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Polyetheretherketone-Metal Composites for Biomedical Applications

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Abstract

Polyetheretherketone-Metal Composites for Biomedical Applications

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Polyetheretherketone (PEEK) implants are widely accepted and adopted in the orthopaedic field. However, PEEK is relatively bioinert and results in limited fixation with bone. In order to improve the initial cell responses and the fixation of the PEEK implants, PEEK-metal composites were investigated using titanium (Ti) and magnesium (Mg). PEEK-Ti and PEEK-Mg composites with up to 60 vol% metal contents were successfully prepared by compression molding the mixed metal and PEEK powders and were analysed for potential use in the medical field.

Ti has excellent biocompatibility and mechanical properties. With the incorporation of Ti in PEEK, the composite materials showed that *in vitro* cell

attachment, proliferation and differentiation were enhanced and the mechanical properties of the composites can be tailored to mimic the bone more closely. With the incorporation of 60 vol% of Ti to PEEK, the compressive strength increased from 132 MPa to 247 MPa and the elastic modulus increased from 3.7 GPa to 7.1 GPa. The significant improvements in biological and mechanical properties suggest that PEEK-Ti composites can be tailored to be used as potential load-bearing implant material.

With biodegradable Mg used as fillers, PEEK- Mg composites were fabricated to enhance the biocompatibility and to provide stronger interlocking of the implant once the Mg is degraded in physiological environment. Hydroxyapatite, a bioactive substance, was coated on the exposed Mg surfaces to enhance the early stage cellular activities and to reduce the corrosion rate of Mg. The post-corrosion morphologies, mechanical properties and preliminary cellular tests were performed to evaluate the potential of PEEK-Mg as a novel implant material. The compressive strength composites were not significantly different from the pure PEEK samples. However, the elastic modulus of 60 vol% PEEK-Mg increased to 5.4 GPa from 3.1 GPa of the pure PEEK. These results show that hydroxyapatite layer on the exposed Mg surfaces can enhance the cellular properties on the surface of the implants, and the pores formed by the degradation of Mg particles can be used to improve the bone-to-PEEK

fixation through the mechanical interlock of the implant with the adjacent bones.

This study investigated PEEK-Ti and PEEK-Mg as potential orthopaedic implant materials. By incorporating biocompatible metal particles into PEEK, the mechanical and the biological properties were enhanced. These results show that PEEK-metal composites are promising materials for use as biomaterials.

Keywords: Polyetheretherketone, polymer composite, PEEK-metal, biocompatibility, mechanical property, orthopedic implants

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Chapter 1

Introduction

1. Introduction

With life expectancies increasing with the development of technology, there is a significant increase in society's demand for orthopedic implants and novel biomaterials. In the 18-84 age group, back or spine impairment is the primary cause of activity limitation [1, 2]. Spinal fusion (also known as arthrodesis) is one of the treatment options. FDA approved the usage of spinal fusion cages in 1996 and many materials were invested for the application. One of the most widely used materials is polyetheretherketone (PEEK). PEEK was first proposed as a biomaterial in the 1980s and received much attention for use in orthopedic applications [2-4]. PEEK is a semi-crystalline thermoplastic that has good mechanical properties, ease of processing, biocompatibility, lack of toxicity and sterilization resistance which make it an attractive material for biomedical applications. Furthermore, the stable structure of PEEK as shown in Figure 1 makes it extremely inert and resistant to chemical and thermal degradation [3].

More recently, PEEK is being considered as an alternative to metallic implants [3]. Unlike metallic biomaterials, PEEK has relatively low elastic modulus which is similar to that of the cortical bone. The elastic modulus of PEEK is approximately 3-4 GPa and that of the cortical bone is approximately 18 GPa [3]. Metal implants have many advantages but often suffers from stress shielding effect which results from the mismatch between the elastic modulus of the implant material and the bone [5-7]. The high elastic modulus of the implant interferes with the stress imposed on the bone, impairs the

proper healing of the bone and causes bone resorption. Table 1 shows the mechanical properties of bone and commonly used biomaterials. For producing biomaterials that mimic the bone more closely, PEEK is a good alternative due to its many advantages mentioned above [8].

PEEK has received much attention in the load-bearing orthopedic applications. However, one disadvantage of PEEK is that it is relatively inert in biological environment and shows slow adhesion of bone tissue to the implants [3, 4, 9]. Thus a considerable amount of research has been done to improve the biological properties and to tailor the elastic modulus of PEEK. In this study PEEK-Metal composites were investigated for potential orthopedic implant materials. In particular, titanium (Ti) and magnesium (Mg) particles were used as filler materials.

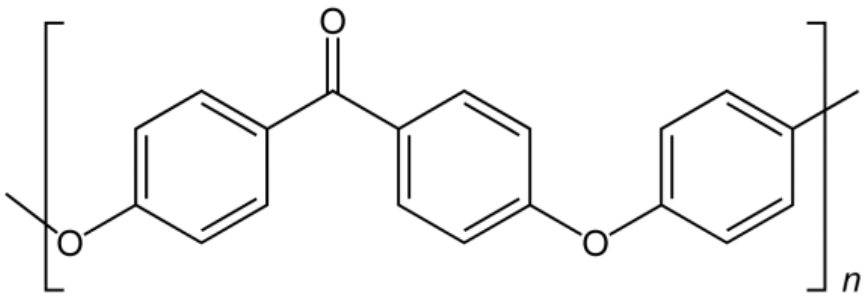

Figure 1. Chemical structure of polyetheretherketone (PEEK).

Chapter 2

Theoretical Review

2. Theoretical review

Studies have incorporated bioactive substances such as hydroxyapatite to enhance the biological properties of PEEK by providing chemical and biological bonding at the surface [10-12]. Other studies have focused on providing strong interfacial bonding between the material and the bone and one of the methods is to produce porous PEEK to allow bone ingrowth and mechanical interlocking of the implant. Below is a brief summary of the techniques that were utilized to improve PEEK as an implant material.

2.1 Surface treatments

Direct surface modifications that have been utilized in the past can be divided into physical and chemical techniques and they involve physical modifications and chemical reactions on the surface, respectively. Some physical methods include flame, laser, corona, electron beam, ion beams, sputter coating, hot plasma, cold plasma and UV/Ozone techniques. These require high energy that is required to modify the inert polymer surfaces which generate atom clusters that are coated on the surface. On the other hand, chemical modifications involve direct reactions or by grafting (covalently bonding molecules to the surfaces). Some examples of chemical modifications are surface etching and surface grafting with amine groups or other biomolecules [13]. A limitation with the surface modification is that the enhanced properties are limited to the surface of PEEK and therefore do not change the properties of the bulk material.

2.2 Porous PEEK

Pores in biomaterials allow tissues to growth inward and integrate with the implant. It enhances fixation of the implant with the tissues and porous bodies more closely mimic the architecture of the host tissues [14, 15]. Porous PEEK have been fabricated through various methods like particulate leaching, heat sintering, selective laser sintering, microwave sintering, micromachining and textiles. Although pores allow the cells to integrate firmly with the material, by increasing the porosity of the material the requirements for the mechanical property are harder to meet [13].

2.3 PEEK composites

Addition of various bioactive substances such as calcium phosphates or bioglass to PEEK can create materials with customizable biological and mechanical properties [16]. Some common calcium phosphates include hydroxyapatite (HA), β-tricalcium phosphate (β-TCP) and bioglass [17, 18]. Various forms of these calcium phosphates have been utilized including whiskers [12, 15, 19], spheres [20, 21], fibers and powders [4, 17, 22]. Bioactive reinforcements have been incorporated into PEEK matrix and processed through injection molding, compounding, cold pressing, pressureless sintering, compression molding and selective laser sintering. The advantages and disadvantages of the manufacturing processes are outlined in Table 2. Some other studies have focused on improving the mechanical properties of PEEK through incorporation of hydroxyapatite whiskers [12, 15, 19] and carbon fibers [23] as well.

2.3.1 PEEK-Metal Composites

In this study, biocompatible metals were used as fillers in PEEK composites. The study is unique in that biocompatible metal particles, and not ceramics, were incorporated into PEEK to improve the biological and mechanical properties of PEEK. Unlike the ceramic fillers, metallic fillers have high strength and elastic modulus that can act as reinforcements to provide strength and flexibility to tailor the mechanical properties. The two metals that were chosen are Ti and Mg.

Ti is a biomaterial that has been used in the medical field for decades due to its excellent biocompatibility, mechanical properties, corrosion resistance, fatigue and wear resistance and high ductility [24, 25]. In addition, Ti possesses a more bio-friendly surface when compared to PEEK [9]. Its ability to promote osseointegration [26, 27] is most likely dependent on the inherent oxide passivation layer as well as topography and surface energy [27-30]. Therefore, by incorporating Ti particles into PEEK, it is expected that the biological and the mechanical properties can be improved to make PEEK a more ideal implant material.

Mg on the other hand, has more recently been considered as a potential biomaterial due to its biodegradability and good biocompatibility [31, 32]. Mg ions are present in the human body therefore is not detrimental to the body. In addition, previously reported studies state that Mg could play a role in osteoblast regeneration [33]. Since Mg is a biodegradable material, it is important to mention the corrosion properties. The corrosion reactions of Mg

are shown below:

$$Mg_{(s)} + 2\ H_2O \rightarrow Mg(OH)_{2(s)} + H_{2(g)}$$

$$Mg_{(s)} + 2Cl^-_{(aq)} \rightarrow MgCl_2$$

$$Mg(OH)_{2(s)} + 2Cl^- \rightarrow MgCl_2$$

Rapid corrosion in aqueous solution will generate a large amount of hydrogen gas and also increase the local pH level significantly causing unfavorable effects in the body [31, 34]. Therefore, a coating on top of the Mg layer to delay and control the degradation rate of Mg is an important step [31, 32, 34-38]. In this study, a hydrothermal treatment reported by Hiromoto et al. was utilized to coat the exposed Mg with HA. When treated in the solution, Mg specimens acquire rod-like crystals that grow with treatment time. A schematic illustration of the HA formation on Mg surface is illustrated in Figure 2. When Mg is immersed in the solution, the following reaction occurs:

$$Mg \rightarrow Mg^{2+} + 2e^-$$

$$2H_2O + 2e^- \rightarrow 2OH^- + H_2.$$

With the release of hydroxide ions, pH increase initiates the HA formation and simultaneously forms $Mg(OH)_2$ on the surface. The continuation of the HA nucleation occurs more frequently than the $Mg(OH)_2$ formation due to lower solubility of HA than that of $Mg(OH)_2$ and the continuous supply of Ca^{2+} ions. As the dome-shaped HA layer covers the surface, the successive corrosion of the surface is slowed down. The crystal growth and the inner $Mg(OH)_2$ layer

growth continues with coating time. The role of the HA layer is not limited to controlling the corrosion rate of Mg. As mentioned earlier, HA is a bioactive substance that can influence cellular interaction with the material. Therefore, by coating the biodegradable Mg on the surface with HA, the three expected outcomes of the material are (1) excellent initial cellular responses due to the bioactivity of HA, (2) controlled corrosion rate of Mg and (3) bone ingrowth into pores once Mg is degraded in the body.

The study show preliminary investigations of PEEK-metal composites for medical use. The PEEK-metal composites in this study are limited to PEEK-Ti and PEEK-Mg and their preliminary biological and mechanical properties for potential use in the orthopedic field.

Table 1. Physical and mechanical properties of bone and various implant materials (obtained from [39]).

Properties	Natural bone	Magnesium	Ti alloy	Co–Cr alloy	Stainless steel	Synthetic hydroxyapatite
Density (g/cm^3)	1.8–2.1	1.74–2.0	4.4–4.5	8.3–9.2	7.9–8.1	3.1
Elastic modulus (GPa)	3–20	41–45	110–117	230	189–205	73–117
Compressive yield strength (MPa)	130–180	65–100	758–1117	450–1000	170–310	600
Fracture toughness ($MPa\text{-}m^{1/2}$)	3–6	15–40	55–115	N/A	50–200	0.7

Table 2. Advantages and disadvantages of the processing techniques to produce PEEK composites [13].

