SYNTHESIS AND CHARACTERIZATION OF ALUMINA NANOFIBER FILLED POLY ETHER ETHER KETONE (PEEK)

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ABSTRACT

This studies aims to understand the effects of alumina nanofiber filled Poly ether ether ketone (PEEK) on the nanocomposite physical-chemical, thermal and crystallization behaviour. Alumina nanofiber has been prepared by sol gel method. The addition of alumina nanofiber into PEEK was carried out via in-situ polymerization to obtain homogenous materials. The nanocomposites were prepared by dispersing the fibre in solvent and then undertaking the in-situ synthesis of PEEK. Composite materials with 1 wt %, 2.5 wt % and 5 wt % of alumina nanofiber have been successfully prepared by dispersing the alumina nanofiber in sulfolane as solvent and upon sonication for 30 minutes. Due to the small size of the nanofillers, high magnifications are required to observe the dispersion of the alumina nanofibers in PEEK matrix. Transmission electron micrograph (TEM) and field emission scanning electron micrographs (FESEM) indicated excellent dispersion and interactions between PEEK matrix with the adding of alumina nanofibers. Differential scanning calorimeter studies show changes in melting and crystallization point and degree of crystallinity with different loading of alumina nanofiber. The result also indicated that alumina nanofiber serves as nucleating agents in PEEK composites. The X-Ray diffractogram (XRD) data indicated that the crystallinity of the PEEK composites was highest with 1 wt % alumina nanofiber. The thermogravimetry analysis (TGA), shows that the thermal stability for 1 wt % alumina nanofiber-PEEK composites was enhanced compared to that of pure PEEK.

ABSTRAK

Kajian ini bertujuan untuk memahami kesan nanofiber alumina poli eter eter keton (PEEK) ke atas sifat kimia-fizikal, termal dan penghabluran. Alumina nanogentian telah disediakan dengan kaedah sol gel. Penambahan alumina nanogentian ke PEEK telah dijalankan melalui pempolimeran in-situ untuk menghasilkankan bahan yang homogen. Nanokomposit telah disediakan dengan menyebarkan alumina nanogentian ke dalam pelarut dan kemudian menjalankan sintesis in-situ PEEK. Bahan komposit dengan 1 % berat, 2.5 % berat dan 5 % berat alumina nanogentian telah berjaya disediakan dengan menyebarkan alumina nanogentian di dalam sulfolan sebagai pelarut dan sonikasi selama 30 minit. Disebabkan oleh kerana saiz gentian yang sangat kecil, pengimbas berkuasa tinggi diperlukan untuk mengkaji penyebarab alumina nanogentian di dalam matriks PEEK. Kajian 'Pengimbas Transmisi Elektron' (TEM) dan 'Pengimbas Medan Emisi Mikrograf Electron' (FESEM) menunjukkan penyebaran yang sangat baik dan interaksi antara matriks PEEK dengan alumina nanogentian. 'Kalorimeter Imbasan Pembezaan' (DSC) menunjukkan perubahan dalam takat lebur dan penghabluran dan darjah penghabluran dengan kandungan alumina nanogentian di dalam PEEK. Keputusan juga menunjukkan bahawa alumina nanogentian berfungsi sebagai ejen penukleusan dalam komposit PEEK. Difraksi sinar-x (XRD) menunjukkan bahawa penghabluran komposit PEEK adalah tertinggi bagi 1 % berat alumina nanogentian. 'Analisis Termogravimetri' (TGA), menunjukkan bahawa kestabilan terma bagi 1 % berat alumina nanogentian-PEEK komposit telah dipertingkatkan.

