation to model the longitudinal elastic modulus showed that the modulus of short-fiber composites can be expressed as:

$$E_c = (1/5)E_{11} + (4/5)E_{22}$$

where E_{11} and E_{22} are the same as previously defined.

Piggot (1980) suggested the modulus for composites having fibers which are random in three dimensions as

$$E_c = (1/5) V_f E_f + V_m E_m$$

Lavngood and Goettler (1987) established a general procedure for predicting the average Young's modulus for randomly oriented short-fiber composites. When the fibers are two dimensionally oriented, they derived the Reuss-type expression as:

$$E_c = 24 E_{11} E_{22} / (7 E_{22} + 17 E_{11})$$

where $E_{11} = E_m + V_f(E_f - E_m)$

$$E_{22} = E_m \{ [2 V_f (R - 1) + (R + 2)] / [V_f (1 - R) + (R + 2)] \}$$

in which E_m and E_f are Young's moduli of the matrix and fiber, respectively. V_f is the volume fraction of the fiber; R is the ratio of transverse fiber modulus to matrix modulus.

Tsai and Pagano (1968) showed that the modulus of short-fiber composites can be predicted approximately by:

$$E_c = (3/8)E_{11} + (5/8)E_{22}$$

where the Halpin-Tsai equations for longitudinal (E_{11}) and transverse (E_{22}) moduli of aligned short-fiber composites can be written as

$$\frac{E_{11}}{E_m} = \frac{1 + (2l/d)\eta_L V_f}{1 - \eta_L V_f}$$

where $\eta_L = \frac{(E_f/E_m) - 1}{(E_f/E_m) + 2(l/d)}$

and

$$\frac{E_{22}}{E_m} = \frac{1 + 2\eta_T V_f}{1 - \eta_T V_f} \text{ where } \eta_T = \frac{(E_f/E_m) - 1}{(E_f/E_m) + 2}$$

Christensen and Waals (1972) used the average approach to find the isotropic elastic constants for fiber composites with 2 and 3 dimensional random fiber orientation. For two-dimensional case the results are,

$$E_c = \frac{1}{U_1} (U_1^2 - U_2^2)$$

where $U_1 = \frac{3}{8}E_{11} + \frac{G_{12}}{2} + \frac{(3 + 3\nu_{12} + 3\nu_{12}^2)G_{23}K_{23}}{2(G_{23} + K_{23})}$

$$U_2 = \frac{1}{8}E_{11} - \frac{G_{12}}{2} + \frac{(1 + 6\nu_{12} + \nu_{12}^2)G_{23}K_{23}}{2(G_{23} + K_{23})}$$

For the 3-dimensional case (Christensen, 1979),

Table 1 Mechanical properties of injection molded composites

	Carbon fiber volume fraction			
	40 %	30 %	20 %	10 %
Strength (MPa)				
Tension	239.3	220.8	190.4	161.2
Compression	271.7	246.2	219.4	202.4
Flexure	318.6	301.4	280.1	260.4
Shear	119.7	109.9	98.4	84.7
Modulus (GPa)				
Tension	31.1	23.2	16.2	9.8
Compression	52.0	43.5	34.1	21.8
Flexure	22.8	16.6	10.8	6.2
Shear	6.3	5.7	4.3	3.3
Elongation at Break (%)	1.3	1.8	2.1	2.3
Poisson's Ratio				
Tension	0.37	0.38	0.382	0.39
Compression	0.372	0.387	0.385	0.396

modulus of randomly oriented fiber composite is

$$E_c = \frac{[E_{11} + (4\nu_{12}^2 + 8\nu_{12} + 4)K_{23}][E_{11} + (4\nu_{12}^2\nu_{12} + 1)K_{23} + 6(G_{12} + G_{23})]}{3[2E_{11} + (8\nu_{12}^2 + 4\nu_{12} + 7)K_{23} + 2(G_{12} + G_{23})]}$$

where the transverse bulk modulus, K_{23} , the longitudinal shear modulus, G_{12} , and the transverse shear modulus, G_{23} , can be obtained from the results of Hill (1964) and Hashin (1962).

Weng and Sun (1979) used the Christensen-Waals equations along with micromechanics equations that were modified to account for the effect of fiber length. The effect of fiber length was modeled using a so-called "fictitious fiber", which included the effect of matrix material at the ends of the fiber. This model is not compared in this work since the values show small differences between them. Halpin and Kardos (1978) predicted the stiffness of short-fiber composite using laminate theory. This complex model gives a similar result as Hori & Onogi (1951) and also not discussed in this paper.