	Advantages	Disadvantages
Compounding & injection molding	- Low cost - High- volume commercial manufacturing of net shapes with a dense microstructure	- Increased melt viscosity with the fillers limit reliable mixing and molding and cause equipment wear
Cold pressing & pressureless sintering	- Low cost - Any level of reinforcement possible - Microporosity results in good biological properties	- Absence of pressure during sintering results in microporosity resulting in poor mechanical properties
Compression molding	- Low cost - High- volume commercial manufacturing of net shapes with a dense microstructure - Any level of reinforcement possible - Dense body thus has good mechanical properties - Porogens can be easily added to create pores	- Production rates slightly lower - Machining required to produce non-geometric shapes
Selective laser sintering	- Low cost - Net shape manufacturing - Macroporous scaffolds with tailored architecture possible - Customized shapes can be fabricated directly	- Slow production, maximum porosity limited to 70-74 vol% - Reinforcement volume fraction limited to maximum of 22 vol%

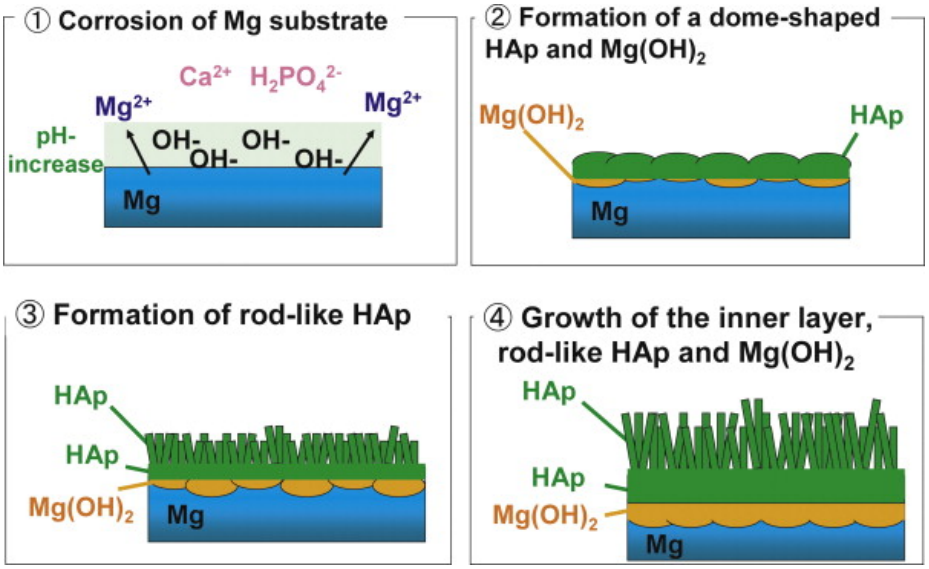

Figure 2. A schematic of the HA growth mechanism (Hap: Hydroxyapatite). Taken from [40]

Chapter 3

PEEK-Titanium for Potential Load-Bearing Implant Material

3. PEEK-Titanium for Potential Load-Bearing Implant Material

3.1 Introduction

In the past, metallic implants have been widely used in the orthopedic field. However, stress-shielding effect was an inevitable problem [3, 5-7]. Metals have a large elastic modulus which causes impaired load transmission at the site of implantation and interferes with the proper healing of the bone. In more recent years, polyetheretherketone (PEEK) has received much attention in the load-bearing orthopedic applications [3]. PEEK is a semi-crystalline thermoplastic that has good mechanical properties, chemical inertness, ease of processing, biocompatibility, lack of toxicity and sterilization resistance which make it an attractive material for biomedical applications[3, 23]. However, PEEK is biologically inert and shows slow adhesion of bone tissue to the implants. Thus a considerable amount of research has been done to improve the bio-inertness [3, 4, 21, 23]. Previous studies have incorporated various forms of hydroxyapatite, a bioactive ceramic, into the PEEK matrix to increase the bioactivity of the composite material [17, 20]. However, there is a limit to the amount of HA that can be incorporated into PEEK before a tradeoff in the mechanical properties occurs [11].

In this study, Ti powder was used as a filler material to reinforce PEEK. Ti is one of the oldest and commonly used implant materials that are used in the medical field. Its excellent mechanical properties, biological properties and its capacity to join with bone make it a reliable implantable

material [24, 41, 42]. By incorporating Ti powder into the PEEK matrix, it is expected that the advantages of using PEEK will be retained and that in addition the added Ti particles will make the composite more biocompatible and enhance the mechanical properties of the composite by acting as reinforcing particles. The objective of the study is to investigate the mechanical and biological properties of PEEK-Ti composites for potential use in biomedical applications. PEEK-Ti composites with 0, 15, 30, 45 and 60 vol% Ti were analyzed in terms of microstructure and phase composition. In addition, compressive strength test was performed to study the mechanical properties and *in vitro* pre-osteoblast responses were analyzed.

3.2 Materials and methods

3.2.1. Processing of PEEK-Ti composite

A commercially available PEEK powder (450PF, Victrex USA Inc., Greenville, SC) with a mean particle size of approximately 50 μm and Ti powder (-325 mesh, Alfa Aesar, Ward Hill, MA, USA) were used as received. A PEEK-Ti mixture with 0, 15, 30, 45 and 60 vol% Ti were prepared and shaken to achieve a homogeneous mixture. The mixture was dried overnight in a 70 °C oven to remove any moisture. The mixture was compression molded at 370 °C in Φ12 mm cylindrical molds with uniaxial pressure of 350 MPa.

Samples used for characterizing the composites were prepared by cutting the Φ 12 mm rods into Φ 12 x 1 mm samples. The samples were

polished and ultrasonically cleaned in acetone, ethanol and distilled water. The samples used for the compressive test were obtained by milling the 12 mm diameter rods to 20 mm in length, and the cell test samples were prepared by cutting the 12 mm diameter rods into 1 mm thick discs.

3.2.2. Characterization

Scanning electron microscopy (SEM, JSM-6360, JEOL, Tokyo, Japan) was used to analyze the composition and the microstructure. The cross-sectional images of the samples were observed as well as the fractured surfaces. BSE (back-scattered electron) mode was utilized to distinguish the Ti particles from the matrix. The phase structures of the composite samples were studied using an x-ray diffractometer with a monochromatic Cu Kα radiation source (XRD, Bruker AXS, D8 Advance Diffractometer). The samples were scanned from 20° to 60° at a rate of 1°/min.

3.2.3. Mechanical properties

A compressive strength test was performed to study the mechanical properties of the samples. It was performed at room temperature using Instron 5565 testing system. Specimens were pressed uniaxially with a load cell of 300 kN at a speed of 2 mm/min. The sample was compressed until a fracture occurred. The compressive strength and strain at break was computed using the collected data. The elastic modulus was obtained by computing the slope of the linear region of each sample.

3.2.4. In vitro biological analysis

MC3T3-E1 cells (ATCC, CRL-2593) were used to test the pre-osteoblast attachment, proliferation and differentiation on the prepared samples. All samples for *in vitro* tests were sterilized overnight with UV irradiation. The cells were cultured in alpha-minimum essential medium (α-MEM, Welgene Co., Ltd., Korea) supplemented with 5% fetal bovine serum (FBS) and 1% penicillin-streptomycin. The numbers of cells seeded on the samples were 5×10^4, 1×10^4, 2.5×10^3 for cell attachment, proliferation and differentiation tests, respectively. The samples loaded with cells were cultured in a 37°C humidified incubator with 5% CO_2. For the assessment of cell adhesion and morphology, cells were cultured for 3 hours and fixed with 2.5% glutaraldehyde and were dehydrated in 75, 90, 95 and 100 % ethanol solutions and observed using SEM.

For the proliferation study, cells were cultured for 3 and 5 days and quantified using methoxyphenyl tetrazolium salt (MTS) method. The absorbance measurements were taken at 490 nm using a microplate reader (Biorad, Model 550, USA). For the assessment of cell differentiation, alkaline phosphatase (ALP) activity was assessed using a commercially available kit. After 24 hours, the culturing medium was replaced with ascorbic acid and β-glycerophsphate containing medium which was replenished every 3 days. The cell lysates were quantified using a protein assay kit (BioRad, Hercules, CA) and assayed with *p*-nitrophenyl phosphate kit. Colorimetric changes were quantified using a microplate reader at 405 nm.

3.2.5. Statistical analysis

The data are presented as the mean ± SE of mean. Statistical analysis was performed using a one-way analysis of variance (ANOVA) and p values less than 0.05 were considered to be statistically significant.

3.3 Results

3.3.1. Scanning electron microscopy

PEEK-Ti composites with 0, 15, 30, 45 and 60 vol% Ti contents were reliably produced at molding temperature of 370 °C and 350 MPa. Figure 3 (a-e) shows the cross-sectional SEM micrographs of the polished surfaces of each of the specimens. The Ti particles are well dispersed in the PEEK matrix, and there are no apparent defects in the matrix or the interface of Ti and PEEK. Figure 3 (f) shows the optical image of the samples from a view from the top. The physical appearances of the composites are dramatically changed with the incorporation of Ti powder. BSE images show that Ti powders are relatively well dispersed within the PEEK matrix. The brighter regions correspond to PEEK and the darker regions are Ti. The PEEK is wrapping around the Ti particles without any noticeable cracks or voids. A fractured surface was observed using SEM as shown in Figure 4. Minimal debonding between the Ti fillers and PEEK matrix is observed.

3.3.2. X-ray diffractometer

The XRD patterns of the composites with 0, 15, 30, 45 and 60 vol%

Ti are shown in Figure 5. As expected, with Ti added to PEEK, the relative intensity of PEEK peaks show a decrease and the Ti peaks appear and increases with more Ti added to the sample. As the amount of Ti powders increase, the relative intensities of PEEK peaks decrease and the Ti peaks grow. The relative intensities of the Ti peaks are much higher than the PEEK peaks. No apparent shifts or alterations were observed.

3.3.3. Mechanical properties

The compressive strength test was performed with five samples of each composition. The compressive strength was determined by analyzing the stress versus strain curve of each specimen. Figure 6 shows the typical stress versus strain graph of composite materials with varying Ti contents. The compressive properties of the samples can be characterized as having an elastic deformation followed by plastic deformation and finally leading to fracture. Figure 7 (a) shows the compressive strength of the samples. The compressive strength of the pure PEEK is approximately 132 MPa and it is close to the value which is provided by the manufacturer's data sheet. The strength increases with the increase in the amount of Ti contents. The 60 vol% PEEK-Ti had an average compressive strength of 247 MPa, which is approximately 1.9 times larger than that of the unfilled PEEK. The test also showed that the elastic modulus increased with the increase in Ti content. As shown in Figure 7 (b), the elastic modulus of the specimens showed an increasing trend as well. The modulus increased from 3.1 GPa (unfilled PEEK) to 5.4 GPa (60 vol% PEEK-Ti). The strain at break, on the other hand, showed

a decreasing trend with an increase in Ti content (Figure 7 (c)). The strain at break for unfilled PEEK was 17.7% which dropped to 10.9% with 60 vol% of Ti incorporated.

3.3.4. In vitro biological analysis

The initial biological responses of the samples were studied through osteoblast attachment to the PEEK-Ti composites. Cell attachment images are shown in Figure 8. The degree of attachment increased with more Ti incorporated in the composite. Cells that were cultured on pure PEEK were characterized by round shapes and poor attachment to the surface. However with the PEEK-Ti composites, cells were more securely attached to the surfaces with filopodia extensions and flattening of the cells.

The cell proliferation data for samples cultured for 3 days is shown in Figure 9. The samples show an increase in cell viability from day 3 to day 5, but there are no significant differences between the samples with and without Ti on day 3 values. For day 5, the cell viability of 15 and 45 vol% PEEK-Ti samples had a significant difference compared to the unfilled PEEK with $p < 0.05$. The amount of cell proliferation for 30 and 60 vol% samples showed a significant increase when compared to that of the unfilled PEEK with $p < 0.005$.

The ALP activity of the cells improved with the incorporation of Ti (Figure 10). A significant increase was observed between unfilled PEEK and 30 vol% PEEK-Ti with $p < 0.05$. The increase from 0 to 60 vol% and 15 to 30

vol% were statistically significant with $p < 0.005$.

