JOURNAL OF Applied Polymer SCIENCE

Processing of a Multi-Layer Polyetheretherketone Composite for Use in Acetabular Cup Prosthesis

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ABSTRACT: The hydroxyapatite polyetheretherketone (HAPEEK) as a non-degradable bioactive polymer composite material with coating of hydroxyapatite (HA) as a bioactive ceramic material can enhance the osteointegration of carbon fiber reinforced polyetheretherketone (CFRPEEK) as a non-degradable bioinert polymer composite. This study describes the joining process of CFRPEEK and HAPEEK beam components and coating process of HA on the HAPEEK substrate to achieve the multi-layer PEEK composite for use in the application of acetabular cup prosthesis. The CFRPEEK and HAPEEK components were ultrasonically welded while the HA was plasma sprayed on the HAPEEK substrate. Ultrasonic welding parameters (length and direction of the energy directors at the interface, welding time, and pressure) were investigated by single cantilever beam and lap shear tests to achieve the optimum bonding strength of CFRPEEK and HAPEEK components. Plasma spraying parameters (e.g., surface speed, powder feed, current, primary gas flow, and system voltage) were altered to achieve the good adhesion of HA coating on the HAPEEK substrate, which was evaluated by scratch test. The results showed that the proposed multi-layer composite was successfully processed by carrying out the ultrasonic welding and plasma spraying coating processes. The outcomes of this study could be used to develop a non-metal acetabular cup prosthesis using the proposed multi-layer composition. © 2014 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2014**, *131*, 40915.

KEYWORDS: acetabular cup; CFRPEEK; HAPEEK; lap shear test; SCB test; ultrasonic welding

Received 10 March 2014; accepted 26 April 2014
DOI: 10.1002/app.40915

INTRODUCTION

By growth of total hip replacement surgeries in the world and the problems of the current commercial acetabular cups, it has been essential to develop the new acetabular cup composition incorporating the new biomaterials. Lewis et al.[1] and Chowdhury et al.[2] reported that in the load-bearing hip joint, an appropriate material for the liner (bearing portion of the acetabular cup) is crucial as wear debris from the material has been associated with osteolysis and loosening of the implant. Carbon fiber reinforced polyetheretherketone (CFRPEEK) has been studied by Latif et al.[3] for use in acetabular cup prosthesis. Based on the Kurtz and Devine[4] review article, CFRPEEK is a non-degradable material with superior fatigue and mechanical properties which can stand for long-term under compression loading of body weight. The minimal range of debris generated has been achieved for this material due to the contact wear. The mechanical properties of this material have been developed close to that of cortical bone to avoid stiffness mismatch and bone resorption due to stress shielding.[5] Scholes et al.[6] showed that the CFRPEEK material has had better wear resistance than UHMWPE. Hydroxyapatite polyetheretherketone (HAPEEK) composite material is non-degradable. It is also bioactive (due to the HA particles inside the composite) and could make a strong bonding with bioactive HA coated material. HAPEEK has been investigated by Converse et al.[7] as a potential tissue engineering scaffold and it was achieved that HAPEEK could enhance the bone-implant integration. Its mechanical and biological properties have also been reported by Abu Bakar et al.[8] and Rashidi et al.[9] by which the good load bearing characteristics of the HAPEEK material have been achieved. Likewise, Abu Bakar et al.[8] revealed that by increasing the amount of HA, the brittleness of the composite would increase as a result. Paital and Dahotre[10] reported that HA is a bioactive degradable material, which could effectively coat on the orthopedic implants to enhance the osteointegration. CFRPEEK and HAPEEK are both non-degradable and has PEEK polymer by which the strong bonding of two composites could be achieved. The load bearing advantage of HAPEEK can enhance the mechanical strength of the CFRPEEK layer. Conversely, HA and HAPEEK are both bioactive, thereby the fusion of HA coating to the HAPEEK substrate could be enhanced compared to the bioinert CFRPEEK substrate. In fact, the HAPEEK layer acts as a tie layer between CFRPEEK and HA coating layer.

