Investigation on the effects of poly-ether-ether ketone (PEEK) veneer thickness on the reciprocating friction and wear behavior of PEEK/Ti6Al4V alloy substrates in artificial saliva

M. Sampaio^a, M. Buciumeanu^b, E. Askari^a, P. Flores^a, J. C. M. Souza^{a,c*}, J. R. Gomes^a, F. S. Silva^a, B. Henriques^a

^a*Center for Microelectromechanical Systems (CMEMS-UMinho), University of Minho, Azurém, 4800-058 Guimarães, Portugal*

^b*Cross-Border Faculty of Humanities, Economics and Engineering, “Dunărea de Jos” University of Galați, Domnească 47, 800008 Galați, Romania*

^c*School of Dentistry, Post-Graduate Program in Dentistry (PPGO), Federal University of Santa Catarina (UFSC), Campus Trindade, Florianópolis, SC 88040-900, Brazil*

*Corresponding author at:

Júlio C. M. Souza

email: jsouza@dem.uminho.pt

Center for Microelectromechanical Systems (CMEMS-UMinho)

University of Minho

Azurém, 4800-058

Guimarães, Portugal

Tel.: +351911174475

Fax.: +351253516007

Abstract

Poly-ether-ether-ketone (PEEK) veneers are attractive for biomedical contacting parts. In the present study, PEEK veneers with thicknesses between 0.1 and 2 mm were synthesized on Ti6Al4V substrates by hot pressing. The influence of PEEK thicknesses on the friction and wear behavior of veneering PEEK to Ti6Al4V structures was studied under reciprocating sliding conditions against an alumina counterbody immersed in artificial saliva at 37 °C. The results revealed that the coefficient of friction and the wear rate increased with decreasing the PEEK thickness. Numerical simulations were carried out to evaluate the influence of PEEK thickness on the contact stress. It was revealed that the increase of both coefficient of friction and wear rate are correlated with increased contact stress level on PEEK veneer. Such factors are determinant on the long term success of veneering biomedical PEEK to Ti6Al4V for oral applications.

Keywords: biotribology; Ti6Al4V; PEEK; sliding wear; thickness; FEM model.

1. Introduction

Nowadays, titanium and its alloys are still largely used to produce structures for biomedical applications, such as dental and orthopedic implants or prostheses. Biological aspects of titanium linked to high corrosion resistance and biocompatibility, as well as mechanical properties (e.g. Young's modulus and tensile strength) are proper for biomedical applications [1–6]. Nevertheless, several issues on failures or negative effects on the long term performance of titanium-based structures such as: wear on contacting surfaces, corrosion in specific

environments (e.g. oral cavity containing fluorides or acid lactic), peri-implant inflammation and excessive stress on bone due to a mismatch in Young modulus between bone and implant [1-6].

Several surface treatments have been performed on titanium and its alloys to decrease corrosion and wear, and consequently to increase the biocompatibility and long term performance of biomedical structures. It involves mechanical treatment, thermal spraying, ion implantation, chemical and electrochemical [7–17]. For instance, the deposition of thin films by PVD and plasma nitriding on Ti based alloys significantly improved the wear behavior of Ti alloys, although the long term performance of these coatings strongly depends on the loading level [13, 15]. It has been concluded that the TiN PVD film had a substantial reduction in mass loss under lower normal loads (10 and 20 N), although a significant mass loss occurred at higher normal load (40 N). On plasma nitrited samples, the mass loss was at about 35% lower than that recorded on uncoated and PVD-coated samples. Garbacz et al. [8] reported the synthesis of a hybrid surface treatment whereas Ti6Al4V alloy was pre-coated with aluminum and then subjected to a glow discharge treatment. That produced a multimaterial intermetallic TiAl-based layer containing Al₂O₃ on the outer surface resulting in a substantially increase in hardness and wear resistance of the Ti6Al4V structure. Yildiz et al. [11] compared three different surface treatments in order to improve the tribocorrosion behavior on Ti6Al4V: plasma nitriding (2-3 µm thickness); TiAlN thin film deposition by magnetron sputtering (3-5 µm thickness) and Al₂O₃ coating by plasma spray method (100 µm thickness). Such three surface treatments improved the wear resistance of Ti6Al4V alloy with the best results obtained for Al₂O₃ coating using plasma spray method.