3.4 Discussion

PEEK-Ti were successfully produced by forming a well-dispersed mixture of the two powders and compression molding it above the melting point of PEEK (T_m = 343 °C) at 370 °C. The cross-sectional SEM images show that there are no apparent defects in the material and that Ti particles are well-distributed in the matrix. The fractured surfaces observed using BSE imaging shows minimal debonding that occurs at the Ti and PEEK interface, but it is not completely detached from the matrix suggesting that sufficient bonding is keeping Ti particles in place. As expected, the XRD diffraction pattern shows no significant change in the phases. The relative intensities of the peaks are altered as more Ti is incorporated but no other changes are present. The fractured surfaces for each of the samples exhibit a torn texture suggesting that the PEEK particles melted and successfully formed a matrix.

The gradual increase in the mean compressive strength as more Ti powders are added to the PEEK matrix suggests that the Ti particles act as reinforcements and restrain the movement of PEEK matrix as the material undergoes deformation. The role of the particles is to hamper the movement of the matrix. It is a general understanding that the extent to which the particle can reinforce the matrix depends on the strong bonding at the PEEK-Ti interface and a good distribution of Ti particles. The increase in the mechanical properties suggests that the physical bonding between PEEK matrix and Ti is relatively strong, which could be a result of strong

mechanical interlocking of PEEK with the well-dispersed Ti particles that distribute the stress throughout the body. Although no chemical bonding exists between PEEK and Ti, the melted PEEK wraps around Ti particles securing them in place as high pressure is applied at high temperature. Since the heat conductivity of the metal particle is much higher (approx. 20 W/m-k) than that of PEEK (0.250 W/m-k), the distributed metal particles allow heat conduction to occur quickly and more evenly throughout the composite.

When stress is applied on the composite material during the compressive test, the stress is partially distributed from the matrix to the particles and a gradual barreling occurs in the specimen as plastic deformation occurs. Eventually the crack propagates and the materials are fractured. The majority of the composites showed fracture occurring along the 45° line. As Ti is incorporated into the composites, the material becomes stiffer and the ductile properties are reduced. Consequently, with the increase in the Ti content, the strain at break decreases as shown in Figure 6.

In terms of cellular responses, with more Ti particles exposed on the surface, it provided regions that are relatively more biocompatible for cell interaction. The degree of which the cells have spread shows significant improvement in the biocompatibility of the material. The number of cells found on Ti surfaces after 3 hours of culturing is much higher than that of the PEEK surfaces. The viability test showed that all samples were biocompatible and that no signs of cytotoxicity are present. The results from the two culturing periods show that pure PEEK showed the lowest viability among the

samples suggesting that Ti has a positive effect on cell growth. On day 3, the cells showed no signs of cytotoxicity and proliferated well on all samples but did not show a significant increase. On day 5, however, a statistically significant increase in cell viability was observed with all PEEK-Ti samples when compared to that of the unfilled PEEK, suggesting that the increase in the Ti content enhanced the biocompatibility of the materials. Finally, cell differentiation results show that by incorporating Ti into PEEK, the ALP activity increased which signifies that PEEK-Ti is a promising alternative as load-bearing biomaterial.

3.5 Conclusion

In our experiment, PEEK-Ti composites with 0, 15, 30, 45 and 60 vol% were successfully produced with compression molding. The mechanical properties of the composites showed that the composite can be strengthened with the addition of Ti particles. The compressive strength of 60 vol% PEEK-Ti was approximately 1.8 times stronger that of the unfilled PEEK. The elastic modulus can be tailored by varying the Ti content as well. The elastic modulus of 60 vol% PEEK-Ti was 7.1 GPa which is approximately 1.9 times greater than the value of unfilled PEEK. In addition, the biological responses of the samples were enhanced by incorporating Ti into PEEK, which was shown through improvements in initial cell attachment, proliferation and differentiation. This study shows promising results for potential use of PEEK-Ti composites as implant materials in the load-bearing orthopedic applications.

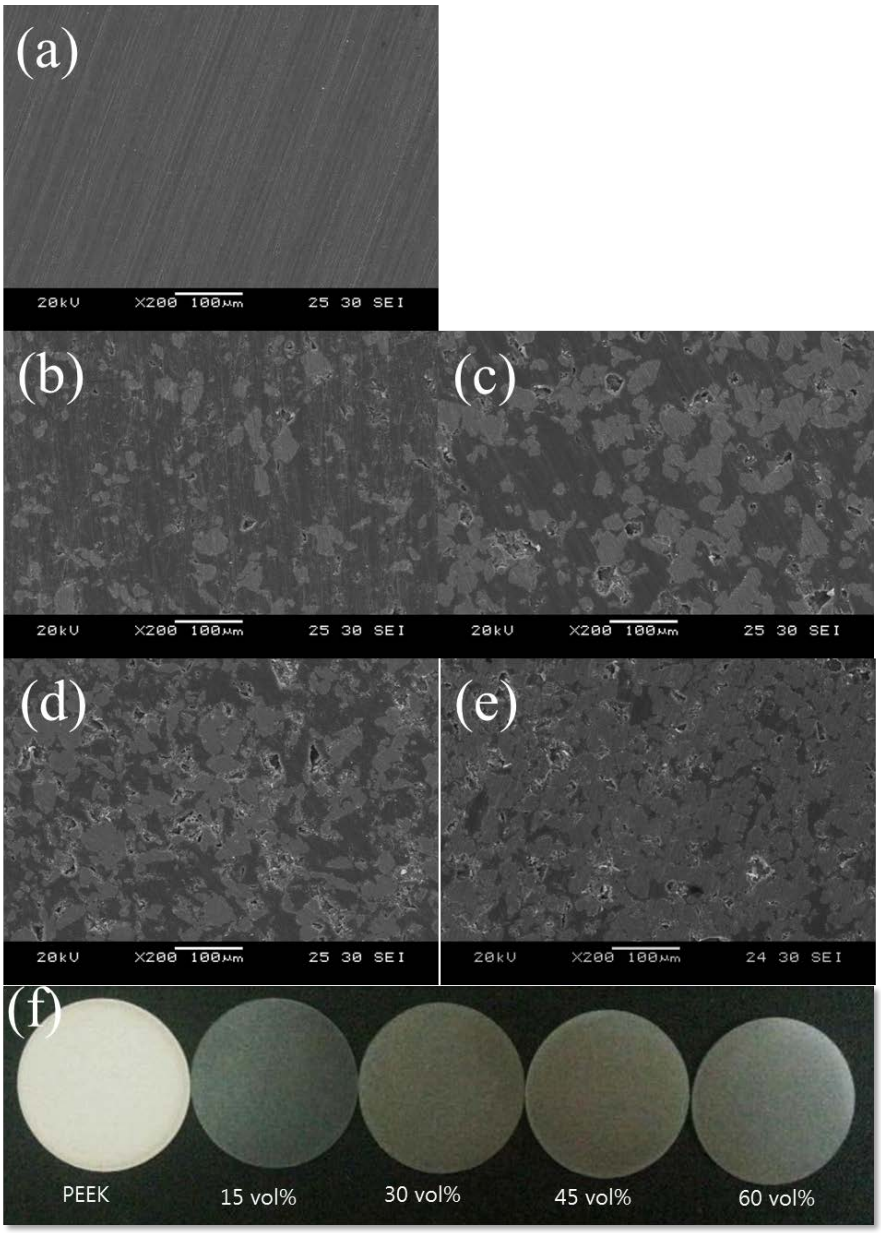

Figure 3. SEM images of (a) unfilled PEEK, PEEK-Ti composites with (b) 15, (c) 30, (d) 45, (e) 60 vol% Ti contents. (f) Optical image of 0, 15, 30, 45 and 60 vol% PEEK-Ti (left to right).

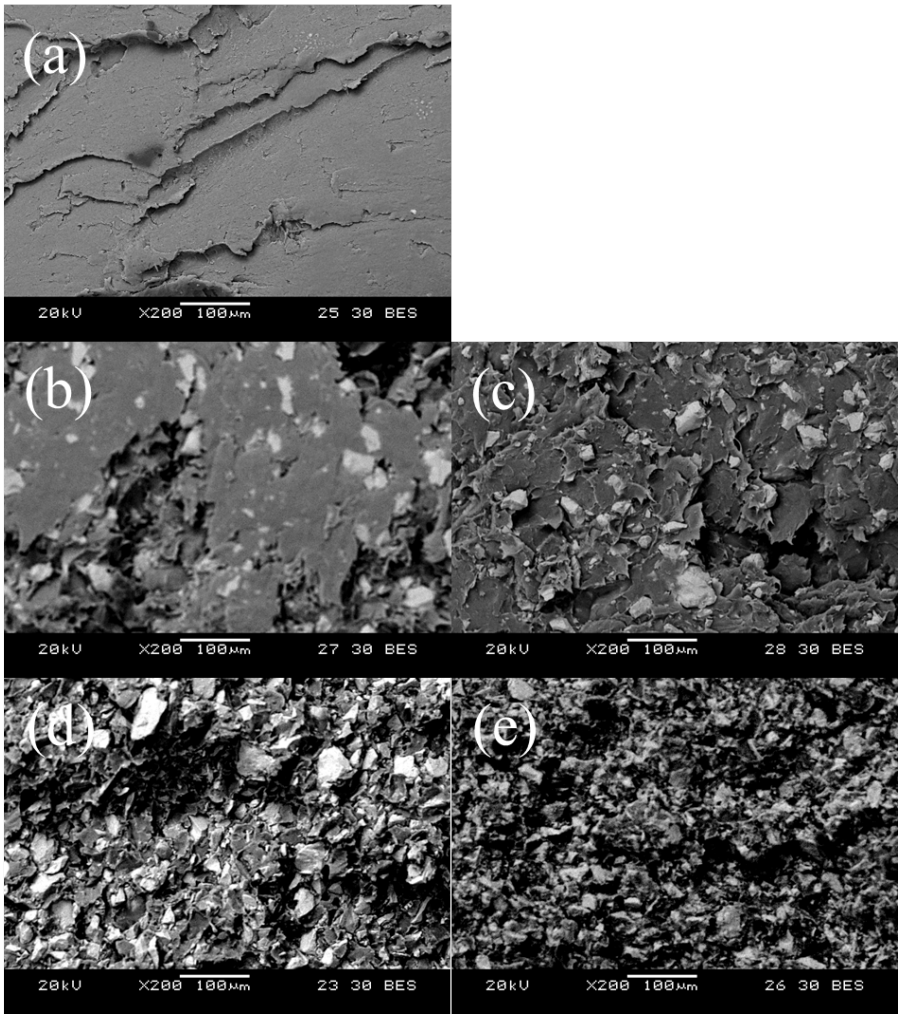

Figure 4. SEM images of the fractured surfaces of (a) unfilled PEEK, PEEK-Ti composites with (b) 15, (c) 30, (d) 45 and (e) 60 vol% Ti contents.

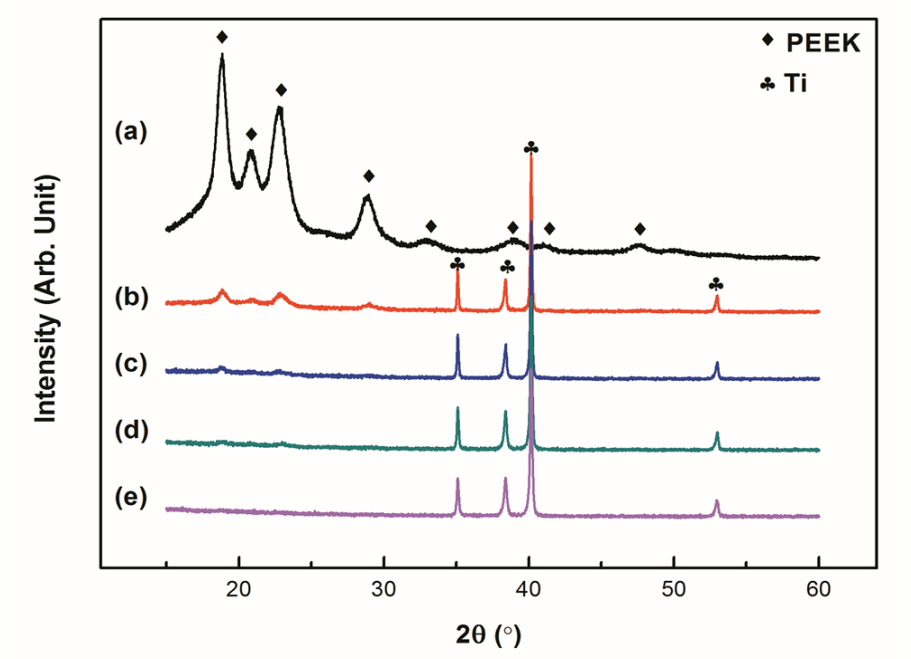

Figure 5. XRD patterns of (a) PEEK, PEEK-Ti composites with (b) 15, (c) 30, (d) 45 and (e) 60 vol% Ti contents. The PEEK and Ti peaks are marked with ♦ and ♣, respectively.