Injection-overmolding method has been used by Bateman and Scott[11] and Brooks et al.[12] to fabricate various two layers acetabular cups with different melt temperatures. Because of the existence of PEEK polymer in both CFRPEEK and HAPEEK materials, the injection-overmolding method could not be effectively used for joining of these two composites. Ultrasonic welding process has been intended by Liu et al.[13] and Troughton[14] for welding of thermoplastic polymers. The advantageous of ultrasonic welding has been reported by Troughton[14] as a process that does not generate contaminants to the weld area that may influence the biocompatibility of the implant. However, this process has not been used for joining of the CFRPEEK and HAPEEK composite materials. Kiratisaevee[15] and Reyes and Cantwell[16] used single cantilever beam (SCB) test to evaluate the adhesion of various hybrid compositions of polymers while lap shear test was used for evaluation of the welding strength of the ultrasonically welded components in recent published articles.[17–19]

The physical and chemical vapor deposition coating methods were used by Kwok et al.[20] for the coating of bioactive ceramics (e.g., HA) on the titanium substrates. These methods could not be effectively used for non-electricity induction polymer composite materials (e.g., PEEK composites). Campbell et al.[21] and Martin et al.[22] used successfully the plasma spraying coating process to coat the HA on the carbon fiber reinforced polyamide 12 and pure PEEK substrates, respectively. The advantages of this process are simplicity, high deposition rate, and high porosity. This process is a non-electricity process that could be used for polymer composite material. However, the plasma spraying coating has not been used to coat the HA on the HAPEEK substrate which was contributed in this investigation. The scratch test has been efficiently performed to investigate the adhesion between the substrate and coating layer.[20,23] Vencl et al.[24] revealed that the scratch test eliminated the risk of penetrating epoxy or glue into the coating layer compared to the pull-off test.

The objective of this study was to contribute the manufacturing processes of a multi-layer composition comprised of CFRPEEK, HAPEEK, HA materials, which could enhance the wear resistance and osteointegration of the acetabular cup prosthesis. A multi-layer PEEK composite has been achieved using the ultrasonic welding process for joining of the HAPEEK and CFRPEEK components and plasma spraying coating process for coating of HA on the HAPEEK substrate. SCB, lap shear, and scratch tests were used in this study to assess the welding strength and coating adhesion. The results showed that the proposed multi-layer composite was successfully processed by carrying out the ultrasonic welding and plasma spraying coating processes, which could be used to develop a non-metal acetabular cup prosthesis.

Figure 1. Shape of energy directors at the interface.

MATERIALS AND METHODS

Materials

The CFRPEEK granules with 30% (by weight) chopped carbon fibers (MOTIS G, Invibio) were used in the experiments. Polyetheretherketone polymer (PEEK-OPTIMA-LT1, Invibio) and hydroxyapatite ($Ca_5(PO_4)_3(OH)$, Sigma-Aldrich) were used to produce HAPEEK polymer composite granules with 20% weight of HA.

Component Fabrication

Single screw extrusion machine was used to compound HA and PEEK. The HAPEEK compound was processed in granule form. Because of the high melting temperature (343°C) of PEEK polymer (compared to the other polymers) and high melt viscosity of 0.73 kNs/m^2 for MOTIS G and 0.44 kNs/m^2 for PEEK-OPTIMA, higher temperature range controllers and ceramic heaters were installed on the injection molding machine to allow maximum temperature of up to 450°C. The injection pressure and mold temperature were set to 150 MPa and 210°C, respectively. HAPEEK and CFRPEEK granules were separately fed into the injection molding machine to obtain the HAPEEK and CFRPEEK specimens in the dimension of $140 \times 15 \times 3$ mm^3. The components were cut to $120 \times 12 \times 3$ mm^3 for SCB test based on the specimen dimensions that Kiratisaevee[15] used to perform the SCB test. The specimens for lap shear test were cut according to the ASTM D3163.[25]

Ultrasonic Welding

The ultrasonic machine with the high output power of 2.8 kW (KSONIC^{TM}) was used to perform the ultrasonic welding experiments. In this process, the horn is transmitting the ultrasonic energy to the interface while it is vibrating to convert the ultrasonic energy to the thermal energy for welding of the components at the interface. The vibration produces a sharp rise to the melting temperature and transforms the energy directors to a flow state. Then the melted material is solidified under pressure. The application of weld pressure influences the joint formation as it keeps the specimens to be joined in intimate contact while ultrasonic energy generates heat by friction. The intimate contact eliminates the presence of air gaps and hence reduces energy loss. V-based energy directors (0.6 mm in height and width) were created on the interface surfaces (Figure 1). The holding time and the vibration amplitude were set to 3 s

