Surface modification methodologies for polycrystalline alumina: effects on morphology and frictional coefficients

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Several methodologies of surface modifications were applied to polycrystalline alumina (PCA) samples to study their effects on surface morphology and frictional coefficients. Modified surfaces were first tested in a specially designed frictional apparatus against wires of stainless steel (SS) and beta-titanium (β -Ti) alloys and then evaluated by scanning electron microscopy. Techniques included ion implantation of chromium ions (Cr⁺), ion beam assisted deposition of diamond-like carbon (DLC), coatings of gamma-irradiated polymers (PEO), and electroless nickel plating of a composite material of polytetrafluoroethylene dispersed in a nickel phosphorus matrix, Niflor NT[®] (NF). Implanting ions into the bulk material had no effect on surface morphology. Although covering the surface, the DLC coating mimicked the underlying topography. The coatings of PEO and NF obliterated and smoothed over the normally rough and faceted PCA surfaces. When compared to control samples, neither the Cr⁺ or DLC process reduced the friction normally seen against SS and β -Ti wires. When tested in both the dry and wet states, the PEO coated samples retained their traditional levels of frictional resistance. Only the composite material, NF, successfully reduced the friction when compared with controls. Although this composite coating is not recommended for oral use, the results show that simply smoothing over the rough surface is inadequate for friction reduction; the surface must somehow also be made lubricious.

1. Introduction

Because of patient demand for aesthetic 'clear' braces (brackets), polycrystalline alumina (PCA) materials are widely used in orthodontics, in spite of poor mechanical properties. Low fracture toughness and high brittleness [1-3] lead to bracket failure, and their high friction [4-10] can also complicate tooth movement. In the latter case, efforts to reduce this friction could decrease the forces applied clinically and provide a more efficient, reproducible, and biologically compatible system for moving teeth.

In the clinical setting where the bracket must occasionally translate along a wire, frictional resistance occurs as the hard and faceted PCA surface removes wire material all along the floor and walls of the bracket's slot, as well as, stripping wire particles away the sharp slot edge formed by the floor or wall of the slot and the external side of the bracket [10]. In an effort to study methods to reduce this interaction and thus reduce the friction, several methodologies of PCA surface modification were evaluated. They included ion implanting chromium ions (Cr^+) into the outer layer of the PCA material to alter its near-surface physical characteristics. In addition, a hard inert coating of diamond-like carbon (DLC), which can have

a low coefficient of friction [11], was applied onto the PCA surface by ion beam assisted deposition. Since the environment where the bracket and wire interact often contains moisture (saliva), a polymer material that becomes lubricious when wet, poly(ethylene) oxide (PEO), was also applied to PCA surfaces. Finally, a composite material of lubricating polytetrafluoroethylene (PTFE) particles dispersed in a hard nickel phosphorus matrix was applied by electroless nickel plating, Niflor NT® (NF). In selecting these surface modification techniques, key considerations were specie adherence, wear resistance, and thickness. Samples treated by each of these modification techniques were paired with either stainless steel (SS) or beta-titanium $(\beta$ -Ti) wires, which have been shown to cause the lowest and highest frictional resistance, respectively, against these PCA materials [10]. These pairs, or couples, were tested in a specially designed apparatus to determine the static and kinetic coefficients of friction. The surface morphology of the treated samples was studied by scanning electron microscopy (SEM) to demonstrate any surface changes as a result of surface modification, as well as, to reveal how each methodology succeeded or failed in frictional reduction.