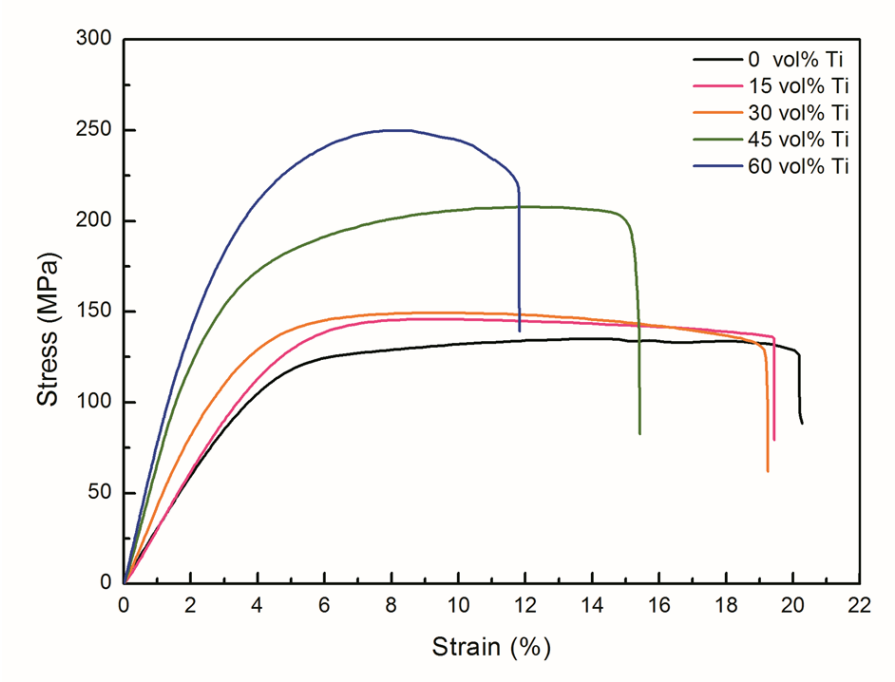

Figure 6. Typical stress-strain curves of PEEK-Ti composites

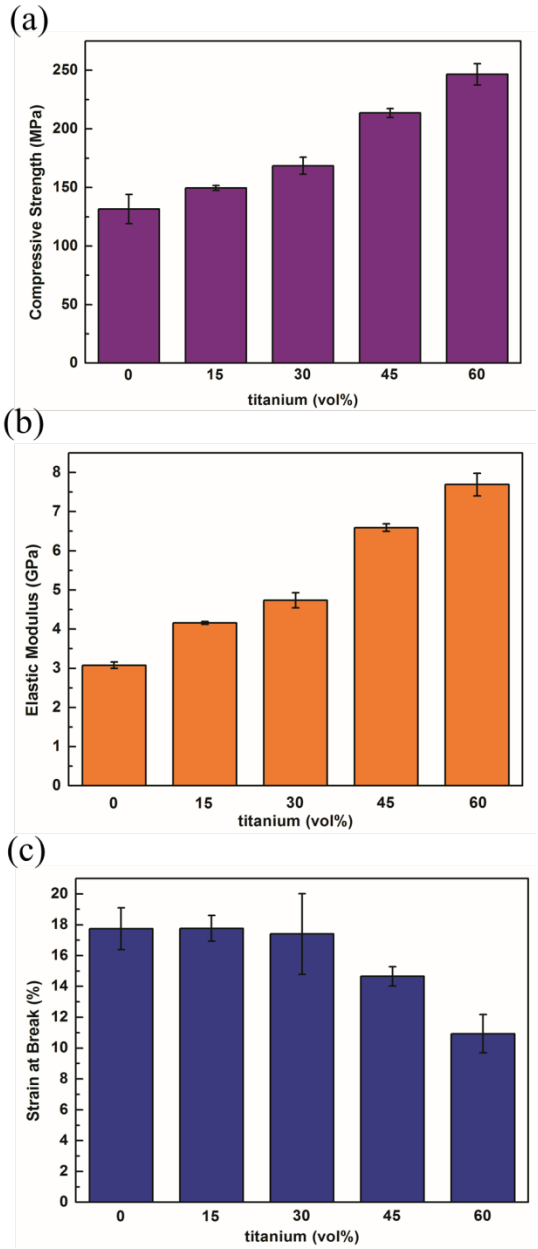

Figure 7. Mechanical properties of PEEK-Ti composites. (a) Compressive strength, (b) elastic modulus, (c) strain at break of 0, 15, 30, 45 and 60 vol% PEEK-Ti composites.

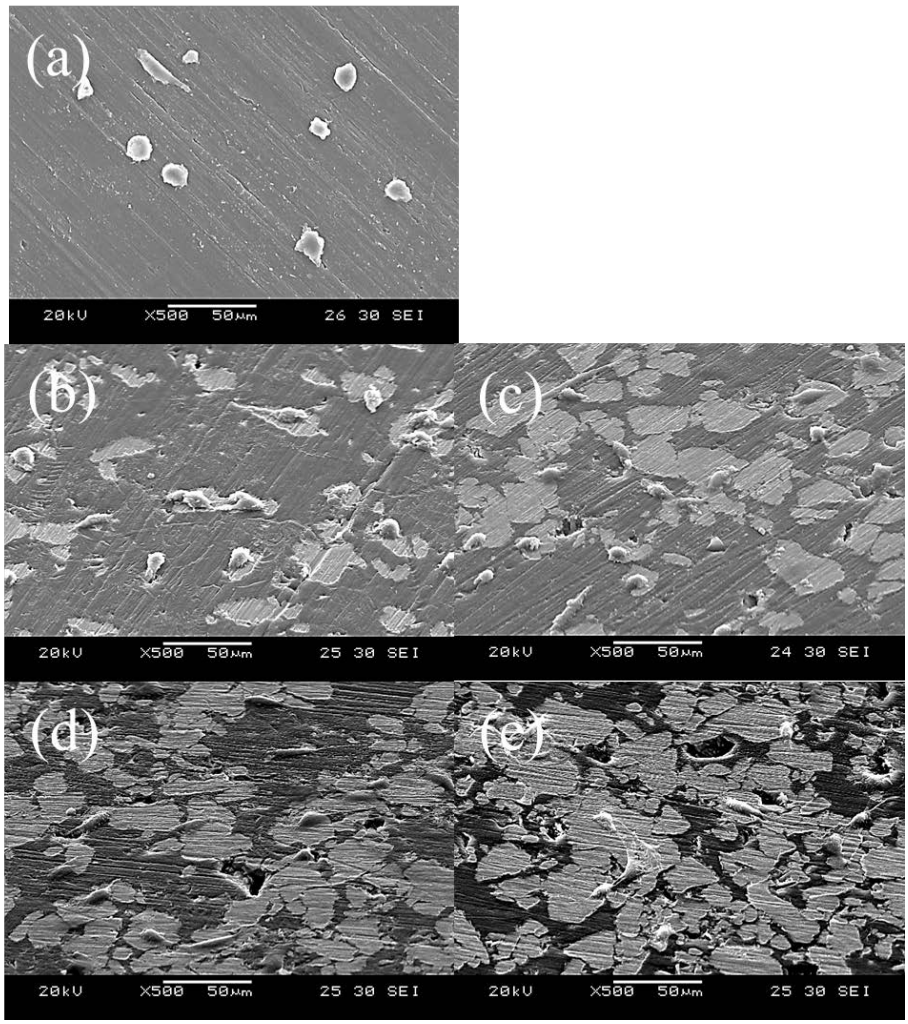

Figure 8. SEM images of cell attachment on (a) unfilled PEEK, PEEK-Ti composites with (b) 15, (c) 30, (d) 45, (e) 60 vol% Ti contents.

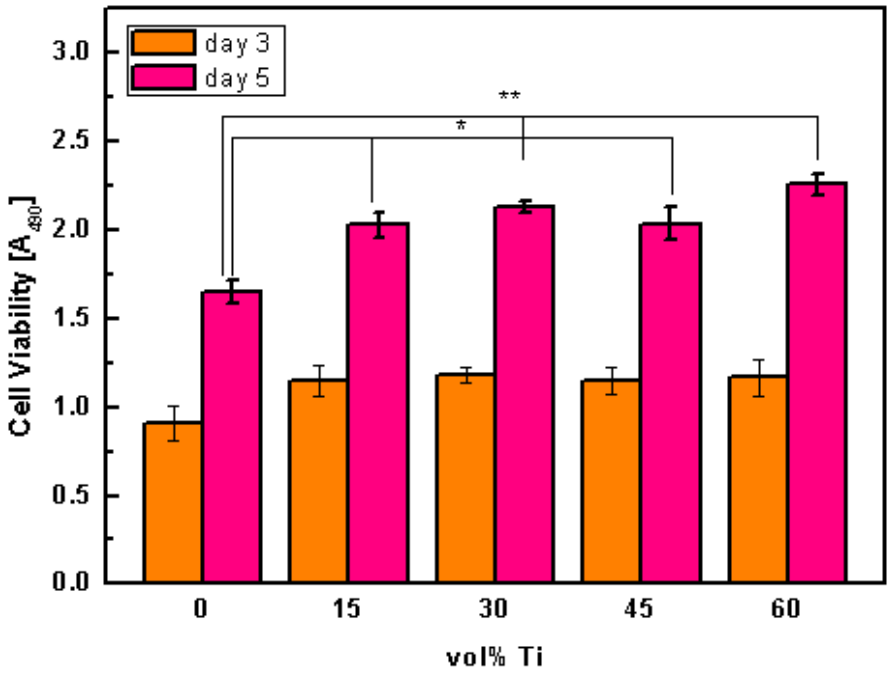

Figure 9. Cell viability of PEEK-Ti composites cultured for 3 and 5 days with 0, 15, 30, 45 and 60 vol% Ti contents.

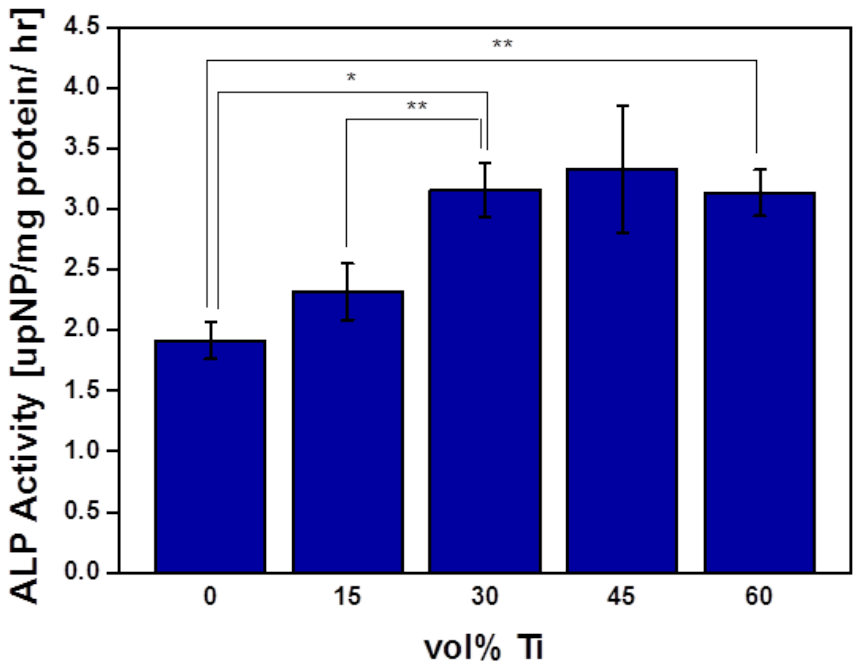

Figure 10. ALP activity of cells cultured for 10 days with 0, 15, 30, 45 and 60 vol% Ti contents.