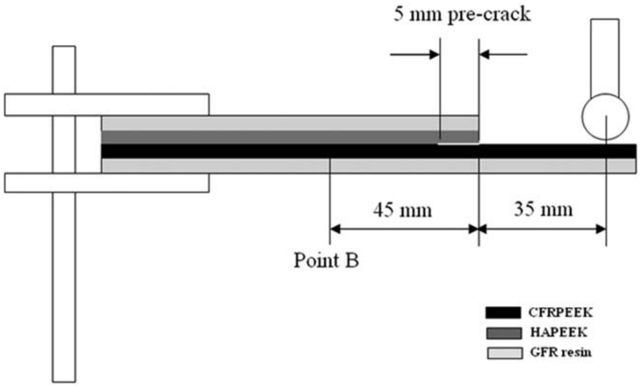

Figure 2. Schematic view of SCB test setup.

and 80 μm, respectively, whereas the pressure and welding time were varied to obtain the optimum parameters.

SCB Test

To perform the SCB test, the combined HAPEEK/CFRPEEK specimens were fixed at one end and an increasing load at a rate of 1.2 mm/min was applied to the other end (Figure 2). A pre-crack with a length of 5 mm was made as shown in Figure 2. During the testing, the crack was allowed to propagate 40 mm from the pre-crack. To maintain the rigidity of the HAPEEK and CFRPEEK layers, woven glass fiber resin (GFR) beams were adhesively bonded to the composite (Figure 2). This test was used to evaluate the effect of the energy director length and direction on the strength of bonding between two PEEK composite components. The SCB experiments were performed three replicates per experiment for the whole and partial energy director forms for various GFR thicknesses. In the partial form, the energy directors were produced from the pre-crack point up to point B (Figure 2) while in the whole form, the energy directors were made on the whole interface surfaces.

The debonding force of CFRPEEK/HAPEEK interface was measured using eq. (1)

$$F_d = F_T - (F_{\mathrm{CFRPEEK}} + F_{\mathrm{GFR}}), \tag{1}$$

where F_d is the debonding force and F_T is the total force (recorded by the testing machine). F_{CFRPEEK} and F_{GFR} are the bending forces of the CFRPEEK and GFR layers, respectively, which were obtained by eq. (2).

$$F = \frac{3\delta EI}{L^3}\left(I = \frac{bh^3}{12}\right), \tag{2}$$

where F is the force, δ is the extension, E is the Young's modulus, I is the second level of torque, b, h, and L are the width, thickness, and length, respectively. Table I shows the constant values that were used to calculate the F_{CFRPEEK} and F_{GFR}.

Lap Shear Test

The CFRPEEK and HAPEEK specimens were joined by ultrasonic welding process according to the ASTM D3163.[25] Trial experiments were needed to determine the ranges of the welding time and pressure at which the weld bonding would occur. Then the maximum, minimum, and medial values were selected to define the nine experiments (three replicates per experiment) for the two variables and three value levels. A servo-controlled universal testing machine was used to carry out the lap shear test. Adjustable clamping grips were used to ensure that the applied tensile force was in line with the overlapping section and prevent unnecessary bending during the testing. The shear strain energy was calculated using eq. (3) to represent the shear properties of the welding joint.

Table I. Geometrical and Material Properties of CFRPEEK and GFR Beam Layers

Parameters	CFRPEEK beam layer	GFR beam layer
E (GPa)	13	20
L (mm)	100	100
b (mm)	12	12
h (mm)	3	3, 3.5, 4, 5

$$U = \frac{3P^2L}{5Gbh}, \tag{3}$$

where P is the debonding force (tensile shear force), G is the shear modulus of the PEEK (1.15 GPa), L is the total thickness of the HAPEEK and CFRPEEK beam specimens (6 mm), b is the width of the specimens (12 mm), and h is the overlap length (12 mm).

Plasma Spraying Coating and Scratch Test

The plasma spraying experiments (two replicates per experiments) were performed to achieve the appropriate fusion of HA to the HAPEEK substrate by altering the plasma spraying parameters. The plasma spraying coating parameters for first experiment were contemplated based on the[22] research in which the plasma spraying coating of HA was evaluated on the PEEK substrate. The adhesion of HA-coated layer was determined by the scratch tester. This test evaluated the strength of the coating layer by making a scratch line. Although the indenter was making the scratch line, the applied load increased linearly from 0.3 N until the force at which the indenter touched the substrate. The applied force (normal force), friction force, and penetration depth were recorded.

