Surface roughness and fatigue performance of commercially pure titanium and Ti-6Al-4V alloy after different polishing protocols

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Statement of problem. Surface quality of cast metal is directly related to service performance under fatigue stress. Surface heterogeneities resulting from either finishing or polishing processes or by corrosive agents such as fluoridated solutions, can negatively affect fatigue life. Cast titanium frameworks are difficult to polish, and an accepted polishing protocol has not been established.

Purpose. This study evaluated and compared surface roughness of cast commercially pure titanium (CP Ti) and Ti-6Al-4V alloy submitted to conventional or electrolytic polishing, correlating the results with corrosion–fatigue strength testing performed in artificial fluoridated saliva. Specimens were also tested in air at room temperature to evaluate the effectiveness of the corrosion-fatigue test model.

Material and methods. For each metal, 40 dumb-bell–shaped rods, 2.3 mm in diameter at the central segment, were cast. Conventional polishing was performed on 20 specimens of each metal following the manufacturer's instructions. A source of continuous electrical current was used for electrolytic polishing of the other 20 specimens of each metal, which were immersed in an electrolytic solution containing 5% fluoridric acid, 35% nitric acid, and 60% distilled water. Surface roughness, Ra (μm), was measured with a profilometer, and fatigue tests were carried out with a universal testing machine using a load 30% lower than the 0.2% offset yield strength. After failure, the fractured surfaces were examined using scanning electron microscopy. Surface roughness means were analyzed with a 2-way analysis of variance and the Tukey multiple comparisons test (α=.05).

Results. Electrolytic polishing (0.24 ± 0.05 μm) provided significantly ($P<.05$) lower surface roughness values than conventional polishing (0.32 ± 0.06 μm). Regardless of the polishing protocol, surface roughness of Ti-6Al-4V alloy (0.25 ± 0.06 μm) was significantly lower ($P<.05$) than that of CP Ti (0.31 ± 0.05 μm), and the fluoridated environment did not influence fatigue performance. There was no correlation between fatigue performance and surface roughness.

Conclusion. Surface roughness of Ti-6Al-4V was significantly lower than that of CP Ti. For cast titanium frameworks, the electrolytic polishing regimen was found to be more effective than the manufacturer's polishing instructions with abrasives and rotary instruments. After polishing, differences in surface roughness values did not affect corrosion-fatigue performance. (J Prosthet Dent 2005;93:378-85.)

CLINICAL IMPLICATIONS

For titanium frameworks, electrolytic polishing may be more effective than the manufacturer's recommended polishing instructions. Small differences in surface roughness values after polishing did not affect corrosion-fatigue performance.

For decades, investigators have attempted to identify a viable, cost-effective alternative to precious alloys for fixed and removable partial dentures.[1,2] Since the 1970s, nickel-chromium (Ni-Cr) and cobalt-chromium (Co-Cr) alloys have been popular choices for manufacturing fixed and removable partial denture frameworks.[1-3]

However, the exploration of alloys containing nickel and beryllium has been limited due to persisting doubts as to biological safety.[4,5] Titanium was introduced in the 1970s for cast dental applications and became popular in

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Table I. Metals evaluated

Metal	Manufacturer	Chemical composition* (% wt)
CP Ti (Tritan)	Dentaurum, Pforzheim, Germany	98.5 Ti; 0.25 O_2; 0.03 N; 0.30 Fe; 0.10 C and 0.015 H
Ti-6Al-4V	Camacam Industrial, Sao Paulo, Brazil	Ti: bal.; 6.15 Al; 4.08 V; 0.026 C; 0.21 Fe; 0.009 N

*Information provided by manufacturers.

the 1980s for crowns and removable partial dentures (RPDs).[6] The emergence of titanium as a suitable choice for manufacturing dental frameworks was primarily due to improved dental casting devices. Its high melting point (±1700°C), reactivity, and low density make casting with conventional centrifugal casting machines difficult.[7] The increased use of titanium can be attributed to the development of casting machines that allow for vacuum injection of the melted alloy.[7,8] In addition, the high strength and low weight of this metal result in mechanical performance similar to that of Type IV gold alloys.[8]