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LIST OF SYMBOLS

d	Spacing between the planes in the atomic lattice
D	Crystallite size
f	Bond strength
$m1$	atom 1
$m2$	atom 2
n	Integer
T_c	Crystallization Temperature
Tg	Glass Transition Temperature
T_m	Melting Temperture
υ	Vibrational Frequency
W	Weight fraction
X_c	Degree of Crystallinity
B	Full width at half maximum
λ	Wavelength
θ	Diffraction angle angle between the incident ray and the scattering planes
σ	Standard Deviation
ΔHc	Heat of Crystallization
ΔHc°	Heat of Fusion for pure crystalline of PEEK

LIST OF ABBREVIATIONS

CTE	Coefficient of Thermal Expansion
DSC	Differential Scanning Calorimeter
EDX	Energy dispersive X-ray fluorescence
EPD	Electrodeposition
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transform Infrared Spectroscopy
MW	Microwave
MWNT	Multi-walled Carbon Nanotube
PEEK	Poly Ether Ether Ketone
PNC	Polymer Nanocomposite
TEM	Transmission Electron Miroscopy
TGA	Thermogravimetry Analysis
SWNT	Single–Walled Carbon Nanotubes
XRD	X-Ray Diffractogram
ZIF-8	Zeolitic imidazolate frameworks

CHAPTER 1

INTRODUCTION

1.1 RESEARCH BACKGROUND

In recent years, the field of polymer composite materials had the attention of scientist and researchers from all over the world. This is because of the achievement in improving the polymer composite materials properties. In addition, polymer composite materials now have the potential to replace metals, ceramics or other engineering materials with better performance and it has become the materials of choice for many engineering applications.

A lot of researches on incorporating various fillers into polymer matrix have been done. Incorporation of various types of fillers in the polymer can improve physical properties of the polymers. The factors that can affect the properties of the polymer composites (Gay et al., 2003) are:

- Type of fillers used
- Synthesis method
- Polymer stability

There are various types of fillers available such as clays, feldspar, silica, glass, mica and others. Most of them are less expensive than the polymer itself and could reduce the production cost of polymeric materials. Fillers are added into polymer to increase the modulus of stiffness, reduce shrinkage, improve crack resistance and control viscosity (Pal, 2007). In general sense, the addition of fillers is to improve the physical properties of the polymer without affecting its chemical bonding.

Polymer composite materials can be classified into two broad groups which are fiber-reinforced polymer composite and particle-reinforced polymer composite. Fiber reinforced composites consist of fibers of high aspect ratio as the dispersed phase. The fibers bear the major portion of the applied force and the function of the matrix is to distribute and transmit load to the fibers. Fiber reinforced composite can be divided into two types. They are continuous or long fiber composites and discontinuous or short fiber composites. Idicula et al. (2006) have shown that the fiber length distribution and the orientation of the fibers play an important role in determining the strength of the composites. The reinforcement of fiber fillers into polymer matrix was developed in order to achieve a combination of properties which are not available in unmodified polymer. For example, when nanofibers were mixed with polymer matrix, they become strong and light. They are often stronger than steel, but weight much less.

Polymer composites with nanoparticles have become a new high performance materials (Godovski et al., 1995). The nanocomposites have been applied in the commercial and industrial field (Safadi et al., 2003). They are used widely especially in aircraft, space, automotive, sport, plastic and electronic industries.

1.2 POLYMER BASED ENGINEERING MATERIALS

Polymer can be categorized into two classes being thermoplastic and thermosetting. Thermoplastic materials have very long chain like molecules and are held together by relatively weak Van der Waals forces. The most common thermoplastic are polyethylene sulfone (PES), polyether ether ketone (PEEK), polyether imide (PEI) and polyimide (PI). When the material is heated the intermolecular forces are weakened so that it becomes soft and flexible. Thermoplastic can simply be melted at high temperature and formed.

In contrast to the thermoplastic polymer, thermosets cannot be melted after crosslinking process but suitable solvents or hardener can be applied to facilitate processing. They also cannot be shaped thermally, mechanically or chemically into compounds or different structure from the starting materials. Epoxy resin, amino resin, Polyurethanes, Silicone, Melamine and Vulcanized rubber are examples of thermosets polymer. The crosslinking process is illustrated schematically in figure 1.1. The molecular structure is irreversibly modified by the crosslinking process. Figure 1.1 illustrated the resin initially not crosslinked, then it started crosslinking and finally nearly fully crosslinked. The materials will change to solid again when cooled. This cycle of softening by heat and solidifying on cooling can be repeated more or less indefinitely and is a major advantage in most of the processing method for the materials.