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Studying these methods and their effects on both morphology and frictional coefficients provided insight into what was the overriding cause of the high friction associated with these PCA materials. Determining the importance of layer thickness, surface coverage, and lubricity will help to establish future

2. Materials and methods

The PCA samples were brackets normally used for orthodontic treatment. Although several brands of PCA brackets are commercially available, earlier efforts have demonstrated their similar frictional characteristics [10]. Each bracket was a small block of PCA that had a slot machined into its face, which was 0.022 inch wide and 0.028 inch deep (Fig. 1). The slot was angled so that its floor was at a 7° bias (torque) to the bracket base (Table I). The brackets were paired with a SS or β -Ti wire for subsequent friction testing. These rectangular wires, 0.021 inch×0.025 inch in dimension, have been shown to produce the least and greatest friction, respectively, when tested against PCA materials [10]. Prior to friction testing, the surfaces of the as-received PCA samples were modified by several methodologies: (1) a metallic element was placed into the near-surface by ion implantation (Cr⁺); (2) a hard and inert diamond-like carbon coating was deposited on the surface (DLC); (3) a soft polymeric coating was placed on the surface that became lubricious when wet (PEO); and (4) a composite coating of soft lubricious particles was dispersed into a hard matrix and deposited on the surface (NF).

Ion implantation of chromium ions (Cr⁺) into the floor and walls of the slot was done at 77 K using energies of 125 keV and doses of 3×10^{16} ions cm⁻³ and 2.5×10^{15} ions cm⁻³, respectively.

The ion beam assisted deposition of amorphous DLC was done by a dual ion beam process. Methane and argon gases were introduced into an ion source, and a plasma was generated. The resulting ions were removed and accelerated toward the sample target by electrically charged grids. The DLC layer was formed by the dense packing of the carbon and hydrocarbon ions as they impinged on the surface of the PCA target. Ion bombardment was done using an 11 cm Kaufman source (Ion Tech, Boulder, CO) at levels to produce a uniform coating 300 nm thick. Technical details of the process are proprietary.

Ion implantation and DLC coating are both line-ofsight processes. Thus the bracket, which has an angled slot of -7° , was mounted on a 7° biased surface to place the slot floor perpendicular to the ion beam so that the entire floor could be treated. In contrast to line-of-sight processes, the PEO and NF coatings were both done in baths, so mounting on a biased surface was not necessary.

Three different molecular weights of PEO (PEO1*, PEO2[†], PEO3[†]) were dissolved in deionized water to

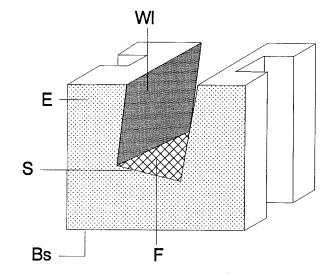


Figure 1 Schematic representation of a PCA ceramic bracket showing the wall (WI), external side (E), slot edge (S), base (Bs), and slot floor (F).

form 1% (w/w) solutions. The brackets were submerged in the PEO baths and placed in a vacuum oven at 80 °C until the water was evaporated and only a thin film of PEO remained. The samples were then placed in a Cs-137 gamma ray source at an ambient temperature of 35 °C and subjected to 50 Mrad of radiation at a dose rate of 0.85 Mrad h^{-1} to produce a dense mat of crosslinked molecules [12].

The NF coating was produced by Anoplate's (Anoplate Corp., Syracuse, NY) standard manner of autocatalytically applying a composite of nickel phosphorus binder and sub-micrometre particles of PTFE. A plating bath composed of sodium hypophosphite and 25% (v/v) PTFE reduced and deposited the nickel with the PTFE dispersed throughout. Because the method was designed for use on metal and metal alloys, sputter coating of the alumina surface with copper was required to enhance surface adhesion. All modification processes are summarized, along with their manufacturers, in Table II.

Once modified, all bracket samples were paired with either a SS or β -Ti wire for subsequent frictional testing in a low-load, low-velocity device [13] (Fig. 2). The wires were held stationary and pressed into the bracket slot by two strands of 0.010 inch SS ligature wire (Item PL 1010 ligature wire, GAC International, Commack, NY) at a constant normal load (N). The brackets, cemented to a 7° biased surface to negate torque effects, were drawn along the wires by the crosshead of the screw-driven testing machine (Instron TTCM, Canton, MA) at a rate of 1 cm min⁻¹. Drawing force (P) versus displacement (δ) tracings (Fig. 3) were generated for each run, and five runs were made for each arch wire-bracket pair using normal loads of 0.2, 0.4, 0.6, 0.8, and 1.0 kg. The static drawing force was measured as the first peak on the $P-\delta$ tracing, and the kinetic drawing force was computed by averaging all subsequent data points. Frictional forces (f) equalled one-half of P, since the wire was contacted on two

^{*} Grade 1 500, Polysciences, Warrington, PA [molecular weight (MW) = 1.5×10^3].

