Reinforced Effect of Wollastonite on Phenolphthalein Poly(ether ketone)

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ABSTRACT: The mechanical properties of wollastonite-filled phenolphthalein poly-(ether ketone)(PEK-C) composites have been studied at room temperature and 200°C. The dispersion of wollastonite particles in PEK-C matrix were investigated by means of scanning electron microscope. The modulus and strength of the composites increased with filler content. The reinforced effect of wollastonite on PEK-C is more marked at elevated temperature. The glass transition temperature of the composites is higher than that of PEK-C and is independent of filler content. The restriction effect of filler particles on the molecular mobility of the polymer matrix should be attributed to the reinforcement. © 1997 John Wiley & Sons, Inc. J Appl Polym Sci **65:** 649–653, 1997

Key words: phenolphthalein poly(ether ketone); wollastonite; composite; mechanical property; reinforcement

INTRODUCTION

Particulate reinforced thermoplastic composites have received considerable attention for many years. Great efforts have been taken on polyethylene, polypropylene, and nylon based composites for exploiting new engineering material with the combination of unique properties and low cost.[1–4] These results describe the relationship of mechanical properties and structure at room temperature. In general, the addition of inorganic filler into the polymer matrix offers both benefits, such as modulus and heat resistance, and disadvantages, such as toughness and melt viscosity.

Adequate physical properties at elevated temperature are required for high-performance thermoplastics to ensure that such thermoplastics can meet high-performance and cost requirements. Phenolphthalein poly(ether ketone)(PEK-C) is an amorphous high-performance thermoplastic and has been regarded as a potential engineering plastic or as a matrix for advanced composites. Among inorganic fillers, wollastonite provides many processing and performance benefits, including increased stiffness and strength, improved heat distortion temperature, low coefficient of expansion, ease of processibility, and good reinforcement at considerably lower cost.[5] The aim of this article is to further understand the reinforcement effect of inorganic filler on the polymer, especially at elevated temperature.

EXPERIMENTAL

Materials

The PEK-C used in this study was supplied by Xuzhou Engineering Plastics Co., People's Republic of China. Its reduced viscosity in chloroform at a temperature of 25°C is 0.46 dL/g. The original powders were dried at 140°C for 5 h in order to remove moisture.
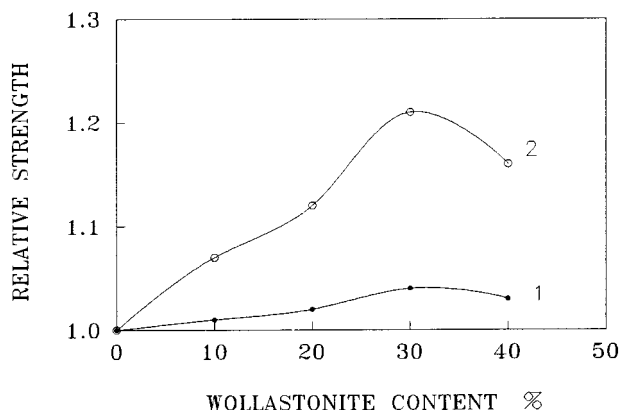
Figure 1 Relative flexural (2) and tensile strength (1) of PEK-C/W composites at room temperature as a function of wollastonite content.

The wollastonite was supplied by Dadingshan Wollastonite Mine, People's Republic of China. It has an acicular particle shape with a 35-μm top size and an aspect ratio of ~5.

Sample Preparation

The PEK-C and wollastonite powder were mechanically mixed in a high speed mixer with compositions ranging from 10 to 40 wt % wollastonite in PEK-C matrix. The unfilled PEK-C and composites were then extruded at ~ 300–330°C in a SHJ-30 twin-screw extruder and pelletized. All specimens were injection molded to the standard specimen size in accordance with ASTM D638 and D790.

Mechanical Testing

The tensile and flexural properties were measured with an INSTRON 1121 type tester according to ASTM D638 and D790, respectively. The crosshead speeds were 5 and 20 mm/min, respectively. Tests were carried out at room temperature and 200°C.

Glass Transition Temperature

A DuPont 9900 dynamic mechanical analyzer (DMA) was used to measure the thermal behavior at a heating rate of 3°C/min.

Morphology Observation

The morphology of composites was observed using a scanning electron microscope (SEM) (Model JAX-840).

RESULTS AND DISCUSSION

Mechanical Properties at Room Temperature

Mechanical properties of PEK-C/W composites were examined first at room temperature and are presented in Figures 1 and 2. The relative property is the ratio of property of the composite to that of the unfilled PEK-C. The flexural strength and tensile strength of PEK-C matrix are 147.6 MPa and 100 MPa, respectively. The flexural strength and modulus of composites increase with increasing the addition of wollastonite, whereas the tensile strength increases gradually. The flexural strength exhibits a maximum value, with an increase of 21.3%, at 30% content of wollastonite. It is apparent that the wollastonite is favorable for improving flexural strength and modulus. Also, from the viewpoint of reinforcement, the most suitable content of wollastonite in PEK-C is 30% in weight.