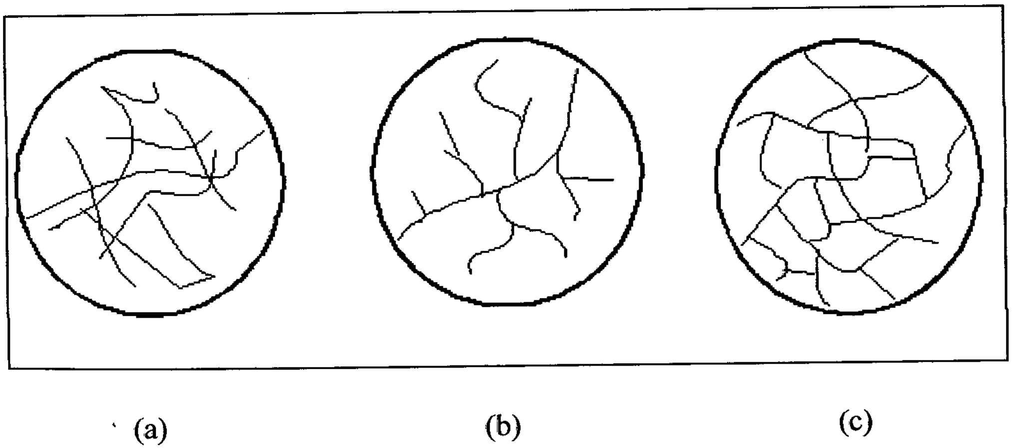


Figure 1.1: Crosslinking process of polymer. a) Chain, b) Branch and c) Network

Source: Mark, J. E et al., 2005

Nowadays, the rate of growth of demand for thermoplastic engineering materials is considerably higher than that of the thermosetting composites. This is because of the broadening use of composite parts and the need for higher production rates of engineering plastics. A number of the materials offer service temperature in excess of 200°C and reinforcement by nanofillers enable them to be used above 300°C.

Table 1.1: Properties of common thermoplastics and thermosetting polymer

	Thermoplastic			Thermosetting		
Properties	PES	PEEK	PEI	Epoxy	Phenolic	Polyester
Tensile Strength (MPa)	84	70	105	50-110	1.6-4.1	35-95
Modulus of Elasticity (Gpa)	2.4	3.8	3	2.5-5.0	2.7-4.1	1.6-4.1
Density (g/cm^3)	1.37	1.31	1.27	1.2-1.4	1.2-1.4	1.1-1.4

Source: Deborah, D. L. C, 2002.

The properties of common thermoplastics and some thermosetting materials are listed in table 1.1. In this table 1.1, PEI strength is the highest compared to the other two thermoplastics while for thermosetting plastic epoxy has the highest strength. PEEK and phenolic shows the highest modulus of elasticity. However, among the thermoplastic, PEEK is the most brittle. While for thermosetting plastic, epoxy shows higher strength and wider range of modulus of elasticity compared to the phenolic and polyester among thermosetting. Overall epoxies are more brittle than PES, PEEK and PEI.

1.3 POLY ETHER ETHER KETONE (PEEK)

In the past, the applications of thermoplastic as a matrix were not popular because of its low resistant to chemical reaction and perhaps, the problems in controlling the strength and stiffness at elevated temperature. However, this changed when high performance PEEK polymer was successfully prepared by Bonner in 1962.

PEEK is one of the polymer families of aromatic compounds with the molecular backbone being ketone and ether functional groups between aryl rings as illustrated in figure 1.2. PEEK is a semicrystalline thermoplastic with outstanding performances. It posses excellent mechanical properties such as strength modulus, toughness, resistance to creep, abrasion and fatigue. It also has, high temperature resistant, high continuous service temperature, good resistance to aggressive solvent and is completely soluble in concentrated sulphuric acid at room temperature (Blundell et al., 1983 and Atkinson et al., 2002). These make PEEK an attractive matrix material in producing nanocomposites for engineering purpose for example in aircraft system. The typical properties of PEEK are given in table 1.3. PEEK posses high decomposition and processing temperature and this make PEEK an excellent thermal and chemical resistance material.