Chapter 4

Polyetheretherketone-magnesium composites for potential biomedical applications

4. Polyetheretherketone-magnesium composites for potential biomedical applications

4.1. Introduction

Polyetheretherketone (PEEK) is a widely used material in medical applications. It has outstanding biocompatibility as well as mechanical properties. PEEK has high strength when compared to other polymers and low elastic modulus compared with metal implants [3]. However, PEEK is biologically inert and is not conducive to fast bone cell attachment, which often results in improper fixation at the defect site. In severe cases, loosening of the implant could occur [43]. In this study, PEEK-Mg composites are investigated for potential use as implant materials which provides better initial cell-to-implant interaction and enhanced long-term fixation of the implant with the bone as well.

With the society's growing interest in biodegradable materials, Mg is a promising material that is being intensively studied for medical applications. Mg is considered a revolutionary metallic biomaterial since it gradually dissolves in physiological environment and has no toxicity in the body [31, 37]. Its mechanical properties are closer to those of the natural bone when compared to those of other metals. Moreover, Mg ions are reported to be beneficial in bone tissue growth in some studies [31, 33, 44].

However its high corrosion rate is still a challenge that needs to be solved for use in physiological environment. Mg rapidly reacts in aqueous

solutions producing by-products like hydroxide ions and hydrogen gas. This results in an increase in the local pH and generation of a large volume of hydrogen gas. One common approach to delay the corrosion of Mg is to coat its surface so that the rate of corrosion can be reduced. Various coating layers were invested to delay the corrosion and enhance the bioactivity at the same time [34, 45-47]. Among various materials hydroxyapatite (HA) is widely known. HA is a crystalline form of calcium phosphate that is similar to the mineral present in bone with a Ca to P molar ratio of approximately 1.67 [48]. Its composition is $Ca_{10}(PO_4)_6(OH)_2$ and it has excellent bioactivity which can improve the implant-to-cell bonding and control the degradation rate of Mg[32]. When using biodegradable materials for implants, it is important to control the rate of degradation and enhance the bioactivity to provide a cell-friendly surface.

In this study, PEEK-Mg was fabricated to enhance the bone-to-implant interaction chemically and mechanically. More specifically, a composite material with a thin surface layer of PEEK-Mg on top of bulk PEEK was analyzed to test the feasibility of the novel approach that provides bioactive substance on the surface and pores which allows for better fixation of PEEK implants.

Mg has two roles: (1) to promote bone tissue growth and (2) to provide stronger fixation of the implant as the Mg is degraded and bone in-growth occurs. HA was coated on the exposed Mg surfaces to decrease the rate of corrosion and to provide bioactive substances on the surface that can

enhance the early stage cell-implant interaction.

In order to test the feasibility of the Mg/PEEK composites, *in vitro* cell tests were carried out using a pre-osteoblast line to study the preliminary cellular responses of the coated and uncoated composite materials. The mechanical properties of the composites were evaluated using compressive test. Finally, the initial corrosion properties were evaluated using simulated body fluid (SBF) and the morphologies of the corroded surfaces were studied at various intervals.

4.2. Materials and methods

4.2.1. PEEK-Mg composite processing

A commercially available PEEK powder (450PF, Victrex USA Inc., Greenville, SC) with a mean particle size of approximately 50 μm and Mg powder (-20+100 mesh, Alfa Aesar, Ward Hill, MA, USA) were used as starting materials. PEEK powder was dried overnight at 70 °C oven to remove any excess moisture. PEEK-Mg mixtures with 0, 15, 30, 45 and 60 vol% of Mg were prepared and compression molded at 370 °C in 12 mm cylindrical molds with a uniaxial pressure of 350 MPa. The processed samples were cut into 1 mm thick discs, polished and ultrasonically cleaned in acetone, ethanol and distilled water. All evaluations were performed using the prepared Φ12 x 1 mm samples except for the ALP analysis in which Φ30 x 1 mm thick samples were used and the compressive test in which Φ12 x 20 mm samples were used.

4.2.2 Hydroxyapatite coating on exposed magnesium surfaces

The HA coating method suggested by Hiromoto et al. was used to coat the exposed Mg on the surfaces [40, 49, 50]. In brief, a solution with 0.05 M ethylenediaminetetraacetic acid calcium disodium salt hydrate (Ca-EDTA) and 0.05 M potassium dihydrogenphosphate (KH_2PO_4) was prepared and the pH was adjusted to 8.9 using sodium hydroxide. The solution temperature was raised to 90 °C for the coating process and was stirred at 200 rpm during the coating procedure. The samples were treated in the solution for at least 2 hours then rinsed with distilled water and dried.

4.2.3. Characterization

The surface morphology and the composition of the composites were observed using scanning electron microscopy (SEM, JSM-6360, JEOL, Tokyo, Japan). The cross-sectional images of the samples were observed as well as the fractured surfaces. Field-emission electron microscope (FESEM; JEOL, JSM-5410LV) and focused ion beam milling was used to estimate the thickness of the coating layer on the Mg surfaces. The samples were deposited with platinum and milled to observe the coating layer. The phase structures of the composite samples were studied using an x-ray diffractometer with a monochromatic Cu Kα radiation source (XRD, Bruker AXS, D8 Advance Diffractometer). The samples were scanned from 15° to 60° at a rate of 1°/min.

4.2.4. In vitro biological analysis

MC3T3-E1 cells (ATCC, CRL-2593) were used to test the pre-

osteoblast attachment, proliferation and differentiation on the prepared samples. All samples were sterilized with UV irradiation prior to seeding the cells. The cells were cultured in alpha-minimum essential medium (α-MEM, Welgene Co., Ltd., Korea) supplemented with 5% fetal bovine serum (FBS) and 1% penicillin-streptomycin. The densities of the cells seeded on the samples in 24-well plate were 3 x 10^4, 1 x 10^4, 2.5 x 10^3 cells/mL for cell attachment, proliferation and differentiation tests, respectively. The samples seeded with cells were cultured in a 37 °C humidified incubator with 5% CO_2.

For the assessment of cell adhesion and morphology, cells were cultured for 3 hours, fixed with 2.5% glutaraldehyde, dehydrated in ethanol solutions (75, 90, 95 and 100 %), and immersed in hexamethyldisilazane. The morphology of the cells was observed using SEM. Cell viability was studied using the number of cells residing on the samples. The cells were cultured for 3 days and were trypsinized and trypan blue and counted using a hemocytometer.

4.2.5. Mechanical properties

The mechanical properties of the samples were characterized using a compressive test. The test was performed at room temperature using Instron 5565 testing system (Instron Corp., Canton, MA, USA). The sample rods were compressed with a load cell of 300 kN at a speed of 2 mm/min. Five specimens were pressed uniaxially and the compressive strength, elastic modulus and strain at break were calculated.

4.2.6. Corrosion properties

The coated and uncoated PEEK-Mg samples were each immersed in 30 mL SBF to evaluate the surface corrosion properties by monitoring the pH change. The SBF was prepared according to the recipe suggested by Kokubo et al [51]. The pH of the solution was measured using a pH meter (sp-701, Suntex, Taiwan) at 12-hour intervals. The sequential morphology on the surface as the corrosion progresses was observed using 30 vol% Mg samples at 6 hours, 1 week, 2 weeks and 3 weeks post-immersion using SEM. In order to visualize the final porous morphology of which all the Mg on the surface is eliminated, 1M hydrochloric acid was used to accelerate the Mg corrosion and the pore morphology was observed using SEM.

4.2.7. Statistical analysis

All the data are presented as mean ± SE of mean. Statistical analysis was performed using a one-way analysis of variance (ANOVA) and p values less than 0.05 were considered statistically significant.

4.3. Results

4.3.1 Composite processing

PEEK-Mg composites with 0, 15, 30, 45 and 60 vol% Mg were successfully fabricated. The SEM images of the polished surfaces in Figure 11 shows that Mg particles are reasonably well distributed in the PEEK matrix. There are no visible voids at the Mg and PEEK interface suggesting that no

debonding is present between Mg particles and PEEK matrix.

4.3.2 HA coating on Mg

The hydrothermal treatment method previously reported by Tomozawa and Hiromoto was utilized to coat the exposed Mg surfaces [40, 49, 52]. The SEM images of the surface before and after the coating procedure are shown in Figure 12, respectively. The pre-treatment image shows the polished surface of 45 vol% PEEK-Mg composite. The post-treatment surface imags showed dome-shaped HA layer covering the exposed Mg particles after treating the sample for 2 hours in the coating solution. Rod-like HA crystals were observed on the dome shaped HA layer. Figure 13 shows the XRD patterns of PEEK, 30 vol% PEEK-Mg, and HA-coated 30 vol% PEEK-Mg. Only the XRD patterns of PEEK and Mg were present in PEEK-Mg composite analysis. Once the samples were coated, HA peaks appeared. FESEM image after FIB preparation showed rod-like HA crystals that have grown on Mg (Figure 14). The approximate thickness of the coating layer is 3 μm after 2 hours of coating.

4.3.3 In vitro biological properties

The surface morphology and the cell morphology following 3 hours of culturing are shown in Figure 15. The cells that were seeded on uncoated Mg samples showed minimal attachment to the surface. However, on the coated samples, the cells have a flatter, more spread morphology on both the PEEK and the Mg surfaces. The cell count was performed to analyze the

proliferation of cells on the composites. Figure 16 shows the cell counts for each of the composites. For the uncoated samples, the cells proliferated less than the unfilled PEEK, and fewer cells were attached on the surface with increasing amounts of Mg on the surface. On the other hand, with the coated Mg on the surface, the number of cells on the 15 vol% Mg samples was almost double that of the unfilled PEEK samples. With increasing amounts of Mg, however, the number of cells found on the surface decreased. When comparing the uncoated and coated samples of the same compositions, more cells were adhered and proliferated on the coated surface than the uncoated surface.

4.3.4 Mechanical properties

Figure 17 shows the morphologies of the fractured surfaces following the compressive tests. The morphology implies that crack propagation occurred along the metal and polymer interface. Figure 18 (top) shows the compressive strength of the composites. The compressive strength of the composites increased slightly with the addition of Mg into PEEK. The differences are not statistically significant, which implies that the compressive strength of the composite materials are not altered much with the incorporation of Mg. The elastic modulus graph is shown in Figure 18 (bottom). Incorporation of Mg particles resulted in slightly stiffer materials. The elastic modulus of 60 vol% Mg composite was approximately 1.5 times higher than that of the unfilled PEEK.

4.3.5 Corrosion properties

The corrosion properties of PEEK-Mg composites were evaluated by immersing them in SBF. The uncoated and coated PEEK-Mg composites were soaked in SBF and Figure 19 shows the pH versus time graph. The initial pH rise is reduced by coating the Mg surface with HA. The morphology of the samples soaked in SBF for 6 h, 1 week, 2 weeks and 3 weeks are shown in Figure 20. Much of Mg is corroded away on week 3 of the corrosion test. Figure 21 shows the images of the composites treated with HCl to eliminate the Mg particles. Relatively well-dispersed spherical pores with diameters of approximately 500 μm are visible.

4.4. Discussion

Mg-PEEK composites with 0, 15, 30, 45 and 60 vol% Mg fillers were successfully compression molded. Due to the big difference in particle size of PEEK and Mg powder, a close to perfect distribution of was not reached. However, utilizing ethanol in the mixing process allowed for better distribution of the Mg particles as shown in Figure 11. No apparent defects in the processed materials were present.

After treating the samples in the coating solution, an HA coating layer was formed on top of the exposed Mg surfaces. The HA crystals grown on the samples had similar morphologies to the previously reported HA coating layer from the Hiromoto's group [40, 49, 50].