[†] WSRN 750, Union Carbide, New York, NY (MW = 3×10^5).

[‡]WSRN Coag, Union Carbide, New York, NY (MW > 5×10⁶).

TABLE	I	Bracket	and	wire	materials	evaluated

Material	Symbol	Product	Specifications	Supplier		
Bracket						
Polycrystalline alumina	PCA	Quasar	0.022 inch, 0° angulation, -7° torque	Rocky Mountain Ortho., Denver, CO		
Wires						
Stainless steel	SS	Standard rectangular	0.021×0.025 inch	Unitek/3M Corporation, Monrovia, CA		
Beta-titanium	β-Τί	ТМА	0.021×0.025 inch	Ormco Corporation, Glendora, CA		

TABLE II Summary of PCA modification processes

Process	Symbol	Description	Processor
Ion implantation	Cr ⁺	Ion implantation of Cr into the floor and walls at 77 K using energies of 125 keV and doses of 3×10^{16} and 2.5×10^{15} ions cm ⁻³ , respectively	Oak Ridge National Laboratories, Oak Ridge, TN
Diamond-like carbon coating	DLC	Ion beam assisted deposition of DLC in a methane/argon gas atmosphere	Diamonex Inc., Allentown, PA
Polymer coatings		Poly(ethylene)oxide $[OH-(CH_2-CH_2-O)_n-H]$ films subjected to 50 Mrad radiation from a Cs-137 source at a dose rate of 0.85 Mrad h ⁻¹ . Trade names and molecular weights:	Processed in our laboratory
	PEO1	Grade 1500, 1.5 × 10 ³	Polysciences, Warrington, PA
	PEO2	WSRN 750, 3 × 10 ⁵	Union Carbide, New York, NY
	PEO3	WSRN Coag, $> 5 \times 10^6$	Union Carbide, New York, NY
Composite coating	NF	Sub-micrometre polytetrafluoroethylene particles dispersed in a nickel phosphorus matrix, deposited by electroless plating	Anoplate Corp., Syracuse, NY

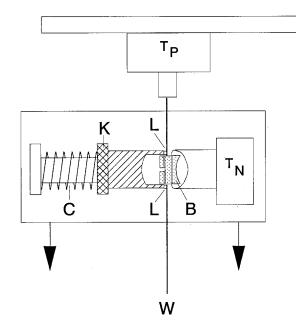


Figure 2 Schematic representation of the friction testing apparatus. The wire (W) is pressed into the bracket (B) slot by two ligature wires (L). The normal force (N), which presses the wire and is recorded by the transducer (T_N) , is adjusted by turning the knob (K) and compressing the coil spring (C). The drawing force transducer (T_P) records the drawing force (P) necessary to move the bracket along the wire.

sides. By plotting f versus N for static and kinetic data, the frictional coefficients (μ_s and μ_k , respectively) equalled the slopes of the plots (Fig. 4). Initially, all tests were performed in prevailing air ('dry 1') at 34 °C.

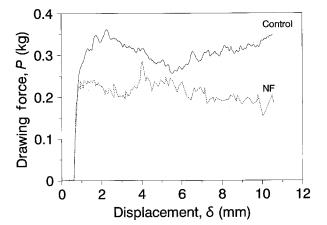


Figure 3 An example of drawing force (P) versus displacement (δ) plots in the 'dry 1' state for PCA/ β -Ti couples in the control and NF coated states at a normal force (N) of 400 g. Note that the P necessary to initiate and maintain movement of the NF coated PCA bracket along the β -Ti wire is less than that of the control couple.