In dentistry, a veneer is a layer of material placed over a tooth or prosthetic infrastructure, either to improve the aesthetics or else to protect prosthetic structures against wear and corrosion in the

mouth. Recent advances have been reported on the use of soft veneers based on thermoplastic-based polymers such as polytetrafluoroethylene (PTFE), biodegradable poly (D,L-Lactide) PDLLA and poly-ether-ether-ketone [2,4,16–18]. It has been already proved that PEEK and its composites have higher wear and chemical resistance associated with a high biocompatibility when compared to titanium or stainless steel [4,19–25]. Davim et al. [26] studied the friction and wear behavior of PEEK and PEEK reinforced with glass (GF-PEEK) or carbon fibers (CF-PEEK) against a stainless steel counter body on dry sliding. Relatively to friction, it was concluded that PEEK reinforced with glass fiber revealed a higher coefficient of friction (COF) than that recorded on PEEK or CF-PEEK. Alternatively, PEEK showed a less wear resistance, comparatively to those composites. Also, Sumer et al. [20] showed a comparative study between PEEK and (GF-PEEK) against a stainless steel counterbody under dry or water lubricated sliding conditions. GF-PEEK revealed lower coefficient of friction than that recorded on PEEK. Zhang et al. [23] concluded that PEEK veneers exhibit lower coefficient of friction than that on PEEK/SiC composite veneer. However, PEEK/SiC showed a higher wear rate than that on PEEK at high sliding test speed. Wang et al. [14] reported the effects of PEEK coating thickness ranging from 15 up to 35 μm on the alumina or stainless steel substrates by finite element modeling domain (FEM). On a normal static or dynamic loading, the coating material was always on the plastic domain. PEEK-coated alumina samples showed a lower coefficient of friction (COF) than those on stainless steel samples. PEEK-coated stainless steel revealed a higher wear depth than that on PEEK-coated alumina once the stainless steel is a ductile substrate, causing a larger contact area. Nevertheless, PEEK has been used to coat different ductile or stiff materials including titanium-based materials. In fact, the effects of PEEK veneer

thickness combined with the mechanical properties of the substrate should be clarified in order to select the proper thickness to different biomedical applications.

The aim of the present study was to investigate the influence of PEEK thickness on the friction and wear behavior of veneering biomedical PEEK to Ti6Al4V substrate under reciprocating sliding in the presence of artificial saliva mimicking dental applications. Also, some numerical simulations were carried out to correlate the coefficient of friction and wear rate with contact stress level on PEEK veneer on changes in veneer thickness.

2. Materials and methods

2.1. Preparation of the samples

A poly-ether-ether-ketone (PEEK OPTIMA 450, Victrex, England) was selected as polymer veneer and Ti6Al4V (VSMPO TIRUS, US, ASTM B 348, Grade V) was selected substrate. Ti6Al4V surfaces were subjected to grit-blasting procedure, using 250 μm alumina particles. The grit-blasting was carried out at a constant pressure of 5 bars, at a distance of 80 mm from the blasting nozzle and with an impact angle of 90° for 10 s [4]. After the grit-blasting on the Ti6Al4V surfaces, the polymeric veneer on the Ti6Al4V substrate was processed by hot-pressing deposition. The PEEK deposition on Ti6Al4V samples was performed inside a graphite cylindrical die. The Ti6Al4V sample was placed in the mold with its upper surface corresponding to the grit-blasted surface. Then, an adequate volume of PEEK particles was introduced in the mold. PEEK was heated up to 380 °C with a heating rate of 80 °C/min; (3) then, the temperature was decreased down to 300 °C where a pressure of 25 MPa was applied and maintained for 4 s. After, the temperature of the system cooled down to room temperature under vacuum [4].