In disagreement with our results, inorganic filler does not enhance the tensile strength for some crystalline polymer, such as polypropylene[1,2] and nylon.[3] For polypropylene/filler composites,[1,2] tensile modulus increased with filler concentration, while tensile strength decreased. The same result can also be seen in ref. 3 for nylon 6/wollastonite with 50% untreated wollastonite, in which the tensile strength is slightly lower, while the flexural strength is higher than that of nylon 6 matrix. On the other hand, for high density polyethylene (HDPE)/mica composites[4] and nylon 6/W[3] with interfacial modifiers by silane or ti-
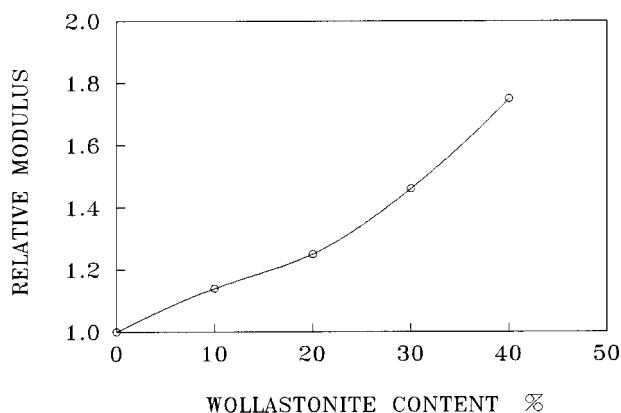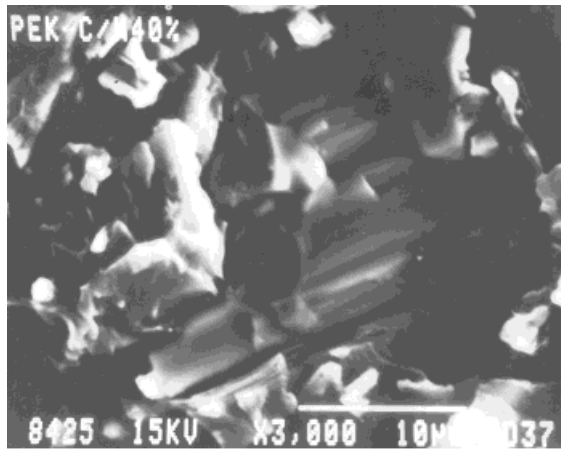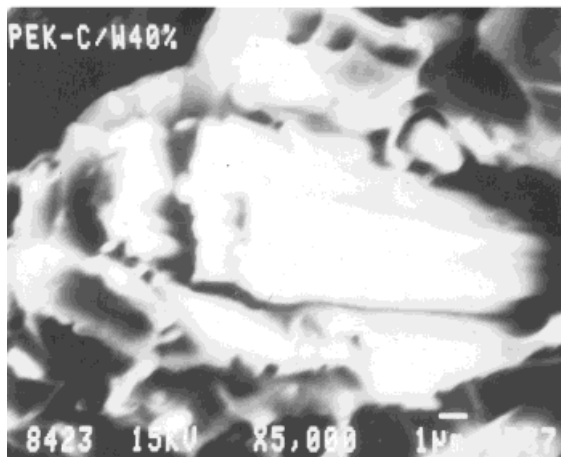

Figure 2 Relative flexural modulus of PEK-C/W composites at room temperature as a function of wollastonite content.

(a)

(b)

Figure 3 SEM photographs of PEK-C/W composites.

tography," is useful for clarifying the fracture mechanism.[6] The fracture surfaces of the specimens were examined and photographed by SEM subsequent to the mechanical testing. Some typical morphologies that can reveal the agglomeration phenomenon of wollastonite particles are shown in Figure 3(a,b). The agglomeration of wollastonite particles should be the initiation point of fracture for the composites, and therefore lower the mechanical strength. Also, the agglomeration phenomenon will result in decreasing the interfacial area, and as a result the stress transmission and strength estimated at large deformations. On the other hand, the modulus of composites, determined at low solid displacement or low deformations, are not affected by agglomeration.[1] Therefore, the decreasing strength value when wollastonite content exceeds 30% should be attributed to the agglomeration phenomenon. This also confirms that the interfacial adhesion between wollastonite particles and the PEK-C matrix is not very strong. Further work on improving adhesion is need.

Mechanical Properties at Elevated Temperature

PEK-C is a high performance thermoplastic; its advantage is the excellent properties at elevated temperature. Therefore, studying the mechanical properties of composites at elevated temperature is necessary. Figure 4 shows the relative flexural strength and tensile strength of PEK-C/W composites measured at 200°C. Figure 5 gives the relative flexural modulus of composites at 200°C. One can see that these values increase signifi-
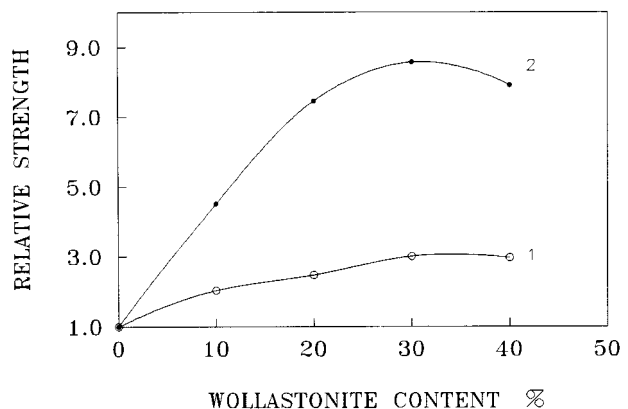
tanate, the flexural and tensile strength increase slightly with increasing filler content. It is evident that the interaction between polymer matrix and filler has an important role in improvement of mechanical properties.