Typical stress-strain curves of the tested materials are shown in Fig. 1. Their mechanical properties under tensile, compressive, flexural and shear loadings are shown in Table 1. The stan-

dard deviation for the moduli is less than 7 % and the standard deviation for the tensile strength is less than 15 %. Figure. 2 shows SEM micrograph of the typical fracture surface with 30 % fiber content under tensile loading. The measured moduli under various loadings are compared in Fig. 3.

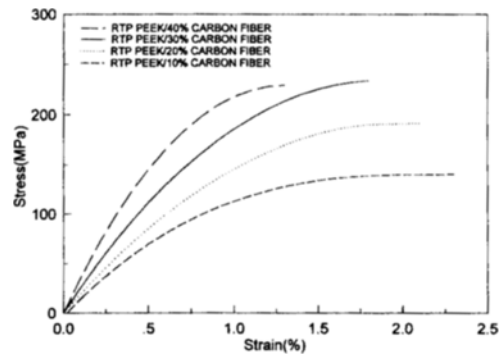


Fig. 1 Stress-strain behavior of PEEK/carbon fiber composites.



Fig. 2 Failed Surface with 30 % Carbon Fiber Content.

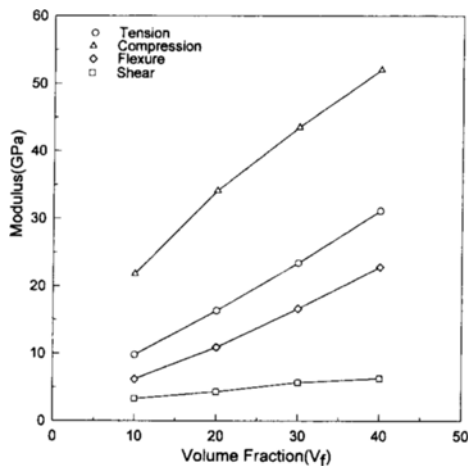


Fig. 3 The measured moduli under tensile, compressive, flexural and shear loadings.

Figure 4 shows the comparison between the predicted moduli by various theoretical models and experimentally measured moduli of short carbon fiber reinforced PEEK composite as a function of fiber concentration. The moduli increase with increasing fiber content as expected. Some models are in fair agreement with the experimental data at low fiber volume fraction. As shown in the figure, the predicted values of Tsai and Pagano model (1968) and Christensen and Waals model (1972) show a fair agreement with experimental data of this composite system.

For the elastic tensile moduli of short-fiber composites, there is no single theory satisfactorily describing the elastic properties of all short-fiber composites. Considering that each model has a

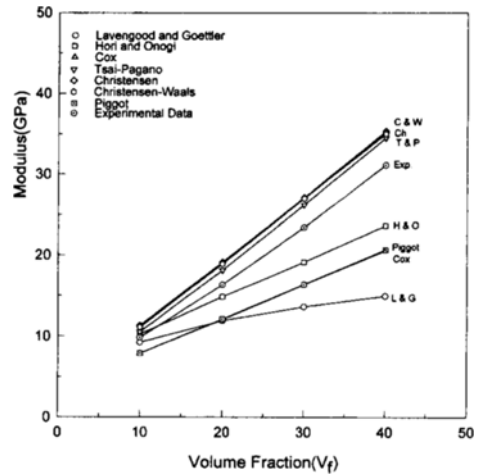


Fig. 4 Comparison between model predicted moduli and experimentally measured moduli.

reasonable agreement for a certain composite, many factors such as fiber distribution, interface/interphases, aspect ratio, etc. should be reconsidered not only to have a maximum performance material but also to monitor the processing technique. Those will be discussed later. However, both models by Tsai & Pagano and Christensen & Waals should be processing criteria for carbon fiber/PEEK composite. Especially, since Tsai and Pagano model even takes the aspect ratio (fiber length/fiber diameter) into consideration, this model is recommended to study the molding operations and material parameters.