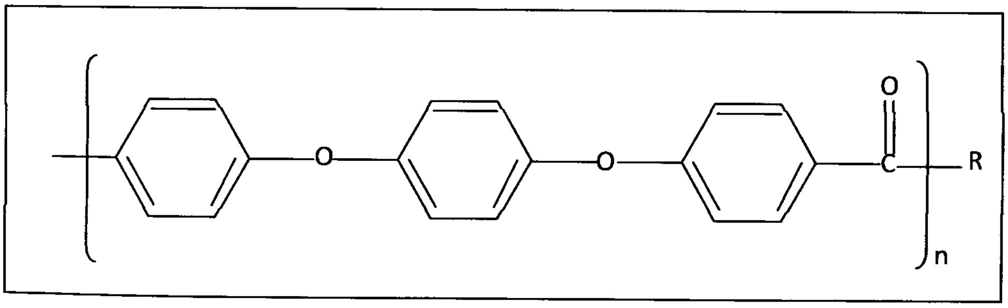


Figure 1.2: Molecular structures of PEEK

Source: Yin et al., 2008

Table 1.3: Typical properties of PEEK

Decomposition Temperature	590 °C
Processing Temperature	380 °C
Melting Point	340 °C
Glass Transition Temperature	143 °C

Source: Deborah, D.L.C, 2002.

1.4 CERAMIC FILLERS

Ceramic particles are fine and inert materials with exceptional hardness and thermal stability. Ceramic fillers have been used in reinforcing polymer to enhance the polymer's properties. The adhesion of the ceramic filler to the surrounding polymer is very limited (Lobland et al., 2010). This is due to the weak interaction between ceramic oxides which is hydrophilic with the hydrophobic polymers.

Because of its strength and hardness, ceramic materials are commonly used as polymer fillers. Alumina is one of the materials in engineering ceramics family and it has been widely used for many applications. Alumina is a common ceramic oxide employed in nanocomposites. The chemical bonding between Al and O ions enable the materials to have outstanding physical stability at high temperature, good chemical inertness and high mechanical properties.

Alumina also shows great potential in relation to electrical properties. Alumina has been widely used as an electrical insulator and semiconductors because the dielectric loss of alumina was low (Goswami et al., 2002). On the other hand, alumina is highly brittle because of its flexural strength and toughness and this has limits its potential applications.

In recent years, extensive efforts have been undertaken on synthesizing nanostructure alumina to improve their properties. Teng et al., (2007) have successfully prepared high density and high flexural strength alumina nanoparticles at lower temperature.