The polymer/filler interface area, and as a result the interaction forces, are expected to increase with increasing the wollastonite content. Thus, the strength of PEK-C/W composite is expected to increase with increasing the wollastonite content. Meanwhile, the opportunity of agglomeration of wollastonite particles will increase for composites with large content of wollastonite in the absence of any modification on filler surface.

The SEM observation, often called "frac-

Figure 4 Relative flexural (2) and tensile strength (1) of PEK-C/W composites at 200°C as a function of wollastonite content.

cantly with increasing wollastonite content. The tensile and flexural strength also exhibit their maximum value at 30% content of wollastonite. For the composite with 30% wollastonite content, the flexural strength and tensile strength increase eight and threefold, respectively, over that of PEK-C matrix. It is obvious that the reinforcement of wollastonite on PEK-C at high temperature is more marked than at room temperature.

It is well known that for any significant improvement in mechanical properties by the filler in the composites, two important factors are the continuity in the structure (dispersion and content of filler), and the interfacial adhesion (filler type, modification, etc.). For the case without any interfacial modifier (or poor interfacial adhesion), the improvement of mechanical properties should be attributed to the restriction on the molecular mobility of the polymer imposed by filler particles. This is in accord with the well-known fact that inorganic filler or fibers represents a restriction to the molecular segmental mobility.[7,8] This restriction will be at its maximum in the immediate vicinity of the fillers, and only at large distances from the fillers will the properties of the polymer become equal to those of the bulk material. In addition, this restriction effect has an intrinsic relationship with the segmental mobility of polymer matrix. Therefore, with increasing the temperature, the restriction of wollastonite to the molecular segmental mobility becomes stronger. As a result of this, the reinforcement of wollastonite on PEK-C at elevated temperature is more marked than that at room temperature.

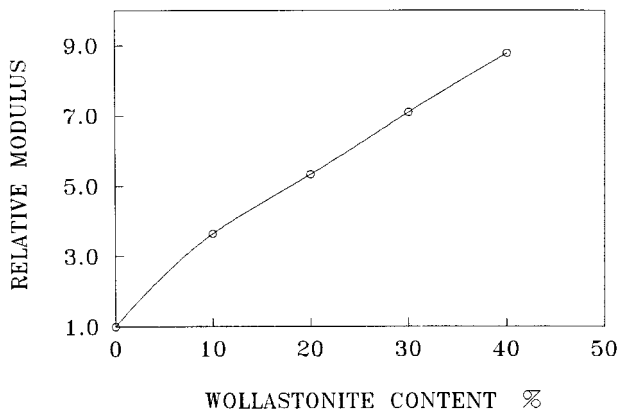To optimize the reinforcement, it is necessary to render the surfaces of the filler and polymer

Table I T_g **of PEK-C/W Composite**

Wollastonite Content (%)	T_g (°C)
0	225.9
10	231.2
20	230.1
30	231.8
40	232.3

matrix compatible. To achieve this aim, the most commonly used reagents are coupling agents and wetting agents. Unfortunately, it is difficult to find such an agent suitable for PEK-C due to its high processing temperature. For glass fiber reinforced PEK-C, blending poly(phenylene sulfide), which acts as a coupling agent, was found to be an effective method for improving the fiber/polymer interfacial adhesion.[9] It is believed that, for high performance thermoplastic/filler composites, coating a layer of polymer is a promising method for optimizing the reinforcement. The work on this subject will be seen later.

Glass Transition Temperature

The glass transition temperature (T_g) of PEK-C/W composite was determined by a DMA method and is shown in Table I.

The T_g of PEK-C/W composites is increased by 5°C with respect to that of the PEK-C matrix. This increase is independent of wollastonite content due to the fact that it is less sensitive to T_g in the presence of filler.[10] This result may be considered as further evidence of the restriction of filler to the PEK-C matrix.

CONCLUSIONS

Incorporation of wollastonite increases the tensile strength, flexural strength, flexural modulus, and glass transition temperature of PEK-C. There exists a suitable filler content, which is 30%, for the reinforcement of strength.

The wollastonite is favorable for improving flexural modulus, whereas for tensile strength and flexural strength the improving effect is relatively lower. The reinforcement is more marked at elevated temperature. The restriction of filler particles on the molecular mobility of polymer matrix should be attributed to the reinforcement.

Figure 5 Relative flexural modulus of PEK-C/W composites at 200°C as a function of wollastonite content.

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