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Interrelation of Scanning Electron Micrographs with Flexural Behavior of Ferrite and Nano-Barium Titanate Reinforced Poly-Ether-Ether-Ketone (PEEK) Composites

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Abstract: The flexural behavior of ferrite filled poly-ether-ether-ketone (PEEK) composites, with and without reinforcement of nano-barium titanate, was studied and was corroborated through scanning electron microscopy (SEM). In this study, ferrite filled PEEK, and ferrite and nano-barium titanate reinforced PEEK composites were prepared. Ferrite filled PEEK composites showed reduction in flexural strength and increase in flexural modulus with the increase in ferrite content, whereas, with the reinforcement of nano-barium titanate, flexural strength increased and flexural modulus decreased at similar ferrite content. The SEM micrographs corroborated well with flexural behavior, as ferrite particles and smooth topographic surfaces of brittle fracture were evident in the samples having higher ferrite content in ferrite filled PEEK composites, whereas, typical yield pattern of crust and trough on fractured topographic surfaces of ferrite and nano-barium titanate, reinforced PEEK composites, was visible.

Keywords: Fracture, poly-ether-ether-ketone (PEEK), scanning electron microscopy (SEM), strength

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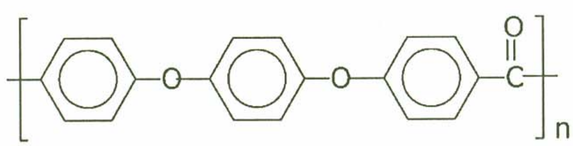
INTRODUCTION

Poly-ether-ether-ketone (PEEK) is a high performance thermoplastic which is used as neat polymer, blends, and matrix material in various applications. M/s. Imperial Chemical Industries (ICI), UK in 1978, first produced high molecular weight PEEK. PEEK is a polymer which is semicrystalline and a highly aromatic thermoplastic belonging to the class of polymers known as poly(aryl-ether-ketone)s. Because of their relatively stiff aromatic polymer backbone, PEEK exhibits some of the highest thermal transitions found in commercially available polymers today. As a result, it can be continuously used at 260°C temperatures and for shorter duration it can be used up to 300°C[1,2]. This polymer possesses a range of good properties, including toughness, very low smoke emission in flammability tests, and resistance to all common solvents[3].

PEEK has a crystalline melting point of 335°C. It is made up of the following repeat unit[4].



Typically, the product has a melting temperature of 343°C, glass transition temperature (T_g) of 143°C[5], and stable in the melt at 400°C for 1 h. PEEK can be obtained in amorphous and semicrystalline form, depending on the processing conditions employed from the molten state[6]. The natural grade of PEEK, manufactured by M/s. ICI, UK, is 35% crystalline. However, the maximum crystallinity level of 40% can be achieved by cooling the melt at very low cooling rates (1°C/minute)[5].

Advanced composites based on the PEEK matrix have attracted the attention of polymer technologists due to their high temperature stability, adaptability to various processing techniques, good mechanical and chemical properties, etc. The most common processing techniques for PEEK composites are compression molding, injection molding, autoclaving, and filament winding. The PEEK matrix may be used in various forms, such as powder, granules, co-woven yarn, film, and fabric with most of the reinforcement.

The purpose of this study was to find out the effect of reinforcement of nano-barium titanate on ferrite filled PEEK composites in flexural mode, for high performance applications, and later these effects were corroborated through SEM of fractured topographic surfaces of these composites.

EXPERIMENTAL

Fine powder grade of PEEK (Grade: 450 PF) manufactured by M/s. ICI, UK was used. The particle size of the powder was 30–50 μm. Ferrite and