RESULTS

SCB Test

Figure 3 shows the debonding of the HAPEEK/CFRPEEK components at the interface. The crack was initiated and propagated at the interface line. The welding joint was gradually debonded while the load was increasing.

The influence of energy director length on the welding strength of HAPEEK/CFRPEEK is indicated in Figure 4. It is apparent that, by decreasing the length (partial energy director), the debonding force is enhanced. The debonding force was obtained 74.6 ± 6.2 N and 106 ± 7.3 N for the whole and partial energy directors, respectively. Likewise, Figure 4 displays that the debonding force, F_d, is constant by altering the GFR thicknesses.

Lap Shear Test

The appropriate ranges of ultrasonic welding parameters for the lap shear test were attained by trial experiments based on the

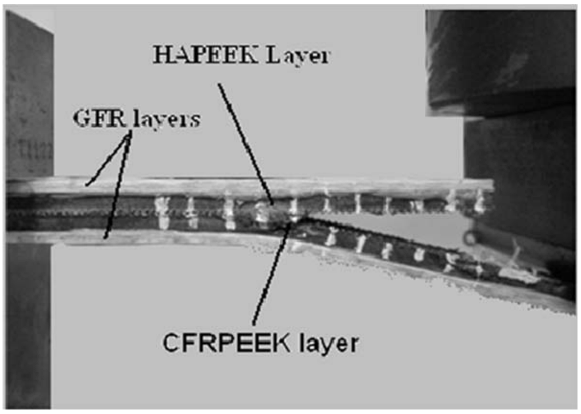

Figure 3. Debonding of CFRPEEK and HAPEEK at the welded interface in SCB test. The debonding is gradually propagated at the interface by increasing of the applied force.

Table II. Debonding Tensile Shear Force and Shear Strain Energy of Lap Shear Test Experiments

Exp. no.	Welding time (s)	Pressure (MPa)	Debonding force (N)	Shear strain energy (mJ)
1	3.5	0.6	1237 ± 25	33.3 ± 1.4
2	3.5	0.7	1303 ± 28	36.9 ± 1.6
3[a]	3.5	0.8	1392 ± 35	42.1 ± 2.1
4	4	0.6	812 ± 15	14.3 ± 0.5
5	4	0.7	853 ± 19	15.8 ± 0.7
6	4	0.8	892 ± 18	17.3 ± 0.7
7	3	0.6	605 ± 14	8.0 ± 0.4
8	3	0.7	660 ± 18	9.5 ± 0.5
9	3	0.8	744 ± 22	12.0 ± 0.7

[a] Optimum experiment.

ultrasonic welder power and horn material. The ranges of 3–4 s for welding time, 0.6–0.8 MPa for pressure, and 2–3 s for cooling time were achieved for the welding of CFRPEEK/HAPEEK interface. Table II shows the debonding force of the nine lap shear experiments for various welding time and pressure values. The highest debonding force of 1392 ± 35 N achieved for the experiment no. 3 at which the welding time and pressure were 3.5 s and 0.8 MPa, respectively. The related shear strain energy was obtained 42.1 ± 2.1 mJ. From the data in Table II, the significant effect of the welding time on the debonding force could be observed compared to the pressure. The maximum debonding force was achieved for the medial value of the welding time at each pressure values. Conversely, the maximum debonding force was achieved for the upper value of pressure at each welding time. Similar dependency of shear strain energy to the welding time and pressure could be observed in Table II due to the direct proportion of shear strain energy with the applied force [eq. (3)].

Figure 5 illustrates the remained HAPEEK material on the CFRPEEK component after debonding. In this figure, the existence of large amount of the HAPEEK material on the CFRPEEK component [Figure 5(b)] in optimum experiment (experiment no. 3 in Table II), revealed the robust adhesion of ultrasonically welded of CFRPEEK and HAPEEK components.