Different PEEK veneer thicknesses (0.1;0.2;0.3;0.6;1 or 2 mm) were deposited on the Ti6Al4V substrate (8 mm diameter and 4 mm thick) as seen in Figure 1.

Figure 1 – Different thicknesses of PEEK coatings on Ti6Al4V: a) 0.1 mm and b) 2 mm.

2.2. Wear tests

A reciprocating ball-on-plate tribometer (Bruker-UMT-2, USA) was used to perform the wear tests. An alumina ball with 10 mm diameter (Al_2O_3 , Goodfellow, USA) was used as a counterbody. An alumina counterbody was used in this study due to its high chemical resistance and therefore it was previously used as a model to validate the friction and wear behavior of titanium and PEEK [2-4]. The reciprocating sliding tests were carried out in 30 ml Fusayama's artificial saliva at 37 °C, mimicking the temperature of the oral cavity [27]. The tests were performed on 50 N normal load, at a sliding frequency of 1 Hz, and a linear displacement amplitude of 3 mm, during 30 min. Also, several dry reciprocating tests were carried out for 2 mm thickness only to see the influence of artificial saliva on the COF mean values. Five tests were carried out for each set of condition.

After testing, the specific wear rate was estimated by measuring the lateral width of the wear scars and using empirical mathematical equations assuming that the wear scars are formed by perfect ball geometry. More details regarding the wear tests and the estimation of the specific wear rate are given elsewhere [4]. Samples were inspected by Field Emission Guns Scanning

electron microscopy (FEGSEM, FEI Nova 200, USA) to identify the dominant wear mechanisms.

2.3. Numerical simulations

According to the Archard's wear law, the amount of material loss is associated with the contact stress, sliding distance and wear coefficient [29]. In the present study, the sliding distance is the same for all tested samples with different **veneer** thicknesses. Moreover, the wear coefficient is assumed similar for all cases, as the **veneer** material is the same, ignoring probable discrepancies due to the manufacturing process. Therefore, a better insight into the effect of **veneer** thickness on wear rates can be gained by considering variations of contact stress as a function of **veneer** thickness. A numerical analysis was performed to evaluate the influence of **veneer** thickness on the contact stress by using the commercial software ANSYS workbench (Release 16.2). Contact elements were sized as small as enough to guarantee the convergence and accuracy of results, as shown in Figure 3.

Figure 3 – A representation of the finite element model of indenter pressed into coated substrate, showing the refined meshes in the region around contact zone.

A quarter of the whole model was set up due to its symmetry with respect to two perpendicular planes. 98996 elements and 387826 nodes were used to discretize the model with refined meshes in the contact region.

Material properties of the components were assigned and large deformations of the **veneering** PEEK were allowed concerning its plastic behavior particularly for thinner thicknesses due to low yield stress, 100 MPa, and high contact stresses. The plasticity was taken into account using a bilinear isotropic hardening model available within the software environment. Friction between contacting surfaces was taken into consideration, although this numerical analysis did not intend to simulate the motion of articulating bodies. **It is worth noting that when bodies slide against each other, tangential friction forces develop, influencing corresponding contact area and normal contact stresses [30].** The substrate was **composed of Ti6Al4V** alloy with Young's modulus of 115 GPa and the alumina ball (5 mm radius) with Young' modulus at 300 GPa was pressed onto the **veneer** surface on 50 N normal load. Furthermore, the Young' modulus of the PEEK was at 3 GPa. The contact area was smaller than the whole model size, hence the effect of the model size on the numerical outcomes can be neglected. The boundary conditions were specified considering the model matched the real indentation process. The bottom surface of the specimen was compressed along its normal direction and the normal load was gradually applied for 1 s.

3. Results and discussion

3.1. Friction and wear measurements

The evolution curves of the coefficient of friction (COF) for 0.1 or 2 mm PEEK thickness during sliding against Al_2O_3 ball in artificial saliva solution **are** shown in Figure 4.