3.2 Models for strength of short-fiber composites

Unlike continuous composites, short-fiber composites with randomly oriented fibers are close to isotropic materials, i.e., macroscopic properties are approximately equal in all directions. For the purpose of predicting the strength of short-fiber composites, various models have been proposed in the literature. Many of those models were developed through theoretical analyses based on micromechanics with simplified assumptions and idealization of complex phenomena. Thus there are limitations of applicability of those models, whereas some models were modified with empirical parameter to improve the accuracy of the model.

Most of short-fiber composite strength models were developed by modifying the rule of mixtures. Kelly and Tyson (1965) modified the contribution of fiber strength based on the assumption that plastic flow will occur during stress transfer between matrix and fibers, giving,

$$\sigma_c = \sigma_f V_f (1 - l_c/2l) + V_m \sigma_m$$

where σ_c is ultimate tensile strength of composite ; σ_f , σ_m , strengths of fiber and matrix, respectively ; and l , l_c , fiber length and critical length of fibers.

Piggot (1980) accounted for both plastic and elastic effects in the matrix in his fiber strength theory. Piggot's composite strength model is expressed by lengthy equations that will not be presented here. For composites having fibers which are random in three dimensions, he also suggested an upper strength bound as

$$\sigma_c = (1/5)\sigma_f V_f + V_m \sigma_m$$

Vinson and Chou (1975) modified the rule of mixtures of continuous fiber composite for the strength of short-fiber composites and derived the following equation :

$$\sigma_c = \sigma_f V_f F(1/l_c) + \sigma_m (1 - V_m)$$

where F is a factor which takes into account the effect of fiber length. At high aspect ratios $l \gg l_c$, it is reasonable to expect that the fibers act as a continuous reinforcement and $F(1/l_c)$ should approach its limiting value of 1, in agreement with shear lag analysis predictions. However, finite element analyses (Chen, 1971) seem to indicate upper bound as low as 0.5 for the function $F(1/l_c)$ at high fiber aspect ratios. In this study, strength is calculated with $F(1/l_c) = 0.5$.

Riley (1968) considered interaction between fibers by taking into consideration of the stress transfer between fibers in a rationalized fiber array such as a hexagonal arrangement, and derived a strength equation as :

$$\sigma_c = \left(\frac{6}{7}\right) \frac{\sigma_f V_f}{1 + (5l_c/7l)} + \sigma_m (1 - V_f)$$

where the critical fiber l_c is determined from three different theories summarized by Robinson and Robinson (1994). But the obtained values are basically the same.

Since there are variations not only in fiber

length but also in fiber orientation for actual short-fiber composites, the rule of mixtures is modified to (Curtis et al., 1978)

$$\sigma_c = \sigma_f V_f F(1/l_c) C + \sigma_m (1 - V_m)$$

where the fiber orientation factor, C , has been determined by experiments. Based on their experimental works, C can be 0.36 for $V_f = 10\%$ and $C = 0.43$ for $V_f = 40\%$ respectively. For a random array of fibers, Cox (1952) classical shear lag analysis leads to $C = 1/3$ and $C = 1/6$ in two and three dimensions, respectively. In this study, C can be in the range of 0.37 for low (10 and 20%) and 0.3 for high fiber volume fractions. Although theoretical estimations of C have been reported (Fukuda and Chou, 1982), it seems quite improbable to consider all effects of fiber orientation.

Fukuda and Chou (1982) developed a composite strength theory based on fiber failure in the composite due to high stress, and stress concentration at the ends of the fibers is one of the contributing factors to cause the high stress. They used a probabilistic approach to account for the fiber ends in a critical control zone :

$$\sigma_c = \sigma_f V_f P + \sigma_m (1 - V_f)$$

where P is a factor which account for the probabilities of finding fiber gaps in the control zone. This model is not considered for comparison due to difficulty in determining P .

With different aspects, Hori and Onogi (1951) developed an empirical strength model for the properties of paper :

$$\sigma_c = (\sigma_l \sigma_t)^{1/2}$$

where the subscripts l and t denote longitudinal and transverse directions, respectively. In this study, both tensile strengths were obtained as $\sigma_l = \sigma_f V_f + E_m e_f V_m$ and $\sigma_t = (E_{22} \sigma_m) / (E_m F)$ where F is the strain concentration factor (Gibson, 1994) for randomly oriented short-fiber composite.