Although many types of titanium alloys are available, the titanium-aluminum-vanadium alloy (Ti-6Al-4V) is the most commonly used, since its physical and mechanical properties are superior to those of commercially pure titanium (CP Ti, 99.8% titanium).[8] Compared to CP Ti, Ti-6Al-4V has greater flexural strength (890 MPa vs 390 MPa), superior hardness (350 VHN vs 160 VHN), and a slightly higher thermal expansion coefficient (11.8×10^{-6} °C vs 11.4×10^{-6} °C).[8] Consequently, the mechanical properties of Ti-6Al-4V are similar to those of Ni-Cr and Co-Cr alloys.[8]

Titanium interacts well with both hard and soft tissues as a result of its excellent biocompatibility, making it the most biologically compatible alternative to Co-Cr and Ni-Cr alloys.[9-12] Due to the oxide layer that forms instantaneously and spontaneously on its surface, titanium is highly resistant to corrosion under static conditions.[13] However, some chemical agents, primarily fluoridated solutions, have been shown to cause surface corrosion.[13,14] These solutions induce instability, generally under cyclic stresses, resulting in corrosion pits and surface irregularities.[14-17]

Surface heterogeneities in conjunction with the cyclical nature of insertion and removal of the prostheses may predispose RPD frameworks to failure caused by fatigue.[2,14,18-19] The fatigue phenomenon begins at the surface and is responsible for 90% of failures in service due to mechanical causes.[20,21] Polishing cast surfaces increases resistance to corrosion and governs fatigue performance.[14,20] In general, the poorer the surface finish, the lower the fatigue strength.

Finishing and polishing cast titanium frameworks are difficult procedures.[22-24] An oxide surface layer differentiates titanium from other alloys and may hinder acquisition of a mirror-like surface finish in this metal. The electrolytic polishing method is used to assist with the arduous procedure of finishing and polishing Co-Cr alloys,[1-2] and the protocol is mentioned as a method for obtaining polished surfaces in cast titanium frameworks,[25] although the authors could not identify a study describing the actual benefits of this method regarding titanium or titanium alloys. Consequently, the aim of this study was to evaluate and compare the surface roughness of CP Ti and Ti-6Al-4V alloy after conventional or electrolytic polishing. Results were correlated with corrosion–fatigue strength tests in fluoridated artificial saliva. A fatigue test was also performed on specimens in air at room temperature to evaluate the corrosion-fatigue test model.

MATERIAL AND METHODS

One hundred dumb-bell–shaped rods with a 2.5-mm diameter at the central segment and a 40-mm length were cast in a vacuum-injection titanium casting machine (Rematitan System; Dentaurum, Pforzheim, Germany). Fifty rods each were cast in commercially pure titanium (CP Ti) and titanium-aluminum-vanadium alloy (Ti-6Al-4V) (Table I).[26] To accomplish this, wax patterns of each specimen were invested (Rematitan Plus; Dentaurum) using 250 g of powder for 40 mL of liquid and mixed under vacuum according to the manufacturer's instructions. After setting, invested specimens were heated using a slow heat cycle.[14] Following identical manufacturing procedures, CP Ti and Ti-6Al-4V specimens were cast using a 31-g ingot. Molds were immediately quenched in cold running water after casting.

Specimens were divested and airborne-particle abraded at a pressure of 80 lb/in^2 with 100 μm aluminum oxide.[27] Radiographs of the specimens were made with radiographic film (Ektaspeed Plus; Eastman Kodak, Rochester, NY)[14,28] and evaluated for indications of internal defects. Those with external heterogeneities identified by visual examination or with internal voids evidenced by the radiographic examination were discarded. A total of 44 intact specimens were selected for each metal.