After all 'dry 1' runs were performed, only the PEO samples were tested in human saliva to observe any effects in the 'wet' state. The saliva, collected from a healthy adult male (viscosity = 1.35 centipoise, T= 34 °C, shear rate = 450 s⁻¹, cone angle = 0.8°) was continually pumped onto the bracket/wire interface. Previous work [10] has shown that no breakdown of the saliva results from continuous pumping. Immediately after the wet tests, the saliva pump was disconnected, and the samples were allowed to dry in prevailing air for 5 min. Thereafter, samples were

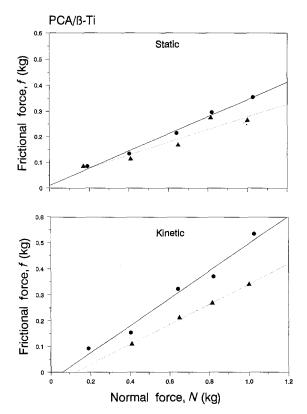


Figure 4 Frictional force (f) versus normal force (N) plots for two PCA/ β -Ti couples in the 'dry 1' state. Note that the NF coated PCA bracket displayed less resistance against the β -Ti wire than the control couple as evidenced by its flatter slope (\bullet control; \blacktriangle NF).

tested in the dry state again ('dry 2') to see if the PEO retained any liquid and maintained its lubricity.

After friction testing, the surface topography of the modified brackets was evaluated using a scanning electron microscope (SEM) (Etec U-1 Autoscan, Haywood, CA). Both the surface changes as a result of the modification technique as well as some indication of the adherence and wear resistance of the new surface material were investigated. An accelerating voltage of 20 kV and condenser current of 2.5 A were used, and the samples required sputter coating with gold-palladium prior to viewing. X-ray elemental analysis (KEVEX, Foster City, CA) was used to confirm the presence of chromium in the ion implanted samples.

3. Results

Although the drawing forces (P) for the modified samples were generally comparable to controls, the drawing forces for the NF samples were reduced (Fig. 3). Regression analysis of the resulting f versus Ndiagrams (Fig. 4, Tables III-V) showed that, for all plots, the correlation coefficients and the statistical probabilities corresponded to r > 0.878 and p < 0.05. respectively. Of the various surface modification techniques, only NF reduced the friction normally seen between the PCA brackets and arch wires tested (Fig. 5, Tables III-V). When compared to untreated samples (Fig. 6), the Cr⁺ samples demonstrated no changes in surface texture (Fig. 7a), although X-ray elemental analysis showed chromium to be present (Fig. 7b). The adherent DLC coating was not thick enough to mask the PCA's surface asperities (Fig. 8a). Consequently, the arch wires were abraded (e.g. the β -Ti debris denoted as 'D' in Fig. 8b), and the friction remained high. Although the PEO coatings were thick enough to smooth over the PCA's surface and did not wear away (Figs 9a, 10a and 11a), β -Ti arch wire debris generated by the hard slot edge was generally evident (contrast Figs 9b and 10b with Fig. 11b), and the lubricating effect of wetting the surface was unable to overcome that interaction (Tables IV and V). The lubricious nature of the NF coating, however, reduced the friction, even though arch wire debris was being generated (e.g. the β -Ti debris of Fig. 12a). Unfortunately, this coating tended to crack and flake off (crack denoted as 'C' in Fig. 12b).

TABLE III Coefficients of friction (μ) of modified PCAs with associated correlation coefficients (r) and statistical probabilities (p)

Couple	Process	Static	_	Kinetic			
		μ _s	r	p	μ	r	р
PCA/SS	Cr ⁺	0.177	0.995	< 0.001	0.218	0.999	< 0.001
PCA/β-Ti	Cr ⁺	0.536	0.964	< 0.01	0.641	0.998	< 0.001
PCA/SS	DLC	0.328	0.908	< 0.05 < 0.02	0.308	0.964	< 0.01
PCA/β-Ti	DLC	0.467	0.954		0.484	0.985	< 0.01
PCA/SS	NF	0.262	0.931	< 0.05 < 0.02	0.092	0.935	< 0.02
PCA/β-Ti	NF	0.267	0.949		0.355	0.995	< 0.001