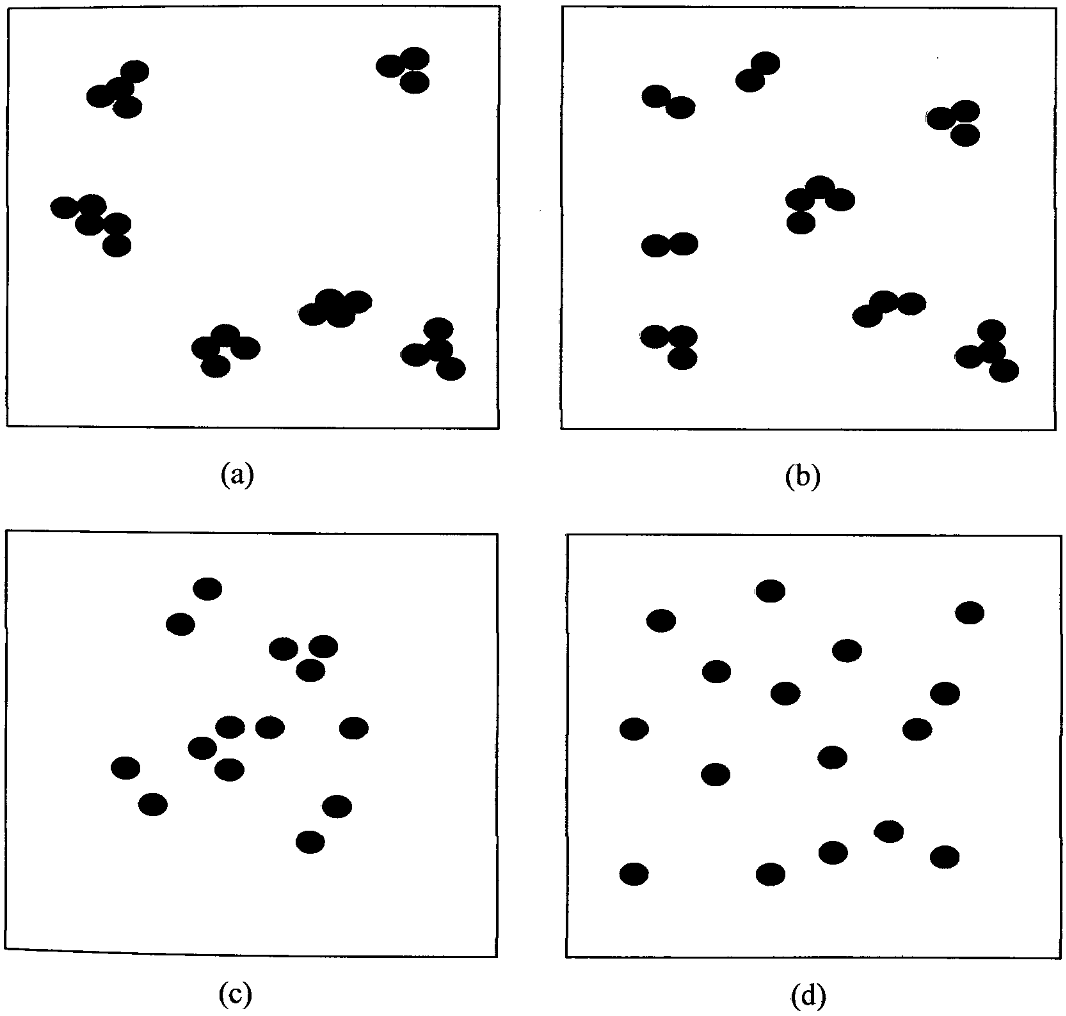
Presents works have successfully explored a new approach to processing of ceramic oxide by using sol gel method (Hertz et al., 2010 and Teoh et al., 2007). Sol gel method provides an attractive and more convenient route compared to other method like polymer impregnation and pyrolisis, melt infiltration, chemical vapour infiltration and hot processing (Naskar et al., 2004). Sol gel method can control the structure of the ceramic materials and produced with high purity materials with uniform pore sizes. (Chen et al., 2001, Ersoy et al., 2004 and Huang et al., 1997).

1.5 POLYMER COMPOSITE MATERIALS

Polymer materials are combined with fillers to make them stronger, lighter, electrically conductive and cheaper. There have been numerous studies on the effect of fillers types on the properties of polymer. Sandler et al. (2002) in their works on reinforcing poly ether ether ketone with carbon nanofiber found that the tensile strength of the polymer increased linearly with the weight loading of carbon nanofiber. They also proved that the nanofiber particle size can affect the polymer matrix crystallization behaviour. Cho et al. (2006) have investigated the effect of inclusion size particles on the polymer composites mechanical properties. It was found that with small amount of fillers loading, the mechanical properties of the polymer was improved by adding smaller particles size fillers.

In Guoliang et al. (2008) studies indicate that higher tensile, flexural and impact performance were achieved by reinforcing PEEK with the smaller size and lower weight loading of alumina particles. In addition, the impact strength has been improved eight times than that of neat PEEK.