Specimens were fastened to a low-speed mechanical grinding device (Kavo Brasil, Joinville, Brazil) and finished with 100- to 500-grit silicon carbide papers (Imperial; 3M Brazil, Sumare, Brazil) to eliminate surface irregularities. During the finishing process, specimen diameter was constantly verified until a 2.3-mm central segment was obtained. An electronic caliper (727 Digital Caliper; Starrett Industry and Trade, Sao Paulo, Brazil) was utilized to assure accuracy within 0.01 mm.[14]

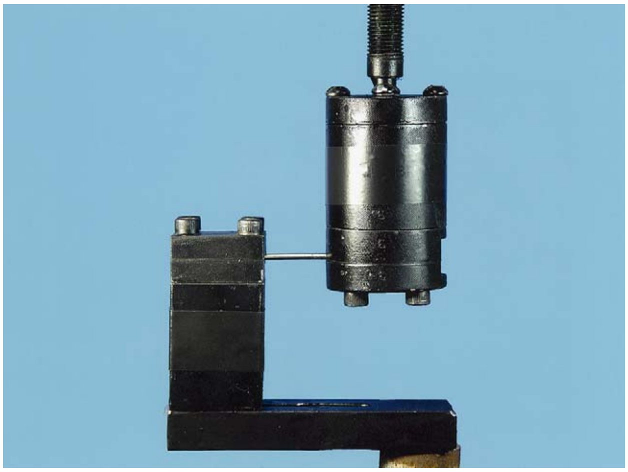

Fig. 1. Fatigue test configuration in cantilever bending.

Fig. 2. Plastic apparatus with fluoridated saliva for corrosion-fatigue test.

Table II. Composition of fluoridated artificial saliva (in 1000 mL)

Solution	Composition
Fluoridated artificial saliva (pH=7)	NaCl–0.4 g; KCl–0.4 g; NaOH (1M)–1.25 mL; $CaCl_2 \cdot 2H_2O$–0.221 g (1.5 mM); NaH_2PO_4–0.124 g (0.9 mM); Na_2S–0.005 g; NaF–2.223 g (1000 ppm); 1 g urea

Table III. Mean values (SDs) of surface roughness (μm) for CP Ti and Ti-6Al-4V alloy

	Polishing protocols		
Metal	**Conventional**	**Electrolytic**	**Mean value**
CP Ti	0.35 (0.05)	0.27 (0.06)	0.31 (0.05) a
Ti-6Al-4V	0.28 (0.08)	0.21 (0.04)	0.25 (0.06) b
Mean value	0.32 (0.06) A	0.24 (0.05) B	

Mean values followed by different uppercase letters in rows and lowercase letters in columns are significantly different (*P*<.05) according to Tukey test.

Table IV. Mean values (SDs) of number of cycles to fracture for CP Ti and Ti-6Al-4V alloy after conventional polishing tested in fluoridated artificial saliva and in air

Environment	CP Ti	Ti-6Al-4V
Saliva	2.084 (934) B b	5.940 (2.196) A a
Air	7.947 (2,065) A a	8.712 (3.094) A a

Mean values followed by different uppercase letters in rows and lowercase letters in columns are significantly different (*P*<.05) according to Tukey test.

Table V. Mean values (SDs) of number of cycles to fracture for CP Ti and Ti-6Al-4V alloy after electrolytic polishing tested fluoridated artificial saliva and in air

Environment	CP Ti	Ti-6Al-4V
Saliva	2806 (1.968) B b	6902 (3.109) A a
Air	8130 (3.679) A a	8610 (5.683) A a

Means followed by different uppercase letters in rows and lowercase letters in columns are significantly different (*P*<.05) according to Tukey test.