TABLE IV Coefficients of static (µs) friction of PEO-treated PCAs with associated correlation coefficients (r) and statistical probabilities (p)

Couple	Process	Dry 1			Wet	Dry 2				
		μ _s	r	р	μ _s	r	р	μ _s	r	р
PCA/SS	PEO1	0.118	0.994	< 0.001	0.197	0.943	< 0.02	0.255	0.999	< 0.001
PCA/SS	PEO2	0.282	0.975	< 0.01	0.372	0.999	< 0.001	0.234	0.964	< 0.01
PCA/SS	PEO3	0.202	0.991	< 0.01	0.175	0.990	< 0.01	0.280	0.969	< 0.01
PCA/β-Ti	PEO1	0.515	0.933	< 0.05						1998 Audit 2
PCA/β-Ti	PEO2	0.457	0.997	< 0.001						
ΡСΑ/β-Τί	PEO3	0.491	0.948	< 0.02						

TABLE V Coefficients of kinetic (µk) friction of PEO-treated PCAs with associated correlation coefficients (r) and statistical probabilities (p)

Couple Process	Process	Dry 1	-		Wet			Dry 2		
	μ_k	r	p	μ _k	r	р	μ	r	р	
PCA/SS	PEO1	0.172	0.992	< 0.001	0.246	0.964	< 0.01	0.240	0.990	< 0.01
PCA/SS	PEO2	0.354	0.980	< 0.01	0.146	0.974	< 0.01	0.257	0.949	< 0.02
PCA/SS	PEO3	0.219	0.969	< 0.01	0.186	0.984	< 0.01	0.299	0.975	< 0.01
PCA/β-Ti	PEO1	0.571	0.993	< 0.001						
PCA/β-Ti	PEO2	0.673	0.995	< 0.001						
ΡСΑ/β-Τί	PEO3	0.633	0.997	< 0.001						

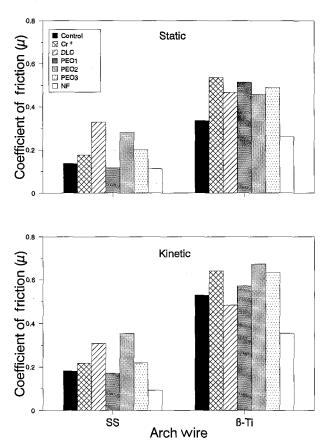
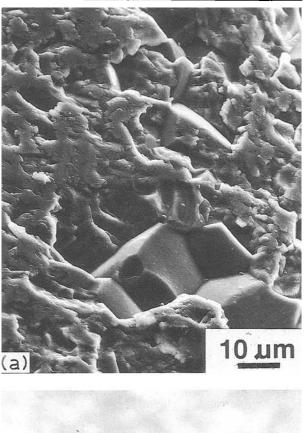


Figure 5 A summary of static and kinetic coefficients of friction in the 'dry 1' state for surface-modified PCA brackets against either SS or β -Ti wires. When compared to controls, only the NF coated PCA brackets generally reduced the friction against either SS or β -Ti wires.

4. Discussion

High friction of alumina brackets is thought to be due to both the hard and faceted surfaces and the sharp slot edges created by the junction of the slot floor and walls with the bracket's external side [10]. By properly covering up the surface, modifying its surface chemistry, or removing the sharp slot edge, the friction should decrease. Preliminary work in this laboratory has shown that bevelling the edge of a PCA sample was not satisfactory because the contact angle between the wire and the slot edge, although reduced, was still present. Consequently, the wire continued to be abraded. Likewise, reducing the friction between PCA and titanium wires required more than simple bevelling because the titanium wire is reactive. Metals like titanium that readily oxidize are very adherent to alumina surfaces because of interactions with the oxygen anion in the alumina lattice [14]. Thus, changing