Figure 4 – Evolution of the COF during sliding on 0.1 or 2 mm PEEK thickness against Al_2O_3 ball in artificial saliva solution at 37 °C

On both cases, the COF exhibited a peak value which is attained after few seconds of running, followed by a running-in period of ~500 s before entering in a steady-state regime. Although the similarity in the evolution of COF curves on the contacts involving the thinner (0.1 mm) and the thicker (2 mm) PEEK samples, lower values of COF were measured on 2 mm PEEK veneer. Therefore, the thickness of PEEK sample did not affect the pattern of COF curves along the sliding period, but it had a significant effect on the friction values corresponding to a specific sliding distance.

Mean values of steady-state COF corresponding to all groups of PEEK veneer on Ti6Al4V substrate against Al_2O_3 are shown in Figure 5. A decrease in COF mean values was evidenced with the increase of PEEK thickness up to 0.6 mm. The highest COF mean value ($\mu = 0.14$) was obtained on 0.1 mm PEEK thickness, while the lowest one ($\mu = 0.09$) was recorded for 0.6, 1 and 2 mm PEEK thickness. On dry conditions, the mean COF values recorded on PEEK was around 0.27. The lower COF value (0.9-0.10) recorded on PEEK in the presence of saliva in this study

was previously reported in a previous article [4]. In fact, the artificial saliva solution performed a lubricant effect that decreased the COF on PEEK [31].

Figure 5 – Mean values and standard deviation on steady-state COF for different thicknesses of veneering PEEK on Ti6Al4V substrate against Al_2O_3 in presence of artificial saliva solution at 37 °C ($F_N=50$ N, 3 mm displacement amplitude, 1 Hz, 30 min of sliding).

To the authors knowledge, previous studies on the wear behavior of PEEK veneers reported in literature are scarce [4,14,22], although there are several studies on the wear behavior of PEEK or PEEK composite structures [32–35]. The COF mean values reported in literature for sliding contacts involving PEEK veneer ranged from 0.07 up to 0.7, under different environments (dry or lubricated conditions). Wang et al. [14], investigated the tribological behavior of a PEEK veneer on alumina and stainless steel substrates considering two different PEEK thicknesses against an alumina ball. For PEEK coating on alumina substrate, the COF mean values increased with the decrease of PEEK thickness, which is in accordance with the results obtained in the present study. It has been shown in literature that PEEK/alumina samples with 35 μm thickness of PEEK coating revealed a COF mean value around 0.20, while COF increased to 0.45 on 15 μm thickness of PEEK coating. The COF mean values were much higher (around 0.60) for both thicknesses of PEEK (15 and 35 μm) when the alumina substrate was replaced by stainless steel.

Also, COF mean values increased during the running-in period due to the plastic deformation of the stainless steel substrate. It is worth to mention that both the static and the dynamic contact stresses were in the plastic regime under the testing conditions. In a previous work [4], the authors of the present study reported COF value at ~ 0.07 under tribocorrosion tests with reciprocating sliding contact of 2 mm thickness of PEEK veneering on Ti6Al4V against Al_2O_3 . Concerning the lubricant effect of human saliva in the oral cavity, it is worth mentioning that the tribocorrosion tests were performed in artificial saliva lubricating conditions. Therefore, friction values of tribological systems involving sliding contacts with PEEK can vary significantly, depending on several factors, like the nature of counterface, load, sliding speed and environment. The values of specific wear rate of PEEK veneer on Ti6Al4V against alumina under reciprocating sliding conditions for different thicknesses of PEEK are shown in Figure 6.

Figure 6 – Specific wear rate of different PEEK veneer thicknesses on Ti6Al4V substrate against alumina in presence of artificial saliva at 37 °C ($F_N=50$ N, 3 mm displacement amplitude, 1 Hz, 30 min of sliding)

In fact, the wear performance of PEEK was influenced by the thickness values of the polymeric veneer material on Ti6Al4V substrate. Corroborating the COF results, the specific wear rate decreased with the increase of PEEK thickness. The highest specific wear rate ($1.47 \times 10^{-4} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$) was obtained for 0.1 mm PEEK thickness, while the lowest one ($3.62 \times 10^{-5} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$) was recorded on the thicker PEEK veneer (2 mm thickness). The highest specific wear rate ($1.47 \times 10^{-4} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$) obtained for the thinner PEEK veneer, which also corresponded to the highest measured COF value (0.14), can be attributed to the increase of the contact stress between the polymeric surface and the Al_2O_3 counterface, as shown in section 3.3. A previous study [14] also reported that the coating thickness at high contact stresses and plastic domain can have a significant influence on the friction and wear behavior of PEEK used as a veneer.