Specimens were equally divided; half of each of the metal specimens (n = 22) were polished conventionally, and the other half were polished electrolytically. For conventional polishing, titanium rubber disks (Gummipolierer; Dentaurum), adapted to a low-speed hand piece, were initially utilized. Afterward, a 60-mm–diameter polishing brush (Chungking; Dentaurum) with titanium polishing paste (Tiger Brilliant; Dentaurum) was used for final polishing. For electrolytic polishing, a continuous source of electrical current was adjusted to 4 V with 0 to 2 A current. Each specimen was immersed for 5 minutes[25] in an electrolytic solution containing 5% fluoridric acid, 35% nitric acid, and 60% distilled water.[25]

After polishing, surface roughness was measured with a profilometer (SurfCorder SE 1700; Kosaka, Tokyo, Japan) with a diamond stylus tip for cylindrical surfaces (AG5; Kosaka), which contacted the surface at a constant speed of 0.05 mm/s with a force of 0.7 mN. Surface roughness was characterized by the height parameter, Ra (in micrometers, μm), which is the arithmetical mean of the absolute values of the profile departures within the length evaluated. The cut-off value was set at 0.08 mm to characterize surface roughness. Statistical calculation of surface roughness regardless of polishing protocol was performed using an average of 3 surface roughness measurements parallel to the long axis at the central segment of each specimen.

To establish the optimal load for the fatigue test, 4 arbitrarily-selected specimens of each metal, 2 for each polishing regimen, were submitted to a flexural test in

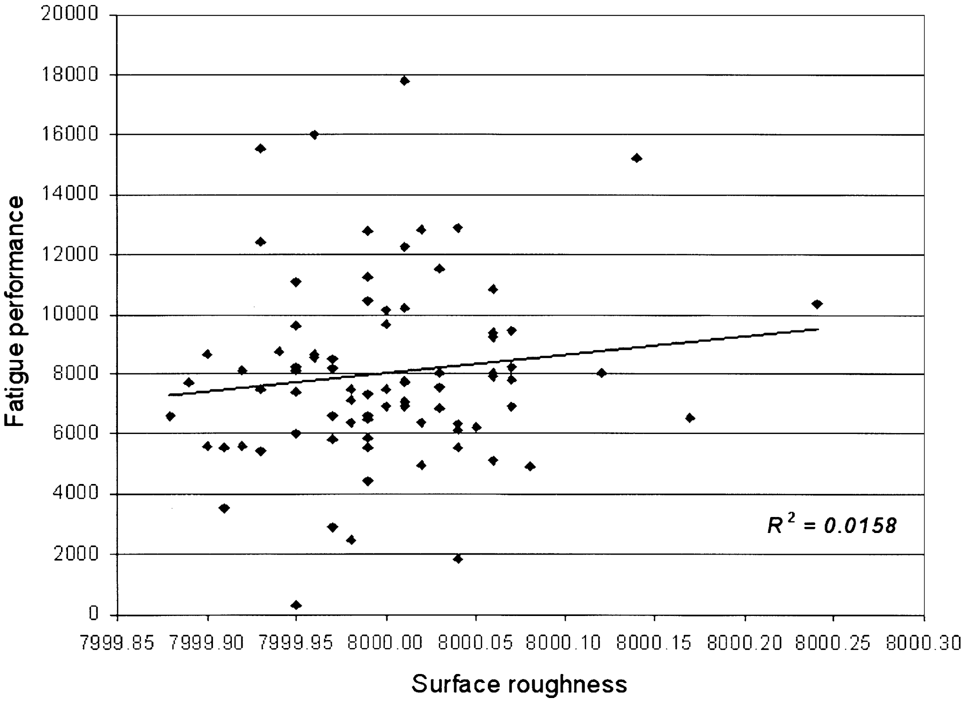

Fig. 3. Dispersion diagram associating fatigue and surface roughness. Note large dispersion of points and low correlation coefficient (R^2=.0158) between variables.