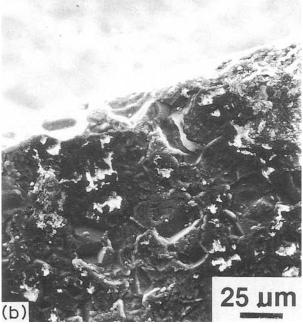
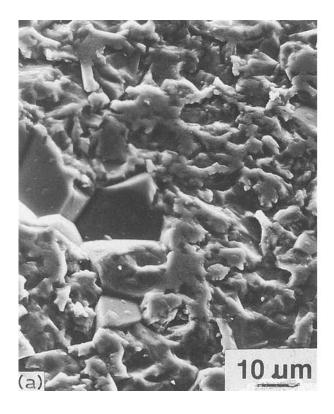


Figure 6 SEM's demonstrating the rough surface texture of the slot (a) and the edge created by the bracket's slot floor and external side (b) in untreated control PCA samples.



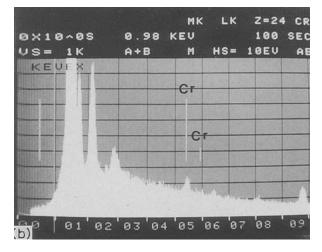
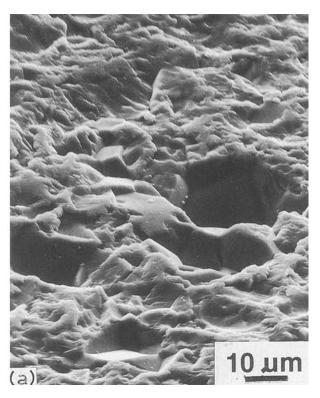


Figure 7 The SEM of the PCA bracket that was ion implanted with Cr^+ ions shows no change in surface topography (a), although X-ray elemental analysis indicates that Cr is present (b).

the surface chemistry of the slot, or placing a nonreactive layer between the slot floor and the wire, would also seem necessary.

Implanting high energy ions into crystal substrates can modify the near-surface crystalline structure by changing its chemistry, although without affecting the properties of the bulk material. When implanting species are ionized and accelerated towards a target sample, the implanting ions collide with surface atoms and displace them from their original lattice positions. These atoms, along with the implanting ions, create more crystal damage as they carom off other lattice atoms, causing a cascade of crystal defects. These detects can alter many of the target material's surface properties, including frictional resistance [15]. Implanting hard chromium ions into the surface of the PCA slot could have rendered the outer layers



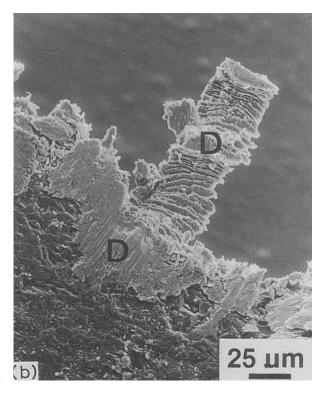
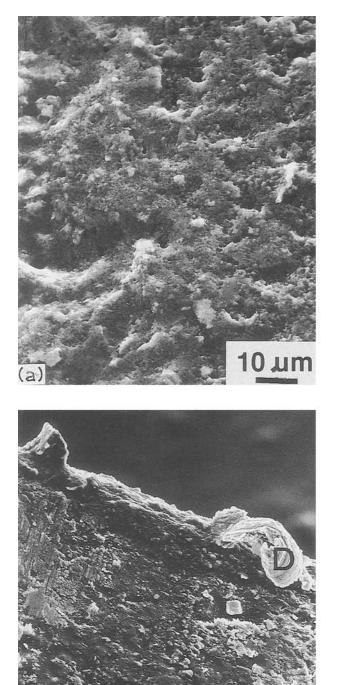


Figure 8 The PCA bracket coated with DLC shows no changes in surface texture of the slot (a), and the slot edge displays large amounts β -Ti arch wire debris (D in (b)).

amorphous, as has been shown for sapphire [16], and soften the normally hard facets. However, this implantation of PCA was not successful, and the faceted surface (Fig. 7a) continued to create high friction (Fig. 5).