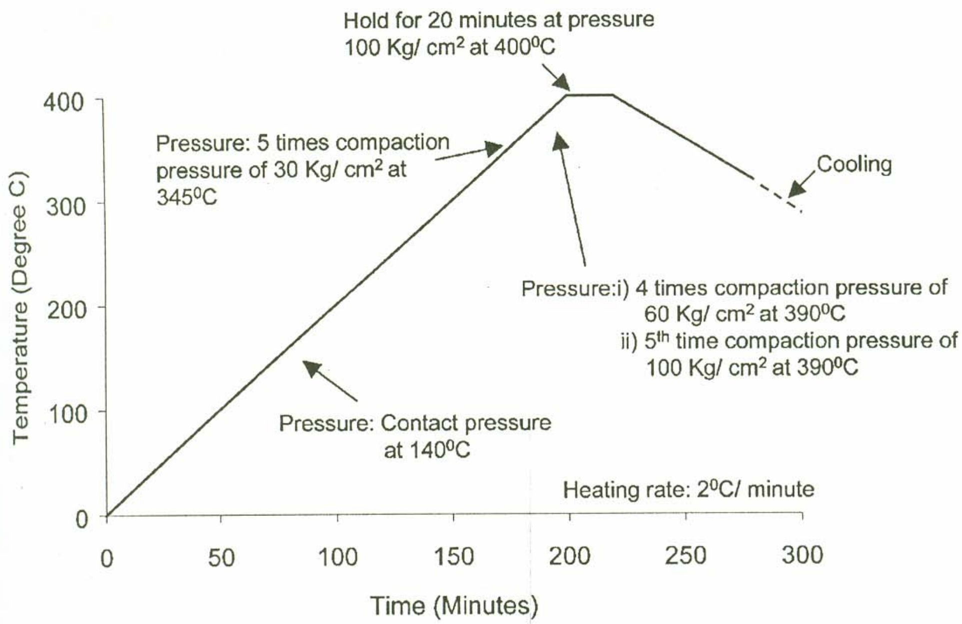


Figure 1. Molding cycle.

barium titanate, in nano particle form, were indigenous. The ferrite used in these experiments was cobalt-silicon (Co-Si) doped barium hexaferrite[7]. The ferrite powder had particle size of 10–15 μm, density of 4.5 g/cm^3, and melting point of 1400°C. The particle size of barium titanate was found to be ~25 nano meter (nm). In all of the composites, fast extrusion furnace (FEF) carbon black, as per ASTM D 1765 No. 550, was used. Carbon black provided moderate reinforcement and stability against oxidative thermal degradation at molding temperature range[8].

The molding of samples was carried out in a compression molding machine, using a matched die mold, at the maximum processing temperature of 400°C and pressure of 100 kg/cm^2, as per the molding cycle mentioned in Fig. 1.

Flexural strength and modulus were evaluated as per ASTM D 790 and scanning electron micrographs of fractured samples in flexural mode were taken at 200 magnification on a scanning electron microscope (SEM) (Model No. JEL-JSM-35F) manufactured by M/s. JEOL. Table 1 defines the designation of the samples and their composition for ferrite filled PEEK composites and ferrite and nano-barium titanate filled PEEK composites.

RESULTS AND DISCUSSION

The flexural strength was found to be decreasing with the increase in ferrite content in PFM and PFBM samples. The flexural strength of

Table 1. Designation of samples and their constituents

Sl. no.	Sample designation	Composition (%)			
		PEEK	Ferrite	Barium titanate	Carbon black
1	a PFM2	97	2	0	1
	b PFBM2	96	2	1	1
2	a PFM4	95	4	0	1
	b PFBM4	94	4	1	1
3	a PFM6	93	6	0	1
	b PFBM6	92	6	1	1
4	a PFM8	91	8	0	1
	b PFBM8	90	8	1	1
5	a PFM10	89	10	0	1
	b PFBM10	88	10	1	1

PFBM samples was observed comparatively higher than PFM samples, whereas, flexural modulus was found to be increasing in both PFM and PFBM samples with the increase in the content of ferrite, but the flexural modulus of PFBM samples was observed comparatively lower than PFM samples (Table 2 and Fig. 2a to 2e). The lowering of flexural modulus of PFBM samples in comparison to PFM samples drew special attention because the flexural strength of PFBM samples was comparatively higher than PFM samples for the same ferrite content.

The scanning electron micrographs of PFM and PFBM samples are given in Figs. 3a to 3e and 5a to 5e, respectively. Coarse failure pattern, with typical yield pattern of crust and trough, was observed on the examination of scanning electron micrographs of fractured topographic

Table 2. Comparison of flexural strength and modulus of PFM and PFBM samples

Sl. no.	Sample designation	Flexural strength (MPa)	Flexural modulus (GPa)
1	a PFM2	151	5.35
	b PFBM2	164	4.96
2	a PFM4	141	5.39
	b PFBM4	154	4.98
3	a PFM6	131	5.49
	b PFBM6	144	5.06
4	a PFM8	118	5.52
	b PFBM8	139	5.12
5	a PFM10	112	5.57
	b PFBM10	134	5.15

surfaces of PFM2 (Fig. 3a) and PFM4 samples (Fig. 3b). This was attrib-
uted to the presence of abundant ductile PEEK matrix which had failed after some elongation. In scanning electron micrographs of PFM 6, 8, and 10 samples (Figs. 3c, 3d, and 3e), smooth failure pattern without