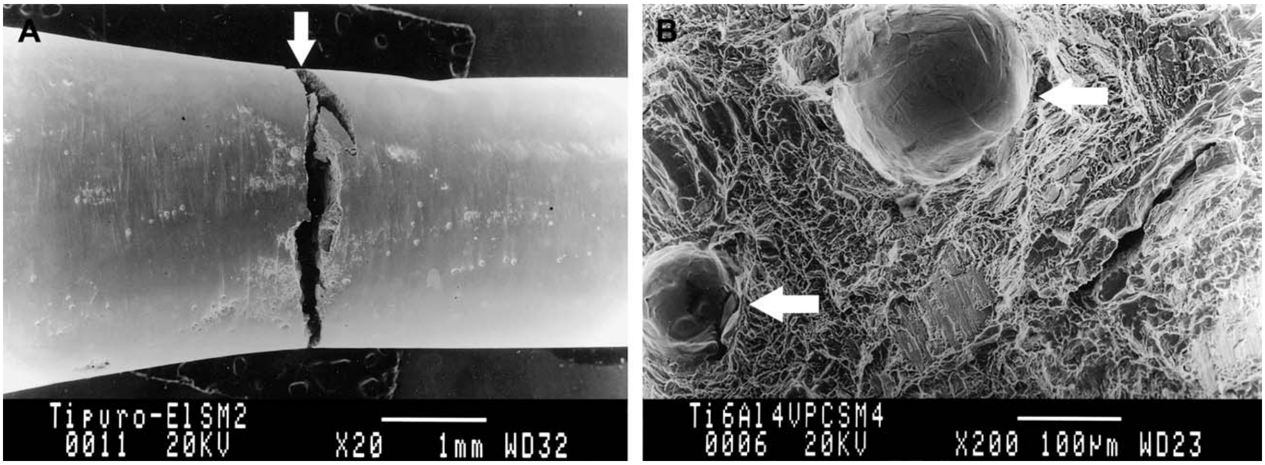

Fig. 4. SEM photomicrographs: **A**, Lateral aspect of electrolytically polished CP Ti specimen that failed after 7876 cycles tested in air (×20). *Arrow* shows fatigue fracture site. Note heterogeneities on entire surface. **B**, Fractured surface of conventionally polished Ti-6Al-4V alloy specimen that failed after 4660 cycles tested in air. *Arrows* show internal pores (×200).

a universal testing machine (Test Star II; MTS Materials Testing Systems, Minneapolis, Minn). Stress-strain curves were determined, and the mean yield strength at 0.2% offset was calculated for both metals. To simulate severe clinical stress without plastic deformation—for example, during insertion and removal of an RPD framework—the fatigue test was performed using a load 30% lower than the calculated yield strength, resulting in 5 kgf for CP Ti and 6 kgf for Ti-6Al-4V alloy. The load frequency of the fatigue test was 10 Hz, and the load waveform was square and R=1 (ratio of minimum and maximum loads).[14]

A high-cycle constant deflection fatigue test in cantilever bending was conducted using the universal testing machine (Test Star II). One end of the specimen was mounted in an articulated superior grip and the other fixed in an oscillatory inferior grip. The traveling distance between the 2 ends was calibrated at 29 mm.[14] The machine automatically registered the number of fatigue cycles prior to fracture (Fig. 1).

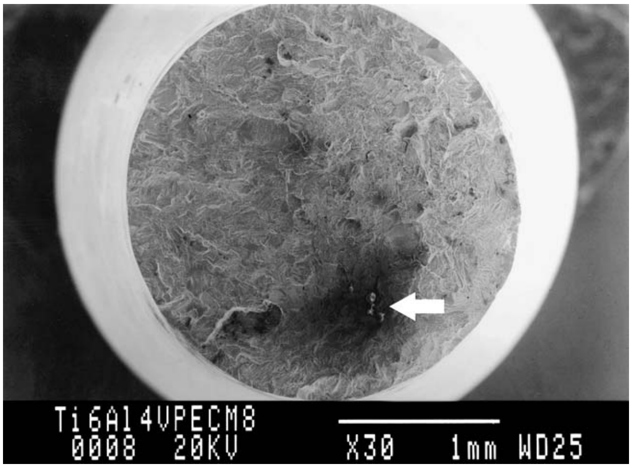

Fig. 5. SEM photomicrograph of fractured surface of electrolytically polished Ti-6Al-4V alloy that failed after 6421 cycles tested in fluoridated artificial saliva. EDX analysis of outlined fractured surface site showed evidence of corrosion and metal reaction with fluoridated solution (×30).