Hahn (1975) proposed a random fiber composite strength model that equals to the average of off-axis strengths of unidirectional composites if the failure is gradual and if the rule of mixtures is applicable for the elastic modulus,

$$\sigma_c = \sigma_t (4/\pi) (\sigma_l / \sigma_t)^{1/2}$$

Similarly, the subscripts l and t denote the tensile

strengths parallel and perpendicular to the fiber direction, respectively. In this study, the same equations as above are used.

Lees (1968) derived the following equation by assuming three failure mechanisms according to the maximum stress criterion for randomly oriented short-fiber composites :

$$\sigma_c = (2\tau/\pi) \{1 + \sigma_t/\sigma_m + \ln(\sigma_t\sigma_m/\tau^2)\}$$

where τ is the in-plane shear strength.

Chen (1971) used the same approach based on Von Mises-Hencky material failure criteria with perturbation effect near the fiber using finite element method :

$$\sigma_c = (2\tau/\pi) \{2 + \ln(\phi \sigma_r\sigma_m/\tau^2)\}$$

where ϕ is a strength efficiency factor which is characteristic of each type of discontinuous system and σ_r is the strength of equivalent unidirectional fiber composite. However, due to difficulty in measuring in-plane shear strength accurately, the last two methods were not calculated in the present study. Several other authors also have reported short-fiber composite strength models in the literature including Halpin and Kardos (1978) who used the laminate analogy to approximate the strength of short-fiber composites.

Figure 5 shows the measured strengths under tensile, compressive, flexural and shear loadings. Figure 6 shows the comparison between the predicted strengths by various theoretical models

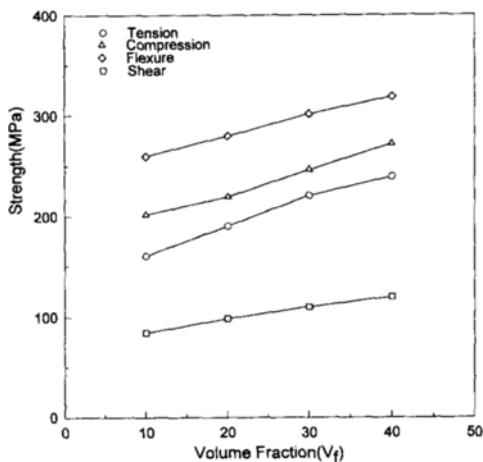


Fig. 5 The measured tensile strengths under various loading conditions.

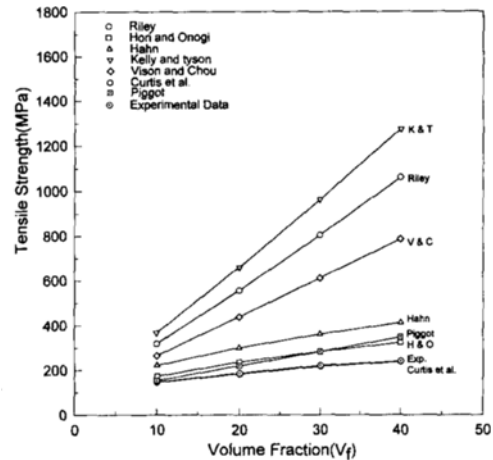


Fig. 6 Comparison between model predicted strengths and experimentally measured tensile strengths.

and experimentally measured strength of short-fiber PEEK composites as a function of fiber concentration. The strength also increases with increasing fiber content, but most models are in poor agreement with the experimental data especially at high fiber volume fractions. As shown in the figure, the experimentally obtained values are lower than all predicted values. However, Piggot (1980), Hori and Onogi (1951) and Curtis et al. (1978) strength models show a relatively better agreement with experimental data of short carbon fiber reinforced PEEK composite.

In order to investigate the reason for this poor strength compared with most models, the micrograph of sample is examined and found that the fibers are poorly distributed in the pellets. Many resin rich areas and conversely fiber rich areas with dry fibers are observed. The resin rich areas represent a potential source of weakness and fiber rich areas represent poor stress transfer due to possible poor wetting of matrix. Thus the poor strength may be attributable to the non-uniform distribution of the fibers resulting the poor interfacial strength between fiber and matrix. As known for the most composite system, the processing temperature and fiber volume fraction are important as fiber distribution and processibility of these compounds. For instance, high fiber fractions generally give difficulty in obtaining

void free processing. And low translation of the inherent stiffness and strength of the fiber are achieved: there is little gain in mechanical performance to offset the processing difficulties. By contrast, low fiber fractions decrease the mechanical performance in proportion to the fiber fraction and, if excess resin is present, controlling resin fiber distribution can be a problem. At this time I know of no theoretical analysis and experimental investigation that express the optimum fiber content for this composite system. Also, voids can be related with fiber distribution. Of course, fiber content and crystallinity affect density independently of void content. In this work, voids are observed both in the matrix and interface as shown in Fig. 2. Therefore, the low strength for high fiber fraction can be due to not only non-uniform distribution of fibers but also the voids. Among many possible ways, this can be overcome by controlling the molding conditions a little bit and by having a better preimpregnation of materials.