Placing a hard non-reactive layer between the wire and slot floor, by DLC coating, was likewise unsuccessful (Fig. 5) because the facets were still present (Fig. 8a) and readily able to strip out pieces of the



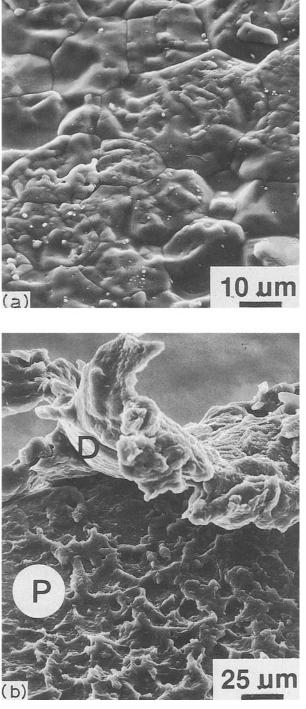
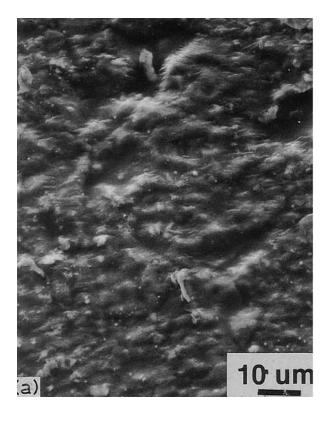
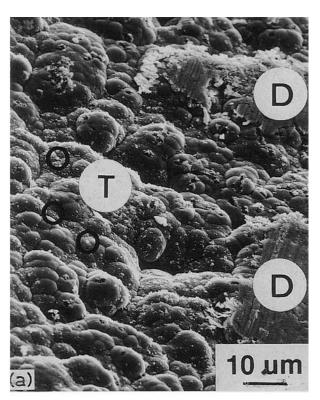


Figure 9 The PCA bracket coated with PEO1 shows the normal PCA surface texture that is partially masked by a porous, low molecular weight polymer (a). The slot edge demonstrates β -Ti arch wire debris (D in (b)).

Figure 10 The PCA bracket coated with PEO2 shows that the normally rough surface is now smoothed over by the medium molecular weight polymer (a). On the slot edge, β -Ti arch wire debris 'D' and a rougher area of polymer coating 'P' can be seen (b).

softer β -Ti arch wire (Fig. 8b). This coating process creates an adherent film of densely packed carbon and hydrocarbon that is reported to have a very low coefficient of friction [11]. However, the 0.3 µm thick coating only mimicked the underlying topography and did not obscure the rough surface normally seen. Making the coating thicker would therefore seem necessary. Recent work [17] has demonstrated that, in a DLC coating that was over three times thicker than the present work, a smooth surface was created which successfully reduced the friction between these same couples. In that work, two DLC coatings were actually evaluated after being formed by different methodologies; SEM's of each sample showed that both coatings were thick enough to mask the PCA surface. Only one method successfully reduced the friction,





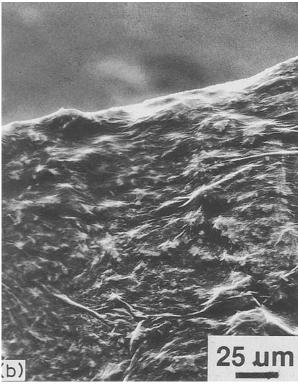


Figure 11 The PCA bracket coated with PEO3 now obliterates the PCA surface and shows no signs of wear (a), and the slot edge is obvious and free of debris (b).

however, suggesting that simply covering up the PCA surface is not a sufficient condition for friction reduction. The surface must be covered by a material that behaves lubriciously.

Thin films of poly(ethylene) oxide, a biocompatible polymer that becomes lubricious when wet, can be created by vacuum drying PEO solutions. The chemistry of this material can be altered by irradiation.