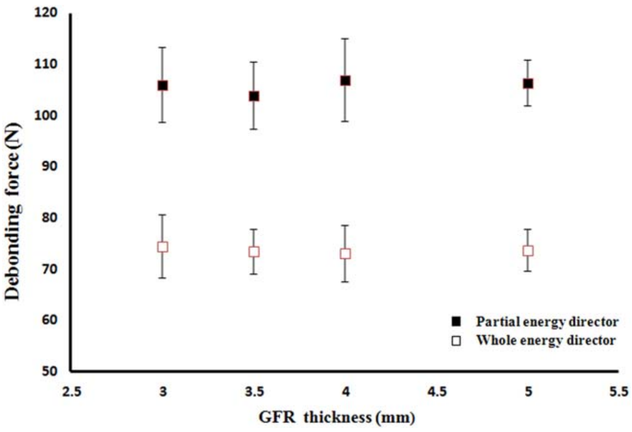

Figure 4. Effect of energy director length and GFR beam thickness on the debonding force (F_d) in SCB test. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Plasma Spraying Coating and Scratch Test

Table III shows the values of the plasma spraying parameters for the trial 10 experiments (two replicates per experiments). Figure 6 displays the visual quality of the coating layer on the HAPEEK substrate and distortion of the HAPEEK substrate in primary trial and optimum experiments. No melting and even distortion were observed in the experiment no. 6.

It was indicated that the surface speed, powder feed, current, primary gas flow, and system voltage have been crucial to achieve the optimum results for this experiment. Further plasma spraying experiment (three replicates) was performed using the experiment no. 6 values, whereas the plasma gun was passing 20 times over the substrate to get the 80 μm of the coating thickness. The drop at the penetration depth of 80 μm (Figure 7) showed that the indenter has reached to the harder layer (HAPEEK substrate) rather than the coating layer, which indicated the coating layer thickness. At the normal force of 30 ± 1.8 N, the HA coating layer was peeled and the substrate was observed. The friction force at this point was attained 20 ± 2.1 N, and consequently, the approximate friction coefficient of 0.67 achieved for scratching of the coating layer.

DISCUSSION

In this study, the new composition was developed on the basis of using three layers of non-degradable bioinert (CFRPEEK), non-degradable bioactive (HAPEEK), and degradable bioactive (HA) materials. Because of the much higher Young's modulus of metal acetabular cups (100–230 GPa) compared to the cortical bone (15–20 GPa), the transferred load to the adjacent bones is significantly decreased compared to the load transferring between the acetabulum–femur bones in a healthy hip joint. According to Wolf's law,[26] the bone becomes weaker to counteract the effect of stress shielding which leads to loosening of the implant. In proposed multi-layer composite, the compatible Young's modulus of the CFRPEEK layer ($E = 15$ GPa)[5] and the cortical bone modulus ($E = 16.4$ GPa)[5] could allow the appropriate stress transferring between the acetabulum and

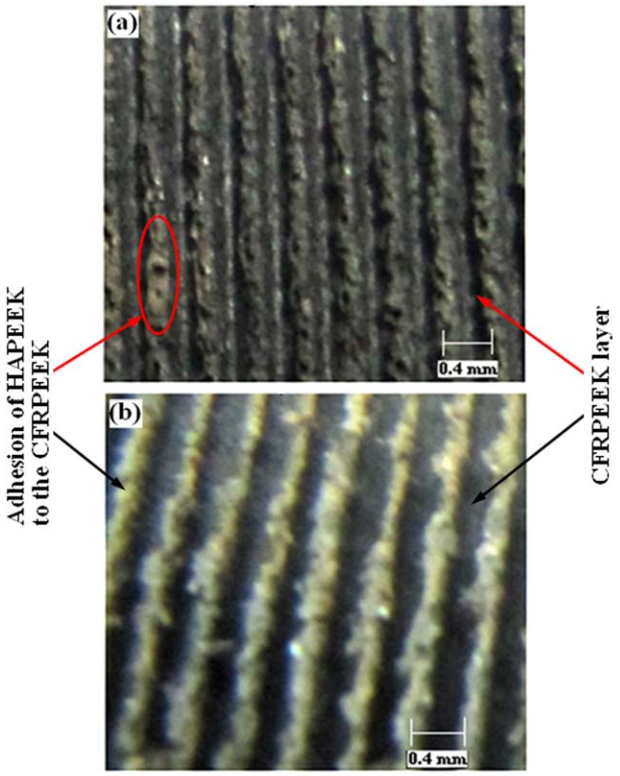

Figure 5. Adhesion of HAPEEK to the CFRPEEK component at the interface after ultrasonic welding; (a) in primary trial, (b) in optimum experiment. The remained HAPEEK material on the CFRPEEK after debonding shows the strong welding between CFRPEEK and HAPEEK components for optimum experiment. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

femur bones while minimizing the stress shielding between the implant and bones. However, the finite element analysis and related experimental testing are essential to investigate the stress shielding mechanism for the proposed composition in further researches.