In vitro cellular tests were performed to study the cellular behaviors on the uncoated PEEK-Mg composites and were compared to the HA-coated PEEK-Mg composites. In culturing mediums Mg surfaces go through an aggressive corrosion reaction. However, as time proceeds the corrosion products form a protective layer on the surface and the rate of corrosion is decreased. The cell attachment on the uncoated Mg surfaces was minimal due to the fast corrosion reaction on the surface and the production of by-products interfering with the attachment of cells. Hydrogen gas formation and the increase in pH of the culturing medium could have had adverse effects on the cell attachments. On the other hand, cells on the coated Mg surfaces showed more stable attachment and their morphologies were more spread out when compared to those on the uncoated Mg surfaces. This suggests that HA coating layer is crucial for better initial cell interactions on Mg surfaces.

To characterize the proliferation of cells on the HA-coated PEEK-Mg and uncoated PEEK-Mg, cell counting was performed to quantify the number of cells residing on the material on day 3 of culturing was performed. The results showed that in a closed system in which no flow exists, excessive amounts of Mg on the surface can be detrimental to cells since the coating on the Mg particles are not perfectly protective from the aqueous environment. A better prediction of the PEEK-Mg composite for use as biomaterials can be achieved in a system that can mimic the physiological environment or through *in vivo* animal tests.

The mechanical properties test showed that the failure of the

composite is initiated at the interface at which PEEK and Mg is weakly bonded. The detached Mg particles from the matrix show that the crack occurred at metal-PEEK interface and propagated around the Mg particles as the material deformed. Along with the debonding of the Mg particles, neighboring cracks join and form longer cracks which result in the failure of the material.

The mechanical test of the samples showed minimal change in the compressive strength with Mg particles acting as reinforcements in the matrix. The results demonstrate that no significant change in the mechanical properties occurs as a result of adding Mg particles into the PEEK matrix. Therefore, these results show that PEEK-Mg composites can be used on the surfaces of bulk PEEK to enhance the fixation of the implant without the tradeoff in mechanical properties.

In order to predict and characterize the corrosion rate of the composites, the pH change was monitored to quantitatively analyze the corrosion behavior of the samples. As expected, the coated samples showed a delay in the corrosion process when compared to the uncoated samples of the same compositions. The rate of corrosion is further reduced as corrosion products such as Mg hydroxide and Ca, P-containing substances form a layer on the bare Mg surface. The gradual increase in pH of the solution containing the coated sample suggest that the corrosion of Mg can be controlled so that the hydrogen gas formation and local pH increase can be reduced in the vicinity of Mg. In physiological environment, there is a circulation of body

fluids thus moderate amounts of hydrogen gas and pH change can be tolerated. The delay in the corrosion process will provide time for the circulation of body fluid to eliminate the reaction products.

The architecture of the composites once all the Mg has been corroded was simulated using hydrochloric acid to dissolve the Mg particles. The pores that are formed at places where Mg particles were present have an approximate diameter of 500 μm, which can allow bone tissues to infiltrate and integrate with the implant providing stronger fixation of the implant.

The aim of the study was to investigate the feasiblity of using PEEK-Mg composite as an implant material to allow for better ingrowth of the cells to enhance the fixation of PEEK implants. The results of this study demonstrate that HA-coated PEEK-Mg shows hopeful results for use in the orthopedic applications.

4.5. Conclusion

PEEK-Mg composites with 0, 15, 30, 45 and 60 vol% were successfully produced with compression molding. The mechanical properties of the composites showed that strength of the material does not significantly change with the incorporation of Mg particles. However, the elastic modulus increased from 3.1 GPa (PEEK) to 5.4 GPa (60 vol% PEEK-Mg). In addition, the biological responses of the samples showed that incorporating Mg into PEEK and coating the exposed surfaces with HA enhances the biocompatibility. However, our study was performed in a closed-system

which can have negative effects on the cells. Mg-PEEK can be further investigated for potential usage in the medical field. By using HA-coated PEEK-Mg composite layer on the surface of PEEK implants, HA offers better cell-implant interaction. Furthermore, bone integration with the implant can be achieved with the degradation of Mg. Ideally, HA-coated PEEK-Mg can stimulate bone growth and allow for quicker recovery of the bone tissue cells and provide stronger fixation of the implant as bone grows into the pores formed on the surface.

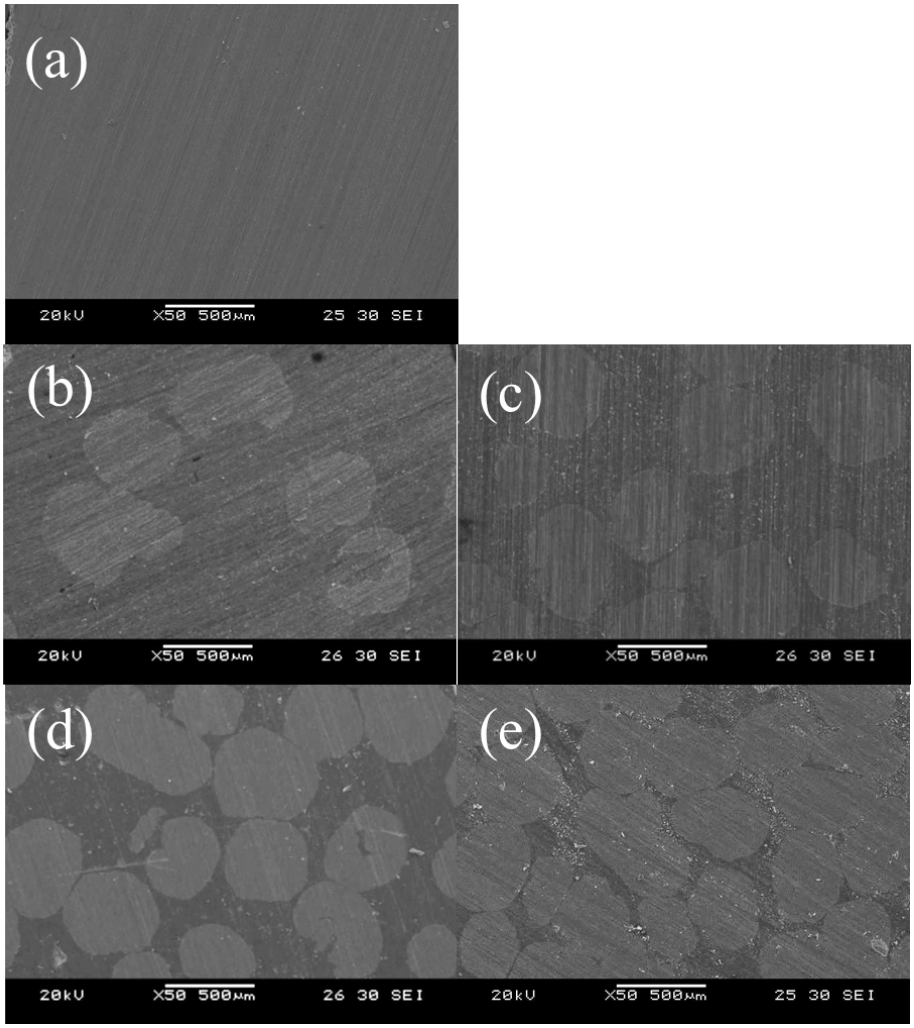

Figure 11. SEM images of (a) unfilled PEEK, PEEK-Mg composites with (b) 15, (c) 30, (d) 45 and (e) 60 vol% Mg contents.

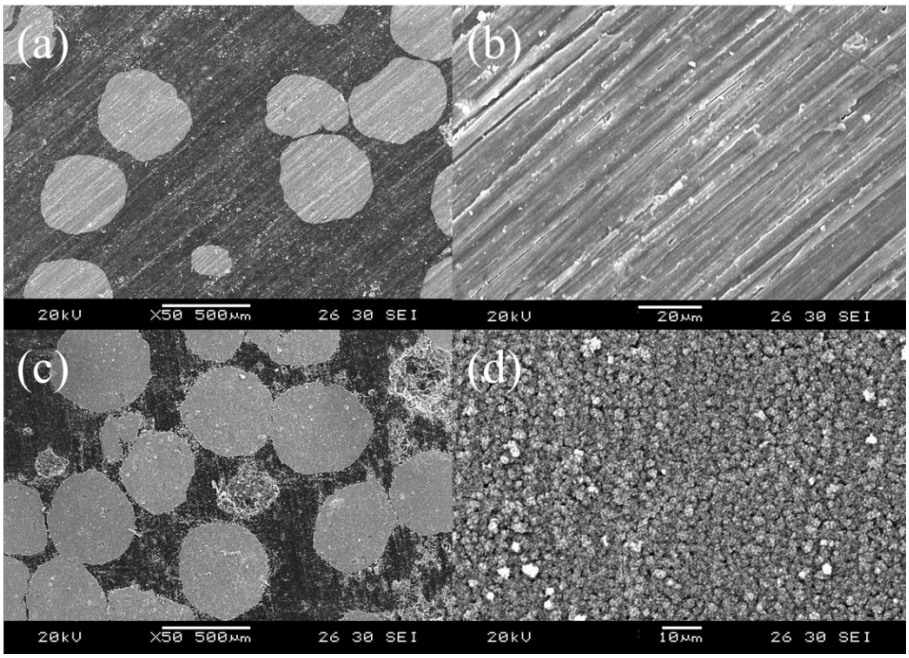

Figure 12. SEM images of (a) PEEK-Mg surface, (b) higher magnification of the Mg surface, (c) coated PEEK-Mg surface (d) higher magnification of the coating layer on Mg surface.

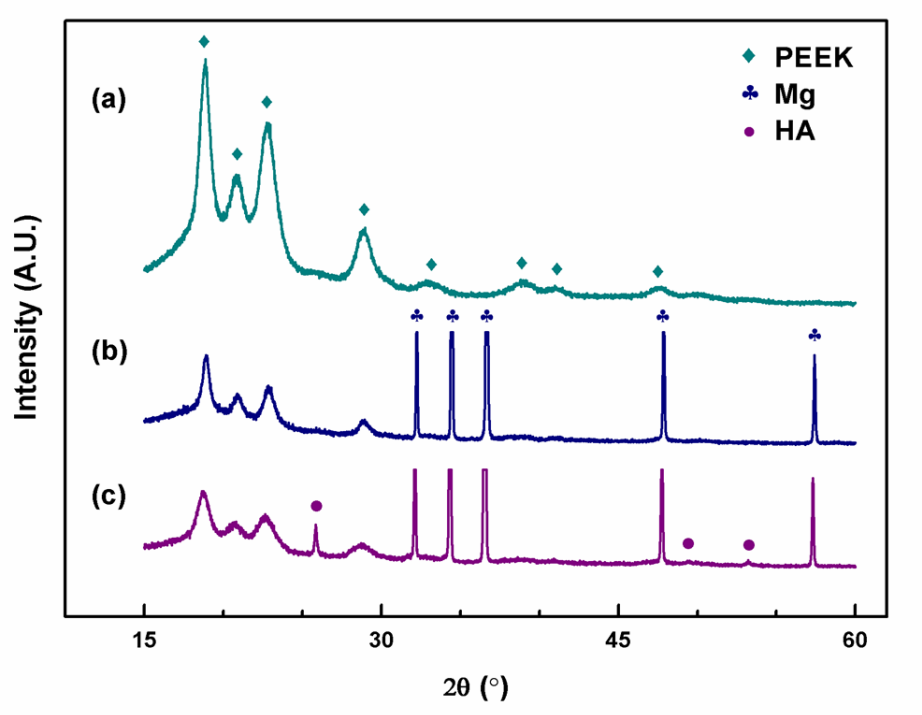

Figure 13. XRD diffraction pattern of (a) unfilled PEEK, (b) 30 vol% PEEK-Mg, (c) HA-coated 30 vol% PEEK-Mg.