3.2. Wear scar morphology

The morphologic aspects of worn surfaces for PEEK veneer thicknesses after sliding against alumina are shown in Figure 7.

It can be seen that the presence of abrasion grooves aligned parallel to the sliding direction was a characteristic morphological aspect for all PEEK thicknesses considered in this work, resulting from the interaction with the harder ceramic counterbody by two-body abrasion wear. In a previous study involving the sliding contact between PEEK and alumina in the presence of artificial saliva [4], it was found that no transfer of polymer occurred to the ceramic counterface. The same behavior was also confirmed in the present study. Therefore, the wear behavior of PEEK against alumina in presence of artificial saliva was mainly attributed to smooth abrasion of the polymeric surface, resulting in low friction values (0.09-0.14), and relatively low wear rate values, from $3.62 \times 10^{-5} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$ to $1.47 \times 10^{-4} \text{ mm}^3\text{N}^{-1}\text{m}^{-1}$, with the highest friction and wear rate values being measured for lower PEEK thicknesses.

Figure 7 - FEG-SEM images of the central area of PEEK wear tracks after reciprocating sliding against alumina ball in artificial saliva for different PEEK thicknesses: (A,B) 0.1 mm; (C,D) 0.2 mm and (E,F) 2 mm. Magnification at x500 (A,C,E) and magnification at x2000 (B,D,F).

In fact, PEEK worn surface appearance tends to be smoother for thicker samples (Figures 7C-F), where abrasion grooves alternate with large smooth regions. Some larger scratchers can be noticed on SEM images in Figures 7E and F. That revealed a loosening of material from

localized areas of the sample surface. Accordingly to those observations, the mean values of steady-state COF, as well as the specific wear rates, were lower than that recorded for thinner PEEK sample (Figures 4 and 5). Considering the detail view of the thinner PEEK worn surface (Figure 7B), morphological features were quite different when compared to the worn surface of thicker samples (Figure 7D and F).

Considering the tribological and microscopic results on veneering biomedical PEEK to Ti6Al4V alloy obtained in this study, it can be stated that the decreasing the PEEK thickness under a critical value around 0.2 mm must be avoided in order preserve the friction and wear properties for tribological application of the veneering PEEK for conditions mimicking the oral environment.

3.3. Contact stress evaluation

Using the numerical analysis, the evolution of the contact stresses and the Von-Mises yield criterion, as a function of the veneering thickness of PEEK is revealed as shown in Figure 8. It can be seen in Figure 8 (a) that contact stresses are influenced by the veneer thickness, and that they increased for thinner veneer. A steeper increase in stress magnitude is registered for thicknesses below 0.5 mm which can explain the worse friction and wear performance, that is, higher COF and wear rates of specimens with 0.1 mm PEEK veneer. Considering the Von-Mises yield criterion, plastic deformation takes place on the veneer when its thickness is at 0.1 mm (Figure 8b), that can represent another factor contributing to the higher wear rate seen in such a case. Von-Mises stress distributions for veneer thicknesses of 0.1 and 2 mm are shown in Figures

9 and 10, respectively. The contact stress distribution for veneers with 0.1 mm and 2 mm thickness are also illustrated in Figure 11 and Figure 12, respectively.

The analysis of these figures reveals that the highest stresses on thicker veneers occur near the surface and stay within the veneer material, not reaching the substrate and thus protecting it from being damaged. On the other hand, besides the higher stresses developed in thinner veneers, these stresses propagate also to the substrate, creating a stress discontinuity at the PEEK/Ti6Al4V interface that can cause crack initiation/propagation and/or veneer delamination. These findings are in agreement with those reported elsewhere [14,23].