In the primary trial experiments, it was indicated that higher percentage of HA allowed the better fusion of HA coating on the HAPEEK substrate and higher percentage of PEEK provided better welding of the CFRPEEK and HAPEEK components. Conversely, it was seen that the extruding and injecting of the HAPEEK granules with HA percentage of 30 and over were not effectively performed. Therefore, the 20% HAPEEK was used in this study to achieve the strong welding between HAPEEK and CFRPEEK components while enhancing the fusion of the HA coating to the HAPEEK polymer composite. However, further experiments and tests are under investigation to quantify the effect of HA percentage (in HAPEEK composite) on coating and welding processes.

As the 70% of the CFRPEEK and 80% of the HAPEEK composites were filled with PEEK material, an appropriate joining PEEK layer was formed at the interface. Therefore, the shear modulus of PEEK material was used to calculate the optimum shear strain energy [eq. (3)] in the lap shear test.

In this study, SCB test was used to evaluate the length and direction of the energy directors in ultrasonic welding process. In trial experiments, it was seen that the appropriate welding strength was achieved once the direction of the energy director was orthogonal to the direction of the horn vibration. The density of ultrasonic energy was enhanced at the partial energy directors compared to the whole energy directors, and thus, the debonding force was increased by decreasing the energy director length. The GFR resin beams were required to induce the bending of the CFRPEEK layer while the HAPEEK layer remained fixed.

In the lap shear test experiments, the maximum power of the ultrasonic welder machine and horn material were influenced to achieve the welding time, pressure, and cooling time ranges. The aluminum-made horn was used in this study, whereas the titanium-made horn could convert the ultrasonic energy to the heat energy at a lower welding time to obtain adequate welding strength. In this way, the trial experiments were performed to achieve the optimum conditions based on the machine

Table III. Parameter Values of Plasma Spraying Coating Experiments

	Parameter							
Sample no.	Surface speed (mm/s)	Powder feed (g/min)	Current (A)	Primary gas (Ar) flow (psi)	Secondary gas (Ar) flow (psi)	Carrier gas (Ar) flow (psi)	Gun distance to substrate (in.)	System voltage (V)
1	900	2	500	130	200	50	4	30
2	1100	2.3	900	130	200	50	4	52
3	1100	2.1	600	130	200	50	4	52
4	1300	2.2	500	130	200	50	4	52
5	1200	2.18	500	140	200	50	4	52
6[a]	1200	2.18	500	120	200	50	4	52
7	1200	2.18	500	120	210	50	4	52
8	1200	2.18	500	120	190	50	4	52
9	1200	2.18	500	120	200	35	4	52
10	1200	2.18	500	120	200	50	4	52

[a] Optimum experiment.

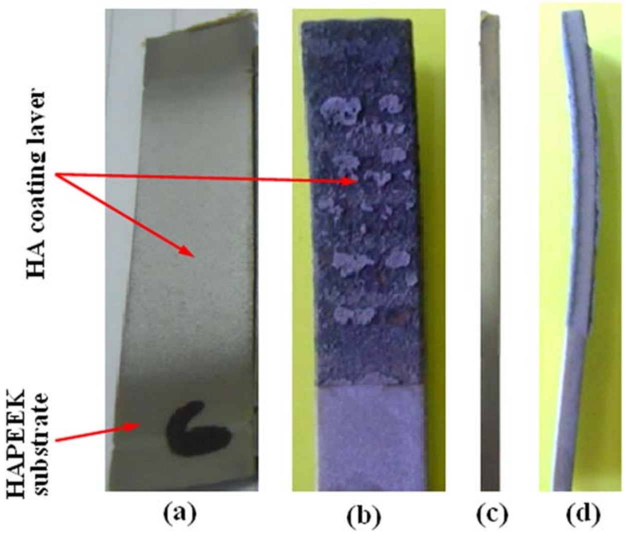

Figure 6. The visual quality of the plasma spraying coating of HA on HAPEEK substrate; (a) optimum experiment, (b) primary trial. The distortion of the HAPEEK substrate due to the thermal effect of plasma spraying; (c) optimum experiment, (d) primary trial. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