To perform the corrosion-fatigue test, a plastic apparatus was adapted around the oscillatory inferior grip of the machine to concurrently test 10 specimens of each metal while submerged in fluoridated artificial saliva prepared by the authors (Fig. 2). Table II lists the chemical constituents and pH of the prepared solution.[14,29] The artificial saliva contained inorganic compounds and urea with no proteins. The remaining 10 specimens of each metal were tested under fatigue stress in air at room temperature to evaluate the effectiveness of the corrosion-fatigue test model. Statistical analysis considered the number of fatigue cycles before failure for the 2 metals tested in either fluoridated artificial saliva or in air at room temperature.

Surfaces of the fatigue fractures were examined in representative specimens of each test group using a scanning electron microscope (SEM) (Jeol JXA 840 A Electron Probe Microanalyser; JEOL, Tokyo, Japan). SEM photomicrographs (×20 to ×200) were made to visually analyze the fractured surfaces and determine possible causes of failure. An energy-dispersive x-ray (EDX) analysis was also performed to substantiate the eventual reaction between metals and the fluoridated solution or other corrosion compounds. Surface roughness and fatigue-strength values were subjected to a 2-way analysis of variance (ANOVA). Significant differences were submitted to a Tukey multiple comparisons test (α=.05). A regression method was used to fit a mathematical function of the variables, fatigue and surface roughness.

RESULTS

Table III shows surface roughness results relative to polishing protocols. Not only did results differ according to polishing technique, but material type also presented significant differences (P<.05). Surface roughness of both metals was significantly lower after electrolytic polishing (0.24 ± 0.05 μm) than after conventional polishing (0.32 ± 0.06 μm) (P<.05). Irrespective of the polishing protocol, CP Ti presented significantly increased surface roughness (0.31 ± 0.05 μm) relative to the Ti-6Al-4V alloy (0.25 ± 0.06 μm) (P<.05).There was no significant difference in fatigue strength of CP Ti and Ti-6Al-4V specimens tested in air at room temperature after both conventional (Table IV) and electrolytic (Table V) polishing (P>.05). However, these specimens had a significantly higher (P<.05) fatigue strength (number of cycles) than those tested under the same conditions in fluoridated artificial saliva (Tables IV and V). While the fluoridated medium did not influence fatigue strength of the Ti-6Al-4V alloy, this was not true of CP Ti, whose fatigue performance was significantly lower (P<.05) (Tables IV and V).

The regression method is presented in Figure 3 as a dispersion diagram. Means were added to 8000 to eliminate negative values. No correlation between surface roughness values and fatigue performance was observed, regardless of polishing protocol, metals, and environments. Figures 4 and 5 illustrate SEM photomicrographs after fatigue tests. Surface heterogeneities and internal pores were easily differentiated (Fig. 4). Evidence of metal reactions with the fluoridated artificial saliva was also observed. EDX analysis revealed elements of the fluoridated solution on the fractured surface (Fig. 5).

DISCUSSION

Advantages such as biocompatibility and improved mechanical properties, including high strength and relatively low weight, support the use of titanium and titanium alloys for prosthodontic purposes.[6-8] Nevertheless, disadvantages for use relative to conventional dental castings include high melting temperature and high chemical reactivity.[2,6,25,26] These conditions predispose the metal to form a thick oxide layer that is difficult to remove with conventional finishing and polishing procedures.