Figure 8 – Effects of the PEEK thickness on (a) maximum contact stresses and (b) maximum Von-Misses stress in the veneer zone.

Figure 9 – Von-Mises stresses onto the **veneer**, substrate and indenter, with the PEEK thickness of 0.1 mm.

Figure 10 – Von-Mises stresses onto the **veneer**, substrate and indenter, with the PEEK thickness of 2 mm.

Figure 11 – Contact stress distribution onto the **veneer**, substrate and indenter, with the PEEK thickness of 0.1 mm.

Figure 12 – Contact stress distribution onto the **veneer**, substrate and indenter, with the PEEK thickness of 2 mm.

A limitation of the finite method analysis conducted in this study should be referred. The FEM model used did not intend to simulate the stresses developed at the specimens during the sliding

test. It is a simple approach that rather analyses the contact stresses occurring at the initial stage of the sliding test ($t=0$), when the alumina ball is put in contact with the PEEK surface when no sliding movement have taken place. Nevertheless, it provided useful insights on the interaction between the contacting bodies, which helped in the clarification of the phenomena underlying the wear results. It was possible to correlate the higher contact stresses occurring in the thinner PEEK veneers to the higher COF and wear rates experimentally measured.

The wear tests performed in this study involved sliding motion between the alumina ball and the PEEK surface, with the presence of friction. The coefficient of friction affects the resultant stresses in sliding contact, which maximum value always occurs at the trailing edge [36]. Friction also influences the onset of yield in sliding contact. It has been shown by Johnson [36], using both the Tresca and von Mises yield criteria, that low values of the coefficient of friction ($COF < 0.25$ by Tresca and $COF < 0.30$ by von Mises) favors a first yield of the material beneath the contact surface, while for larger values of COF the yield should first occur at the contact surface. Based on this, and considering the low COF measured in this study ($0.09 < COF < 0.14$), one may say that yielding in the PEEK veneer under sliding contact conditions, should most probably occur beneath the contact surface, where higher stresses are developed. The presence of higher stresses beneath the surface and not at the contact surface (as would occur for higher COF) increases the risk of delamination especially in thinner veneers, as previously referred. According to the decrease of the PEEK veneer thickness, an increase of the contact stress was noted, even in the elastic regime. The contact stress (static) was influenced by the elastic properties (Young's modulus and Poisson coefficient) of the veneer, substrate, and counterbody. Also, the geometry and normal load affected the contact stress on PEEK veneer.

In the present case, the contact stress strongly increases when the PEEK **veneer** thickness on Ti6Al4V decreases below 0.2 mm. In fact, the elastic properties of the substrate become more relevant for thinner **veneers**, even in the elastic domain. These contact stress results are in accordance with the wear results, considering the increase in COF and wear rate values with the decrease of the thickness of the PEEK veneer.

4. Conclusions

In this work, the influence of PEEK thickness on the friction and wear behavior of veneering PEEK to Ti6Al4V structures was studied in reciprocating sliding tests against alumina in presence of artificial saliva at 37°C, mimicking the oral environment. The main conclusions can be drawn as **follows**:

- PEEK thickness significantly influenced the **friction and wear properties**. Coefficient of friction values recorded are ranging from 0.09 up to 0.14 and specific wear rate decreased with the increasing of veneer PEEK thickness to Ti6Al4V substrate.
- The dominant wear mechanism was identified as smooth abrasion of the polymeric surface promoted by the harder ceramic counterface.
- An increase in contact stress between PEEK and alumina counterbody was noted when the PEEK veneer thickness decreased down to 0.2 mm. The elastic properties of the Ti6Al4V substrate affected the thinner PEEK **veneers** that are in accordance with the **friction and wear** results.

Concerning dental applications, the PEEK thickness under a critical value around 0.2 mm must be avoided in order preserve the **friction and wear properties for tribological application** of the veneering PEEK to Ti6Al4V structures.

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Graphical Abstract