Nanometer filler have large surface area to volume ratio compared to micrometer size filler. They tend to form a large agglomeration or clumping as Van der Waals interaction between the particles becomes stronger. In order to improve the dispersion of the nanofillers, proper chemical treatment and processing method should be utilized and developed in order to improve the properties of the polymer composites materials. The effect of fillers depends on the ability to distribute and disperse them throughout the polymer matrix as seen in figure 1.3. In figure 1.3 (a), the particles stick with each other and are distributed randomly while their agglomerated particles are distributed through the matrix in figure 1.3 (b). Figure 1.3 (c) illustrated particles that are not well distributed and is likely to agglomerate and in figure 1.3 (d) the particles are seen dispersed and distributed uniformly.



(a) (b)

(c) (d)

Figure 1.3: Schematic of degree of dispersion and distribution for particles in the polymer matrix. a) Poor dispersion, poor distribution, b) Poor dispersion, good distribution, c) Good dispersion, poor distribution and d)Good dispersion , good distribution.

Source: Hedayati et al., 2011

1.6 PROBLEM STATEMENT

Most inorganic nanofillers are incompatible with polymer matrix and this may lead to poor dispersion and distribution. While, increasing dispersion of nanofibers may improve the polymer properties, fillers with nanometer size are strongly affected by Van der Waals forces because of their small size and large surface area. The formation of aggregates or agglomerates of the nanofillers make the properties of the polymers become poor.

Another challenge in the processing of polymer nanocomposites is the increase of resin viscosity with the presence of nanofillers .This may cause many difficulties during composite processing. For instance, high-viscosity resins lead to long mold filling time and poor wettability. High viscosity resin also could easily trap air bubbles making air removal difficult, resulting in structure defect in composite materials and hence their poor performances.

1.7 OBJECTIVES:

The objectives of this study are to synthesize and characterize alumina nanofiber reinforced poly ether ether ketone (PEEK) in order to improve their mechanical properties and thermal behaviour. The main goal of this study is to improve the physical properties of poly ether ether ketone reinforced with alumina nanofiber. The research includes developing method in dispersing alumina nanofiber within the poly ether ether ketone and characterizing the polymer nanocomposites. Characterizing the thermal and mechanical properties of the polymer nanocomposites enable better understanding of their performance. The specific objectives are:

1) To investigate the suitable medium (solvent) for dispersing nanofillers.
2) To establish the optimum sonication time to disperse the fillers in polymer matrix.
3) To study the effect of alumina nanofibers on the properties of PEEK composites.
4) To characterize and analyze the synthesized pure PEEK and alumina nanofiber-PEEK composites.

1.8 SCOPE AND LIMITATIONS OF RESEARCH

This study focused on improving the properties of the polymer nanocomposites. The primary outcome will be the optimum method for effective dispersion of alumina nanofiber within the PEEK matrix. However, the mechanical testing of the sample could not be done because the transformation of alumina nanofiber-PEEK composites powder into homogenous solid sample required higher temperature than that could be achieved using available equipment such as:

1) Hot Press
2) Compression Molding
3) Melt Flow

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

This chapter reviews past research efforts on the processing of polymer nanocomposites especially involving ceramic fillers. The method to disperse the fillers within the polymer matrix and the enhancement of polymer properties will be emphasized.

2.2 POLYMER - CERAMIC COMPOSITES

Ceramics are widely used in the last 20 years. High performance ceramics were developed for engineering applications such as for making highly efficient gas turbines, aerospace materials, automobiles and others. Their strong ionic or covalent bonding provides the advantages over metal based materials. However, ceramics shows relatively low fracture toughness and strength, degradation of mechanical properties at high temperature and poor resistance to creep, fatigue and thermal shock.

In order to overcome the weaknesses, many researchers have investigated the properties of nanophase ceramic composites. This is done by incorporating nanometers size fillers into ceramic matrix. Niihara (1991) reported that such dispersion can be divided into three types which are intragranular, intergranular and intra/intergranular types as illustrated in figure 2.1.