Although metal framework surfaces are expected to be more regular, uniform, and smooth after laboratory finishing and polishing stages,[1,23,24] surface heterogeneities may persist. As a result, surface morphology and increased bacterial adhesion[22] may enhance the corrosion process.[1,14,20,21] Surface quality also governs fatigue life.[14,20,21] The fatigue phenomenon, generally evaluated by corrosion–fatigue strength tests, is accelerated by remaining surface irregularities.[2,19] The transverse section area, structure length and width, and intrinsic material properties, as well as dynamic efforts, also accelerate fatigue.[18]

Electrolytic polishing is an auxiliary method that optimizes polishing techniques. The procedure is widely used in commercial laboratories that manufacture Co-Cr alloy RPD frameworks.[1,2] This polishing technique exposes a new surface smoother than the rough areas formed after casting.[2] Furthermore, electrolytic polishing increases an alloy's resistance to corrosion[2] and, consequently, its fatigue performance. Comparison of conventional polishing following the manufacturer's recommendations and electrolytic polishing revealed the latter to produce significantly lower surface roughness. Considering that the surface hardness of titanium is approximately one third greater than that of Au alloys,[2,29] electrolytic polishing may be advisable to facilitate the arduous polishing process until the desired surface smoothness and brightness are reached. Although manufacturers do not recommend electrolytic polishing for titanium prosthesis fabrication, the procedure may minimize residual irregularities not reached by conventional polishing, thereby optimizing the polishing process and providing a smoother surface.

Despite the polishing protocol, a comparison of surface roughness in finished CP Ti and Ti-6Al-4V specimens revealed CP Ti to be significantly rougher (Table III). Differences in melting temperature, density, and reaction to (or interaction with) investment material all contribute toward improved castability of Ti-6Al-4V alloy compared to CP Ti.[7] Abundant surface heterogeneities such as notches and porosity in CP Ti specimens are probably due to poorer casting characteristics (Fig. 4, *A*).

Fluoridated oral solutions may modify titanium's electrochemical activity, making it prone to corrosive attacks.[13,16,17,29] Titanium has been found to be susceptible to corrosion when submersed in solutions containing more than 20 ppm fluoride ions.[17] Occurrence of corrosion is accentuated with acidic-pH fluoridated solutions. The lower the pH solution, the greater the chances of corrosion.[13,22] Boerel[13] considered the influence of pH on titanium surfaces to be greater than the concentration of fluoride ions.

Corrosion resistance of titanium and its alloys appears to be influenced by the type of stress to which they are submitted. Cyclic loading seems to have a dominant effect. Zavanelli et al[14] stated that fluoridated solutions would have the same deleterious effect on fatigue performance of titanium frameworks as an oral medium composed of saliva. Exposure of RPD frameworks to acidulated fluoridated solutions such as prophylactic gels is uncommon. However, tooth brushing, a circumstance in which a removable metal framework is in contact with 1000 ppm fluoridated solutions, is common. The corrosion-fatigue test model adopted in the present study was intended to simulate an extreme clinical situation for RPD frameworks.

An association of fluoridated solutions with cyclic loading, such as that occurring during insertion and removal of RPD frameworks, was evaluated. Results from this in vitro simulation were compared to those obtained in the fatigue strength test executed in air at room temperature. While fatigue strength of CP Ti specimens tested in fluoridated artificial saliva were significantly lower than those tested in air at room temperature, the medium had no significant effect on the fatigue strength of Ti-6Al-4V specimens (Tables IV and V). Fatigue properties of CP Ti in fluoridated solution were different from the Ti alloy, probably due to the poorer surface quality as seen in Figure 4, *A*. Significant differences in surface roughness values most likely played an important role in fatigue performance.[14,20] SEM photomicrograph and EDX analysis of the fractured surface of a specimen tested in fluoridated medium revealed apparent corrosive compounds (Fig. 5), as well as residual elements of solution that probably resulted from surface reactions. Corroded areas precipitated early failure during crack propagation of specimens exposed to the fluoridated solution, in contrast to specimens cycled in air.[14]