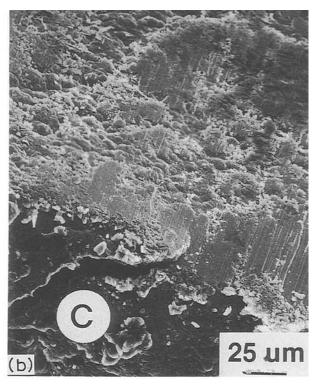


Figure 12 The PCA bracket coated with NF smoothed over the normally faceted surface on which submicron particles of PTFE ('T') and gross debris ('D') of the β -Ti arch wire are evident (a). The edge shows both arch wire debris and an area where the coating has cracked ('C') and separated from the PCA surface (b).

Severing the polymer chain by gamma irradiation induces free radical formation [18], which can promote interaction with the oxygen anion in the alumina lattice and increase the likelihood of the polymer adhering to the alumina surface. Each of the PEO coatings was successful in adhering to the alumina surface and thick enough to obscure its rough texture (contrast Figs. 9a, 10a and 11a with Fig. 6a). The friction remained high, however (Tables IV and V, and also Fig. 5). Either, the PEO coatings were not adequately lubricious, even when wet, or the friction was due to the interaction of the wire with the slot edge. Initially the latter appeared to be case, since arch wire debris was still being generated by the edge (see β -Ti arch wire debris in Figs 9b and 10b). This argument was weakened, after reviewing the NF results .

NF is a composite of soft lubricous polytetrafluoroethylene (PTFE) particles dispersed in a hard nickel phosphorus matrix and is deposited by an autocatalytic (electroless) nickel plating process. This surface treatment is designed to produce a 'low-friction, selflubricating surface' of uniform thickness (as low as $5 \,\mu\text{m}$ [19]. As the outer layer of the coating wears away, fresh particles of PTFE are exposed and continue to provide lubrication. Viewing the NF-coated samples, the material successfully covered over the PCA surface (Fig. 12a), but arch wire debris was still being generated at the bracket edge (e.g. the β -Ti arch wire debris in Fig. 12b). The friction was reduced, when compared with controls (Fig. 5). Therefore, the overriding variable must be the lubricity of the material and not the wire/slot edge interaction. Since this material contains nickel (which can cause allergic reactions in some people [20]) and small PTFE fragments (which have been shown to cause granuloma formation when implanted for prosthetic devices [21]), this modification technique is currently unsuitable for use in the oral environment. Nonetheless, it serves as a model to demonstrate a methodology or technique for successful friction reduction.

In summary, these results show that merely covering the surface, even to the point of obliterating the underlying texture, does not sufficiently reduce the interaction of the PCA and the wire. The overriding factor, more than the slot-edge abrading the wire, is the lubricity of the slot floor and slot walls. Future research in this area should seemingly be directed towards placing a lubricious coating of sufficient thickness into a bracket slot whose edges have been machined into smooth rounded shoulders. This should reduce the friction normally seen and provide a more efficient mechanical system for clinical use.

5. Conclusions

Regarding the four modification methodologies, modifying the surface of PCA brackets by an implantation of Cr⁺ did not alter the surface topography and was not able to overcome the interaction of the wire (whether SS or β -Ti) with the slot floor and slot edge. Thus, the friction remained high. The hard, inert coating of DLC did not mask the normally rough PCA surface; instead the thin coating mimicked the underlying surface. Thus the frictional resistance against SS and β -Ti wires remained unchanged. The soft PEO coatings successfully smoothed over the PCA surface, adhered to it, and did not wear away. Even when wet, though, the friction remained high against either SS or β -Ti wires for each of the molecular weights tested. The composite coating that was comprised of lubricious PTFE dispersed in a nickel compound matrix successfully smoothed over the PCA surface, but was not adequately adherent. The surface, while allowing arch wire debris to be generated by the bracket edge, still demonstrated a reduction in friction against either SS or β -Ti wires.

Regarding the issue of coating thickness versus lubricity, a friction reducing PCA coating should primarily be innately lubricious and yet thick enough to mask the rough PCA surface.

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