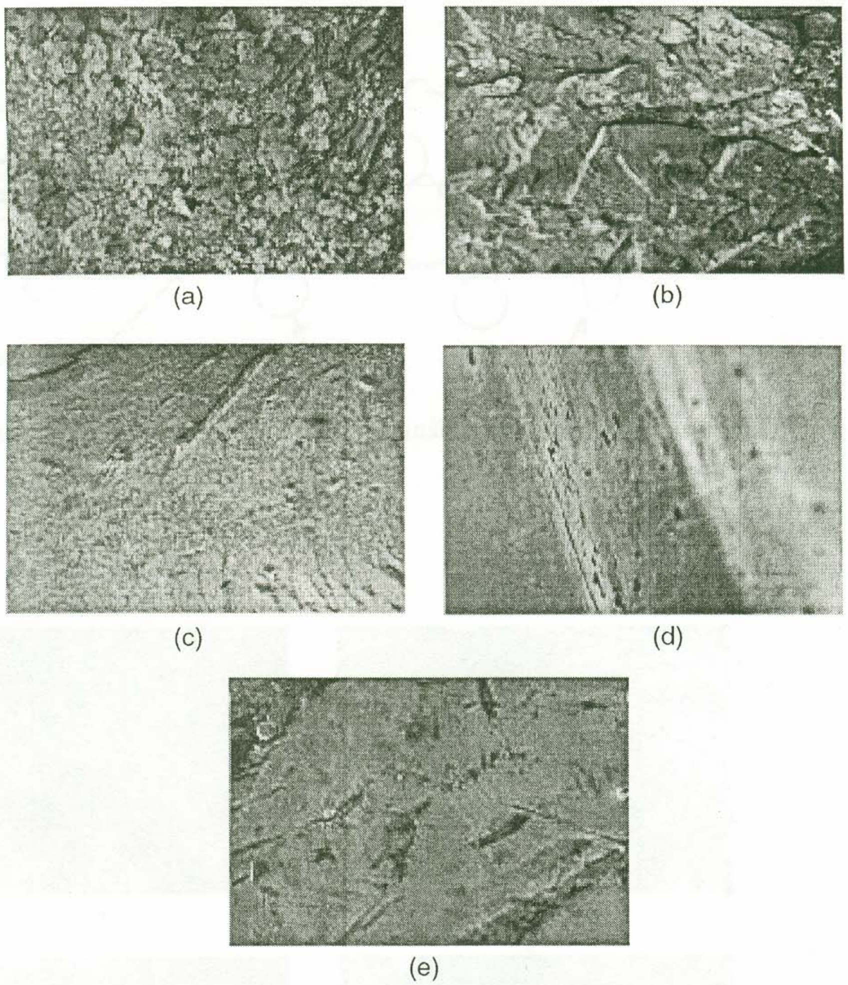


Figure 3. Scanning electron micrographs of PFM samples: a) PFM2; b) PFM4; c) PFM6; d) PFM8; and e) PFM10.

In PFBM samples, the reinforcing effect of nano-barium titanate reduced the discontinuity among the polymeric chain of PEEK matrix, which caused increase in flexural strength and strain. This increase in strain led to the reduction of flexural modulus of PFBM samples over PFM samples. This increase in flexural strength and reduction in flexural modulus could be related through the approach of micromechanics of short fiber reinforced thermoplastic composites. In the case of PFBM samples, nano-barium titanate could be considered as short fiber reinforcement. According to this approach, when a PFBM sample is under flexural load (in three point bend test) then the portion above the neutral axis is in compression and the portion below the neutral axis