Fatigue strength is the stress at which a material fails under repeated loads. The fluctuating stress necessary to cause fatigue failure consists of completely reversed cycles (equal maximum and minimum stresses), repeated cycles (unequal maximum and minimum stresses), and periodic and unpredictable overloads. Fluctuating fatigue cycles could range from values below or above the structural yield strength described, respectively, as low-cycle (high stress) and high-cycle (low stress) tests.[14,20,21] In the present study, a criterion was established for determining load in the fatigue strength test. Based on a preliminary flexural test, a load 30% lower than the yield strength calculated at 0.2% offset was used to simulate insertion and removal of the RPD apparatus with no permanent deformation.[14]

Loss of retention of RPD frameworks in service after a short time is not uncommon, probably due to permanent deformation,[21] which was not considered in the present study. Permanent deformation could indicate that structures were submitted to a low-cycle fatigue, thereby decreasing the number of cycles necessary for failure.[20,21] Mechanical adjustments provided by technicians and dentists could reduce the fatigue life and cause premature failure of a framework, a phenomenon called "overstressing."[14,20,21] Furthermore, galvanic currents between CP Ti and a number of different dental alloys have been shown to be present when exposed to artificial saliva, but at a lower rate than that between gold alloy and the same dental alloys.[15] This polarization may alter surface characteristics and affect fatigue crack propagation.

In general, causes of material cyclic fatigue typically occur from superficial heterogeneities and anisotropy of the material. Metallurgical concentrators such as pores, notches, and inclusions are particularly contributing and can result in catastrophic failure and reduced cycle number. Despite the precision of the titanium casting technique, SEM photomicrographs of fractured surfaces showed large internal pores that were not detected by radiographic analysis (Fig. 4, *B*). Defects highly influence results and are probably the primary cause associated with the high variability of data found in other studies.[14]

Fatigue strength test results facilitate estimating the clinical life of a prosthesis, as well as improving its performance and quality.[14] According to Craig and Powers,[2] alternate applications of fatigue-type stress during the removal of RPD frameworks from the mouth or during their replacement and adjustment into position, probably amount to less than 1500 deflections per year. However, Vallittu and Kökkönen[19] estimated RPD clasps to bend 10 times a day during insertion and removal, resulting in 3600 deflections per year. Based on these reports, as well as the number of cycles to failure in a fluoridated environment found in the present study, a titanium RPD framework may provide 2 to 6 years of use without failure due to clasp fractures.

The fatigue phenomenon is intimately related to surface structure. Fabrication of prosthodontic frameworks requires appropriate physical properties and dimensional precision in addition to well-polished surfaces. Consequently, the less a surface is finished, the less resistant it is to fatigue since the two are intimately related. There was no correlation between surface roughness and fatigue performance found in the present study. Even so, future research should re-attempt the regression method used to fit a mathematical model for the variables of fatigue and surface roughness. This method considered both metals, differences in surface roughness values after the polishing protocols, the different environments, and the highly variable cyclic test results. It should be noted that separate conditions could play a different role in the clinical situation.

In addition, low surface roughness values and similar surface quality characteristics after both conventional and electrolytic polishing protocols were probably not capable of producing results sensitive to cyclic testing. Mechanical properties and the previously mentioned results support the hypothesis that both polishing protocols are suitable for cast titanium RPD frameworks in reducing corrosion-fatigue stress and premature failure. However, it is important to note that standardized and well-polished surfaces were compared in this study. Different results may be expected if different surface qualities are compared, since surface quality may negatively influence fatigue performance.

CONCLUSIONS

Within the limitations of this study, the following conclusions can be drawn:

1. For cast titanium frameworks, electrolytic polishing caused significantly lower surface roughness values than conventional polishing ($P<.05$).
2. Regardless of the polishing protocol, surface roughness of the Ti-6Al-4V alloy was significantly lower than that of CP Ti ($P<.05$).
3. Fatigue performance of CP Ti was significantly lower in a fluoridated environment ($P<.05$), whereas this environment had no influence on Ti-6Al-4V alloy fatigue life ($P>.05$).
4. No correlation between surface roughness and fatigue performance was determined.

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