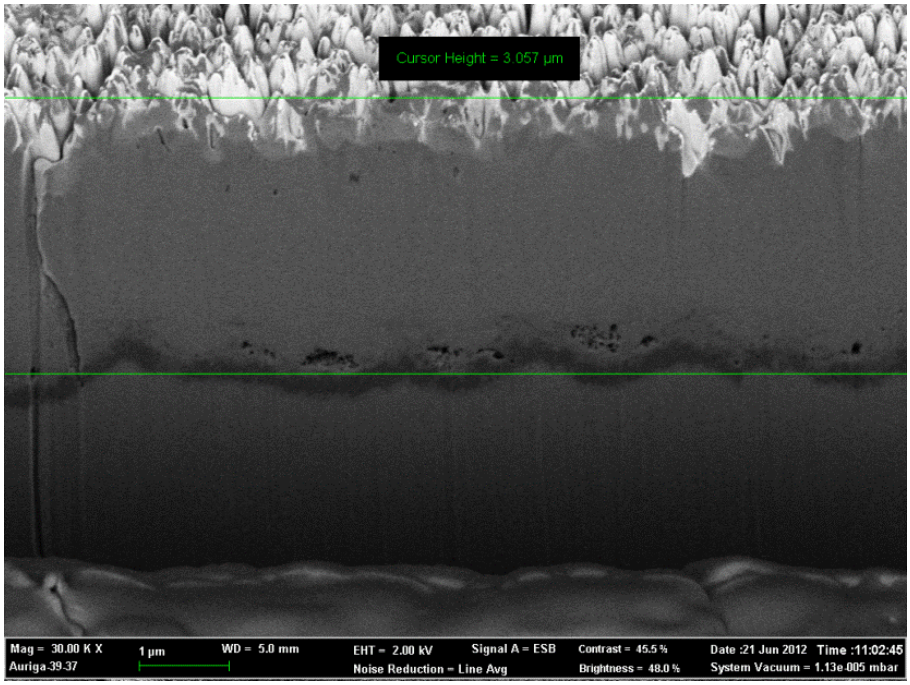

Figure 14. FESEM of HA coating layer. The coating layer thickness is approx. 3µm.

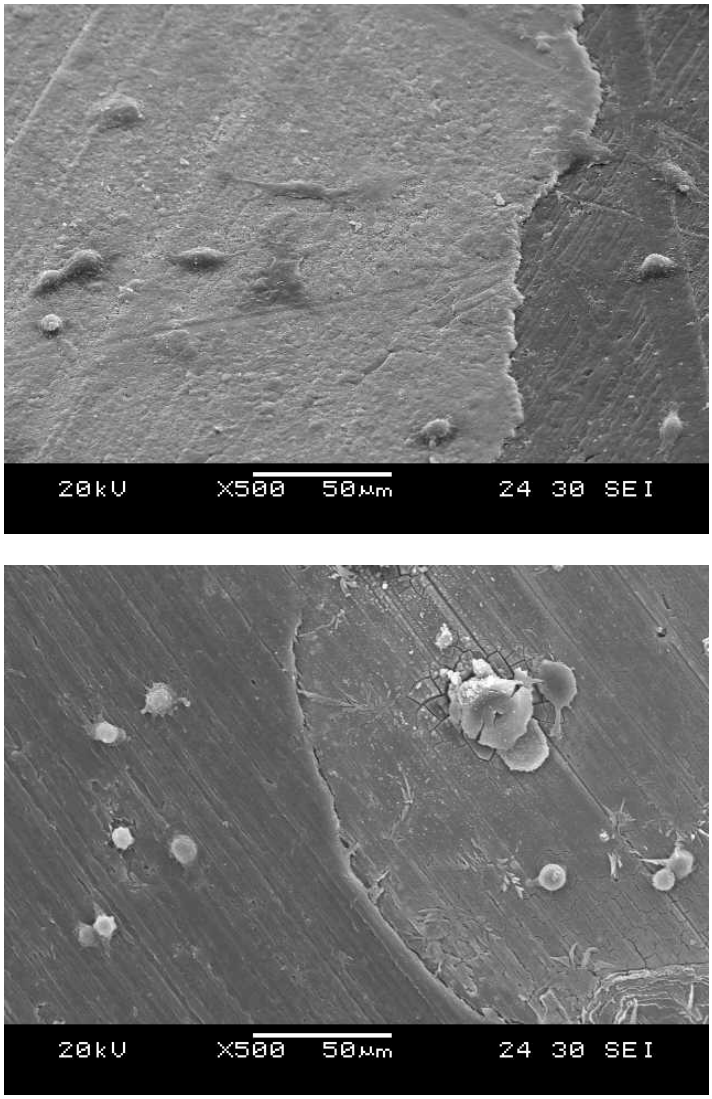

Figure 15. SEM images of the cells on bare Mg surface (top) and HA-coated surface (bottom) after 3 hours of culturing.

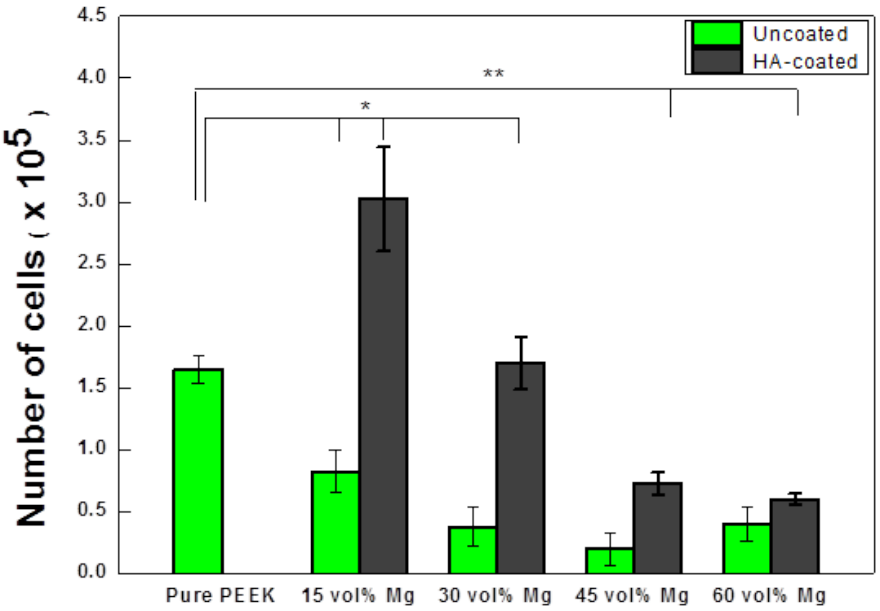

Figure 16. Cell count of the samples after 3 days of culturing for 0, 15, 30, 45 and 60 vol% PEEK-Mg.

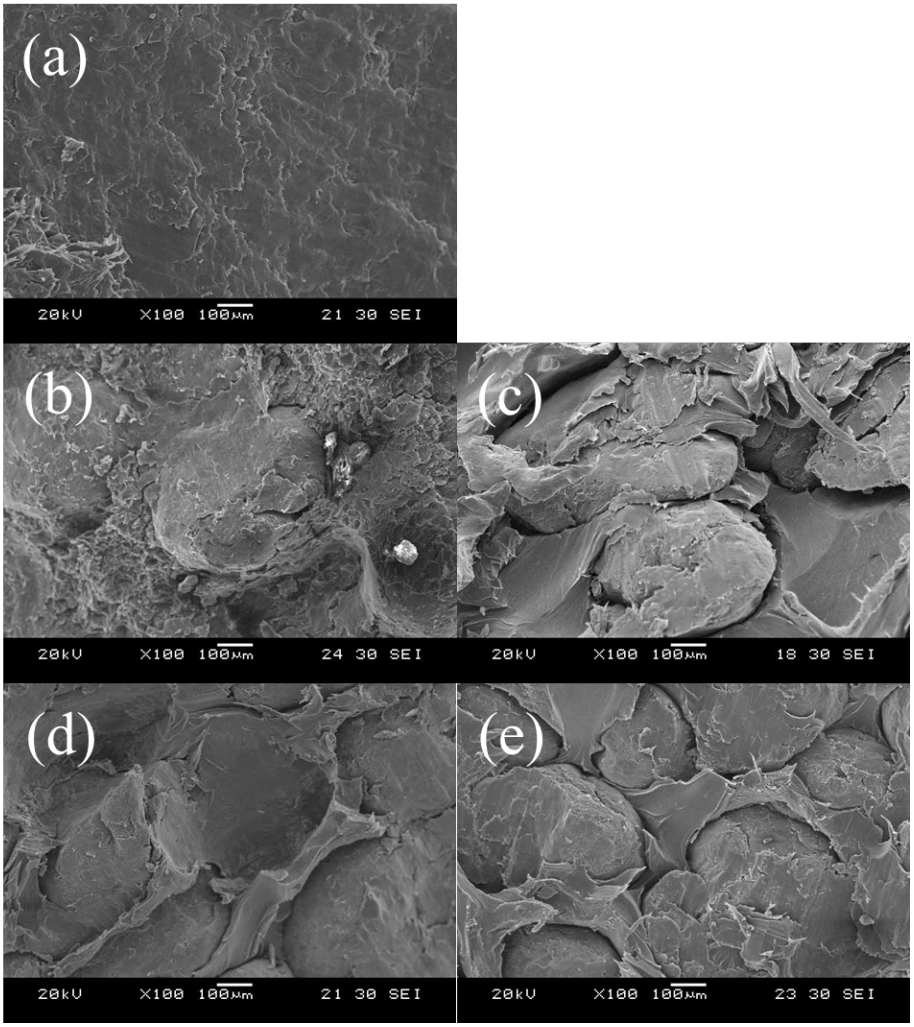

Figure 17. Fractured surfaces of (a) 0, (b) 15, (c) 30, (d) 45 and (e) 60 vol% PEEK-Mg composites.

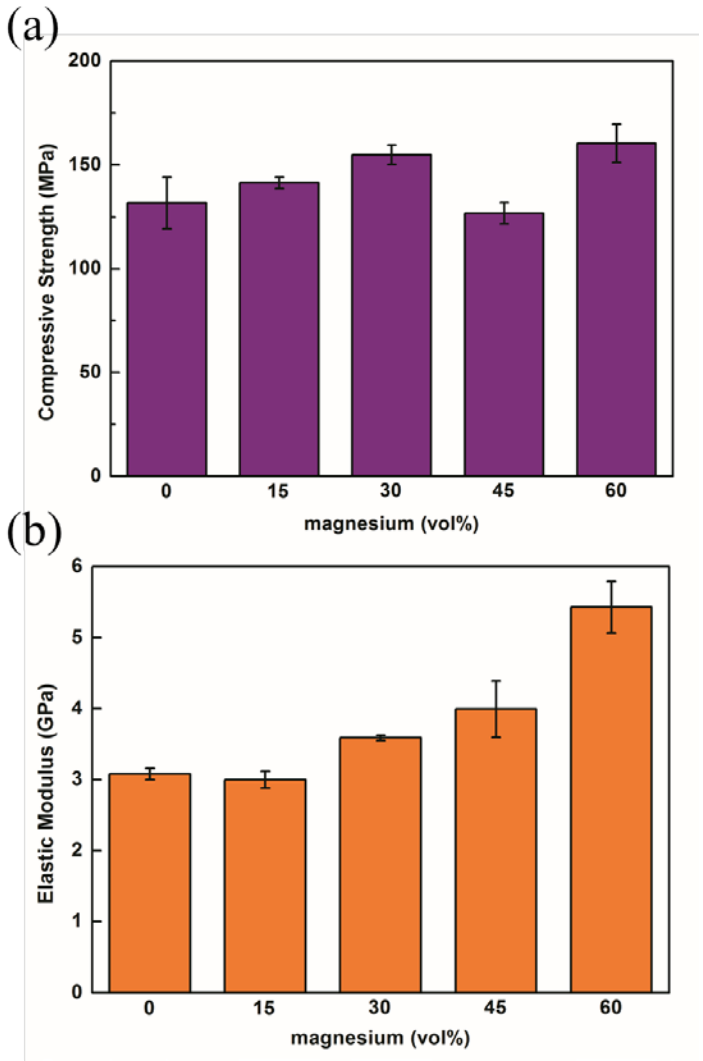

Figure 18. Compressive strength and elastic modulus of PEEK-Mg composites.