maximum power and aluminum-made horn. The welding time and pressure had significant effect compared to the cooling time which was also shown by Liu et al.[13] However, the cooling time was kept in the maximum value of obtained range (3 s) in the experiments. Further nine experiments (Table II) were revealed the significant effect of the welding time on the welding strength compared to the pressure. Physiologically, the acetabular cup prosthesis is under compression loading condition and able to support the welding bonding of the CFRPEEK/HAPEEK interface. The strength of tensile shear force at the optimum condition (1392 ± 35 N) was achieved much higher than the effect of physiological shear force which would be a small portion of the body weight load (800 N in average). The optimum welding time value was achieved in the median value. At below the optimum welding time, the energy was not enough to make strong welding and at over than that, the material was degraded at the interface. The over welding time generated more vibration energy which stimulated the degradation of the welded material at the interface. The degradation was occurred in HAPEEK component as the HA increased the brittleness of the composite.

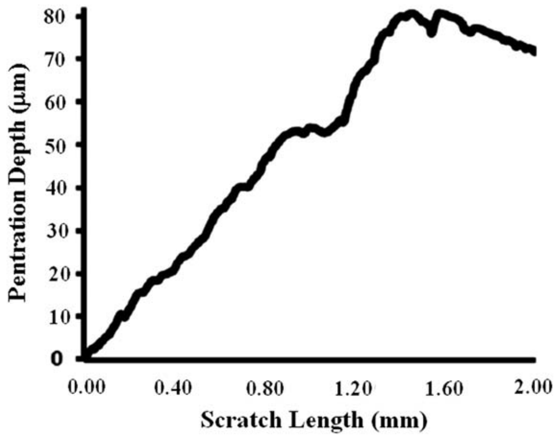

Figure 7. Penetration depth versus scratch length graph. At the penetration depth of 80 mm, the HAPEEK substrate is appeared and the scratch test indenter could not penetrate further.

The adhesion of the coating layer has been an important factor to evaluate the stability of the implant *in vivo* conditions. Duan et al.[27] have reported that the bonding connection of the HA and the cortical bone was stronger than that of the HA and the bioinert material (titanium alloy substrate—Ti6Al4V). This could weaken the bonding of the HA coating layer with the bioinert substrate. Therefore, in this study, the coating of the HA on the non-degradable bioactive substrate (HAPEEK) was contributed to enhance the strength bonding of the coating layer on the substrate. Duan et al.[27] have also reported that the normal force of 11.2 N was necessary for scratching the HA coated layer on the Ti6Al4V substrate. Therefore, the achieved normal force of 30 ± 1.8 N and the friction coefficient of 0.67 could certainly reveal the strong adhesion of the HA coating layer on the HAPEEK substrate using the plasma spraying coating process. Plasma spraying coating process was effectively used to show the possibility of using this method for coating of HA on the HAPEEK substrate. Further experiments in conjunction with biocompatibility tests are under investigation to evaluate the osteointegration of the proposed multi-layer composition.

CONCLUSION

A multi-layer polymer composite was processed in this investigation based on the biomaterials that could enhance the wear resistance and osteointegration of the acetabular cup prosthesis. The relevant manufacturing processes were successfully used and validated by the SCB, lap shear, and scratch tests. The SCB and lap shear tests conducted in this study showed the appropriate bonding strength of ultrasonically welded CFRPEEK/HAPEEK composite. SCB test showed the stronger welding of CFRPEEK/HAPEEK for orthogonal direction of energy directors and horn vibration. This test also indicated that the lower length of energy directors at the interface could increase the bonding strength. Lap shear test results showed that by setting the welding time and pressure at the 3.5 s and 0.8 MPa, respectively, the overlap joint could withstand a tensile shear force of 1392 ± 35 N. The welding time had more effect on the strength of welding compared to the pressure. The suitability of using plasma spraying coating process to coat the HA on the HAPEEK substrate was successfully obtained and the strong adhesion of the coating layer was revealed by the scratch test. The outcomes of this study can be used to investigate the effectiveness of the acetabular cup produced from the proposed multi-layer composite.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the funding from the Ministry of Science, Technology, and Innovation (MOSTI) Malaysia, under Science Fund Grant Scheme (Vote No. 79375).

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