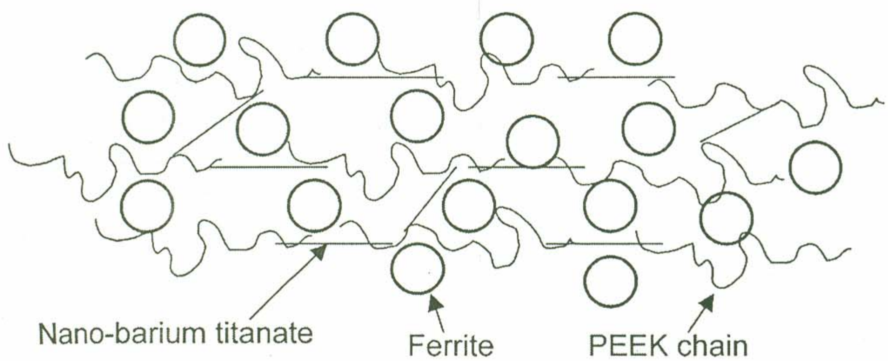
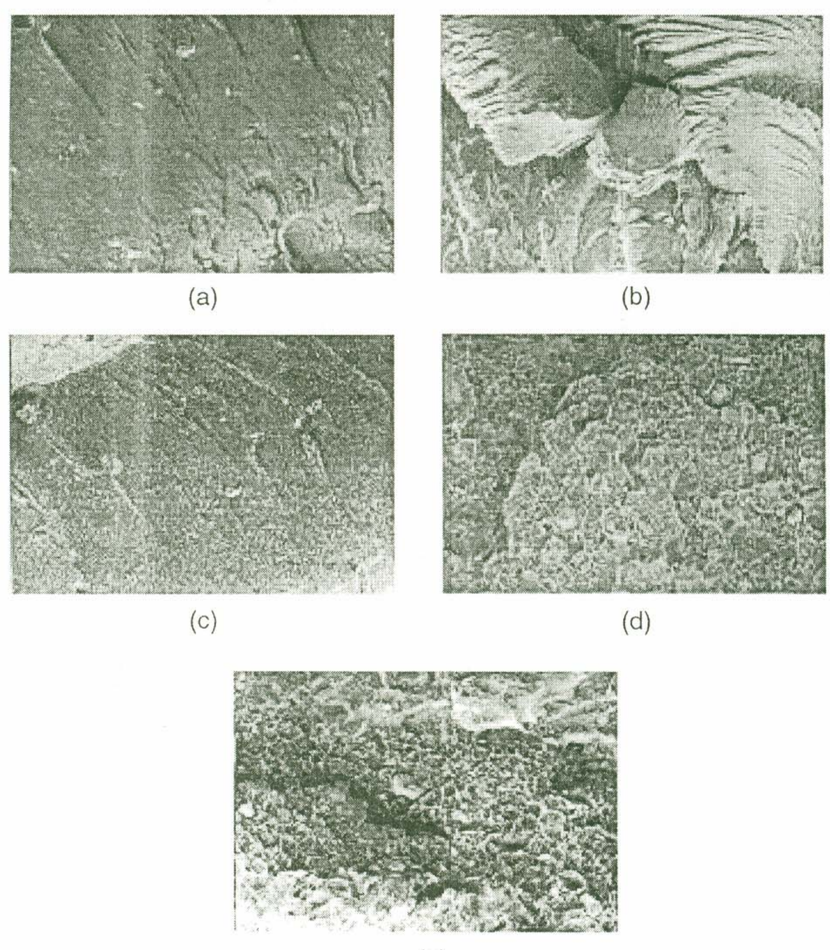


Figure 4. Reinforcing nano-barium titanate causing continuity among PEEK chains.



strength. Similarly, the discontinuity caused by ferrite among the polymeric chains of PEEK was overcome by the reinforcing effect of nano-barium titanate, as shown in Fig. 4. This caused uniform deformation of PEEK matrix throughout the sample, which increased the strain and, in turn, led to the reduction in flexural modulus.

The increase in strain could be interrelated with the elongation pattern seen in scanning electron micrographs of PFBM 6, 8, and 10 samples in addition to PFBM 2 and 4 samples (Fig. 5a to 5e). Whereas, the elongation pattern seen in scanning electron micrographs of PFM 6, 8, and 10 samples (Fig. 5c, 5d, and 5e, respectively) was negligible.

The failure pattern of PFBM 2 and 4 samples (Fig. 5a and 5b, respectively) was found to be coarser than PFM 2 and 4 samples due to the reinforcing effect of nano-barium titanate, apart from presence of abundance of matrix.

CONCLUSIONS

This study corroborates that scanning electron micrographs are closely interrelated to flexural stress-strain related properties of ferrite and nano-barium titanate reinforced composites. The effect of ferrite content was visible on morphology and flexural properties. There was an increase in flexural modulus and decrease in flexural strength with the increase in ferrite content in the composites. With the reinforcement of nano-barium titanate in the composites, the flexural strength increased and flexural modulus decreased comparatively. The same was corroborated by the studies of scanning electron micrographs of fractured topographic surfaces of these samples. Brittleness increased with the increase in ferrite content and elongation increased with the reinforcement of nano-barium titanate which had reinforcing effect.

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