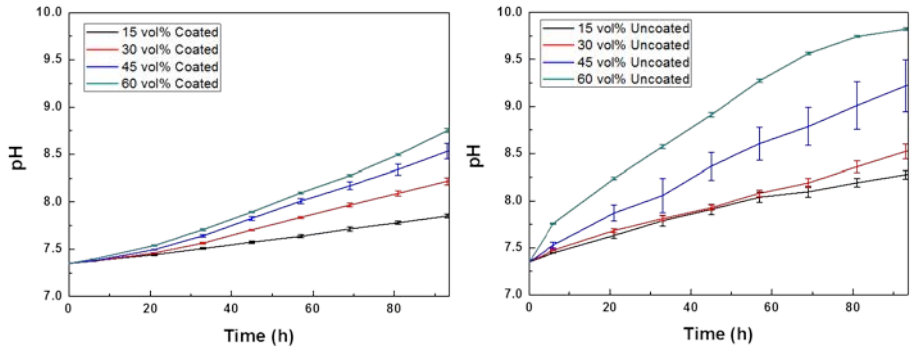

Figure 19. pH change in SBF with PEEK-Mg samples with 0, 15, 30, 45 and 60 vol% Mg. The left graph shows HA-coated samples and the right graph shows the uncoated PEEK-Mg composites.

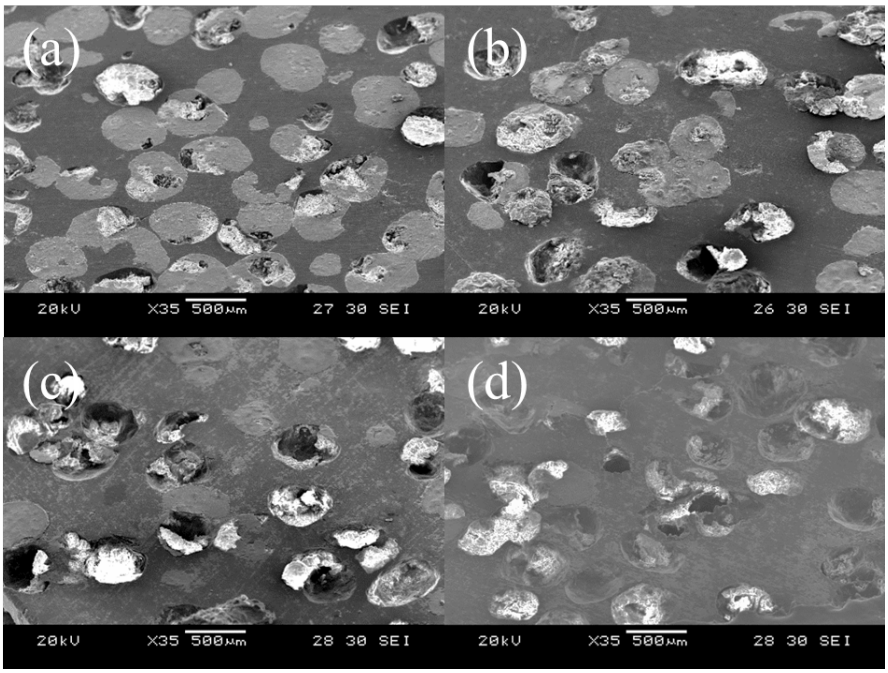

Figure 20. SEM images of the corroded surfaces after (a) 6 h (b) 1 week (c) 2 weeks (d) 3 weeks of immersion in SBF.

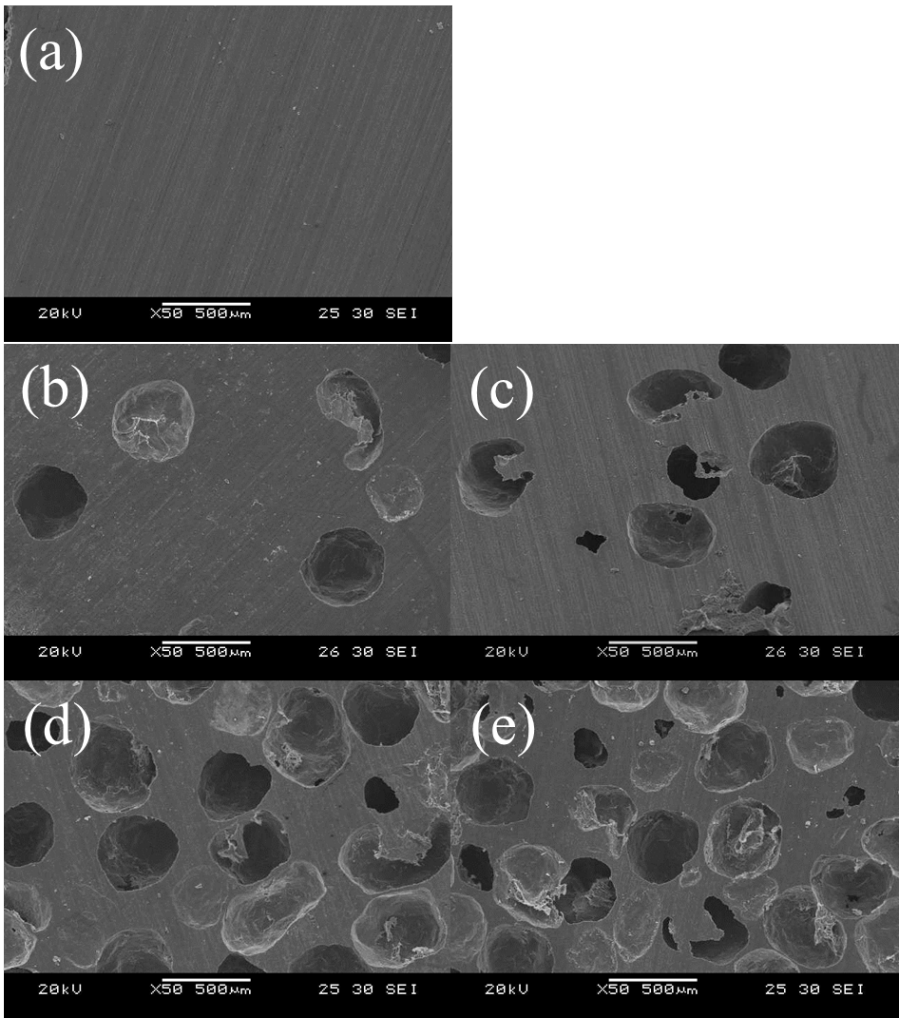

Figure 21. SEM images of the 0, 15, 30, 45 and 60 vol% PEEK-Mg surfaces after HCl treatment.

Chapter 5

Conclusion

5. Conclusion

PEEK is a thermoplastic that has numerous qualities that make it a good biomaterial. However, PEEK is relatively bioinert. In efforts to solve the problem, PEEK-metal composites were fabricated using compression molding and evaluated for potential use in orthopedic applications. Metal contents of 0, 15, 30, 45 and 60 were successfully fabricated. PEEK-Ti and PEEK-Mg composites have shown promising results in terms of biological and mechanical properties for use in the medical field.

Ti is a material that is currently used in the medical field due to its excellent biocompatibility and mechanical properties. The expected outcome of combining PEEK and Ti is that biological and mechanical properties will be enhanced. As expected, by increasing Ti content in PEEK, increasing trends in compressive strength and elastic modulus and cellular activities were observed while a decreasing trend was noticed in Young's modulus. For example, the 60 vol% PEEK-Ti had a compressive strength of 247 MPa and an elastic modulus of 7.1 GPa which is much higher when compared to the unfilled PEEK with a compressive strength of 132 MPa and an elastic modulus of 3.7 GPa.

Mg is a unique metal that is capable of being degraded when exposed to aqueous environment (such as the human body) and has beneficial effects on the growth of osteoblasts as well. PEEK-Mg composite took a different approach to solving the problem. Instead of making a more biocompatible bulk material, PEEK-Mg was aimed to provide a stronger

mechanical interlock at the surface as bone infiltrates the pores that are formed as Mg is degraded. Bare Mg undergoes rapid degradation in aqueous environment and can be detrimental in the body; therefore bioactive HA was coated on the exposed Mg surface using an aqueous treatment method. The coating layer was able to reduce the rate of corrosion and at the same time provide bioactive substances on the surface of the implants for better cellular interactions. The mechanical properties of the PEEK-Mg composites showed no significant change in the compressive strength but a slightly increasing trend was observed in the elastic modulus (unfilled PEEK: 3.1 GPa, 60 vol% PEEK-Mg: 5.4 GPa). The post-corrosion pore structure was observed and preliminary cellular tests show that PEEK-Mg composites have good initial cellular responses.

Two different types of PEEK-metal composites were fabricated and evaluated to improve PEEK as a biomaterial. The study showed that by combining biocompatible metal particles such as Ti and Mg into PEEK matrix the biological properties can be enhanced and the mechanical properties can be tailored to some extent depending on the size and the properties of the metal particles. In addition by using biodegradable material on the surface of PEEK, a better fixation of the implant is expected as bone grows into the pores to secure the implant in place.

Although these results of the preliminary investigations show that combining metal into PEEK can enhance the biological properties, a more detailed study is necessary to evaluate their full potential for use in the

medical field. Some of the subsequent concerns that need to be addressed are consistency of the composites, *in vivo* results that show the post-implantation effects of the materials in physiological environment, optimization of the fabrication process, optimization of the biological and mechanical properties, molecular interaction between the material and the cells, and long-term effects of the metal-PEEK composites in physiological environment.

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초 록

폴리에테르에테르케톤 (PEEK)는 강도, 인성 및 화학적 안정성이 우수하여 정형외과용 임플란트 재료로서 매우 유망한 재료로 평가되고 있다. 하지만 생체불활성 재료이기 때문에 임플란트 시술 후 골과의 접합이 잘 되지 않는다는 단점이 있다. 본 연구에서는 PEEK-금속 복합체를 제조하여 이러한 문제를 해결하려는 연구가 진행되었다. 금속은 생체적합성이 높고 강도가 높은 티타늄과 생분해성 성질이 우수한 마그네슘이 사용되었고 각각에 대해 다양한 평가를 했다.

PEEK의 강도 및 생체적합성을 증진하기 위해 PEEK-티타늄 복합체를 제조하였다. PEEK와 티타늄 분말을 섞은 후 고온에서 압축하였고 다양한 티타늄의 분율 (15, 30, 45 and 60 vol%) 에 관계없이 균일한 시편이 형성되었다. 티타늄의 분율이 0 vol%에서 60 vol%으로 증가함에 따라 압축 강도는 132 MPa에서 247 MPa로 증가하였고 강성도는 3.1 GPa에서 7.1 GPa로 증가하였다. 세포 초기 접착, 증식 및 분화능 평가 실험을 통하여 티타늄의 분율이 증가할수록 우수한 생체 친화성을 보인다는 것을 확인할 수 있었다.

초기엔 생체적합성이 증진되고 생분해가 일어난 뒤엔 골과의 접합성이 증가되도록 PEEK-마그네슘 복합체를 제조하였다. 복합체 제조 후 마그네슘의 분해 속도를 조절하기 위해 바이오세라믹인

하이드록시아파타이트가 코팅되었다. 코팅된 하이드록시아파타이트의 두께는 약 3 um였고 다양한 마그네슘의 분율(15, 30, 45, 60 vol%) 에 관계 없이 균일한 시편이 형성되었다. 마그네슘의 분율이 0 vol%에서 60 vol%으로 증가함에 따라 압축 강도는 큰 변화가 없었던 반면 강성도는 3.1 GPa에서 5.4 GPa로 증가하였다. 표면 코팅을 하였을 때 세포 초기 접착, 증식 및 분화능 평가 실험에서 우수한 생체 적합성을 확인할 수 있었고 생체유사용액 내에서 부식저항성 또한 크게 향상시켰음을 일 수 있었다.

따라서 본 연구를 통해, PEEK-금속 복합체를 제조하여 기계적 물성 및 생체적합성을 증진 시킬 수 있음을 보였고, 이를 통하여 특성화된 치과 및 정형외과용 임플란트 소재로 사용될 수 있음을 보여주었다.

주요어: 폴리에테르에테르케톤, 고분자 복합체, PEEK-금속, 생체적합성, 기계적 특성, 정형외과용 임플란트

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