As the resin-fiber distribution in a composite is non-uniform, so will be the internal stresses and the constraint imposed by the solidification taking place as a result of cooling. The surface layers of a molding are first solidified. They will be placed in compression by the subsequent shrinkage of the internal core and will themselves represent a tensile constraint on that part of the molding. One major area of concern associated with internal stress is the possibility of microcracking. These factors can be critical if the specimen size or components are small. The samples that tested in this work have a cross-section of 6 mm width and 2 mm thickness. This size can be a problem. Therefore, it is recommended that the standard test sample for this type of study should be bigger than the present ASTM recommended size to have a more stable and reliable results. The change in fiber volume fraction is also significant when the internal stresses within the composite due to temperature changes are considered. Such knowledge is also not readily available and should be investigated experimentally.

Close controls of the interface between the matrix and the reinforcement are vital to ensure

the full translation of the inherent stiffness of fibers into forming stability of a composite structure. A variety of mechanisms have been proposed to account for adhesion within the fiber-matrix interphase. These include wetting, chemical bonding, mechanical and crystalline interlocking. Much of the art of manufacturing high quality thermoplastic composites lies in exploiting these mechanisms. It is known (Cogswell, 1992) that PEEK and carbon fiber are generally achieving good adhesion. Also, the good adhesion with characteristics of ductile fracture was observed in fractography as shown in Fig. 2. So, it can be assumed that the interface of this system is not the main cause of poor strength values.

No less significant is the local organization of the short fibers in the matrix, both in terms of longitudinal distribution and orientation. The fiber orientation can be another factor that has a significant effect on composite strength. Previous works (Lee & Chin, 1990 and Chung & Kwon, 1995) show relatively uniform fiber orientation along the specimen's length in practical injection molded tension samples of short-fiber reinforced thermoplastic. Figure 2 also supports this assumption showing the fractured fibers perpendicular to fracture surface. Even though the high utilization of the fibers by their strong collimation in the model predicted values have higher number than the experimental values of this system. Considering this, fiber orientation can not be the only cause of poor strength for this composite.

Despite the efforts of those researchers, there are no theories satisfactorily describing the tensile strength of this short-fiber composite. Also, at this time I know of no theoretical analysis that expresses the optimum fiber content and no appropriate processing parameters to have a maximum utilization of this composite material, and must therefore accept the judgment of experience. However, for this short carbon fiber reinforced PEEK composite, both fiber distribution and the voids as the most important factors should be closely studied and controlled to have appropriate mechanical properties with the given ingredients. A suitable processing window for this system can be

obtained by performing more works by changing the molding conditions.

4. Conclusion

Both the measured tensile modulus and ultimate tensile strength of injection molded short-fiber reinforced PEEK have been measured and compared with the theoretically predicted values. From this study, the following conclusions can be made ;

(1) The experimentally obtained values of tensile moduli show a fair agreement with the predicted values of Tsai and Pagano model, and Christensen and Waals model of both 2-D and 3-D. Since Tsai and Pagano model allows to consider the aspect ratio of filler, it is recommended to apply this model for monitoring the processing conditions and material parameters for this composite system.

(2) The measured values of the ultimate tensile strength show a poor agreement with the many theoretically predicted values. To use the strength model as a selection criterion for the best processing operations, models proposed by Piggot, Hori and Onogi, and Curtis et al. should be considered. As expected, many processing factors are found to be more critical issue for strength than stiffness of short-fiber reinforced composites.

(3) Many processing factors such as fiber distribution, voids, interface/interphase, residual stress and fiber orientation, have been discussed in cases of short carbon fiber reinforced PEEK composites to have an optimum material. Both fiber distribution and voids are the critical parameters to be investigated first for injection molding.

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