COMPRESSIVE FATIGUE IN TITANIUM DENTAL IMPLANTS SUBMITTED TO FLUORIDE IONS ACTION

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ABSTRACT

The aim of this study was to assess the influence of a fluoridated medium on the mechanical properties of an internal hexagon implant-abutment set, by means of compression, mechanical cycling and metallographic characterization by scanning electronic microscopy. Five years of regular use of oral hygiene with a sodium fluoride solution content of 1500 ppm were simulated, immersing the samples in this medium for 184 hours, with the solutions being changed every 12 hours. Data were analyzed at a 95% confidence level with Fisher's exact test. After the action of fluoride ions, a negative influence occurred in the mechanical cycling test performed in a servohydraulic machine (Material Test System-810) set to a frequency of 15 Hz with 100,000 cycles and programmed to 60% of the maximum resistance of static compression test. The sets tended to fracture by compression on the screw, characterized by mixed ruptures with predominance of fragile fracture, as observed by microscopy. An evidence of corrosion by pitting on sample surfaces was found after the fluoride ions action. It may be concluded that prolonged contact with fluoride ions is harmful to the mechanical properties of commercially pure titanium structures.

Uniterms: Titanium; Dental implants; Fluorides; Corrosion; Fatigue.

INTRODUCTION

One of the most important phenomena to be considered for an implant to be successful is resistance to corrosion, as the material to be used will be constantly submitted to mechanical and thermal forces and to the aggressiveness of the host medium[13].

Corrosion is the result of electrochemical reactions that occur between a metal material and the surrounding environment. Titanium is known to present a high degree of immunity against the attack by the majority of mineral acids, such as chlorides, and its biocompatibility associated with good mechanical resistance make it very useful in applications as a biomaterial[4].

Considered to be one of the few means capable of attacking the titanium surface with corrosive action, fluoride ions are effective components in this process. The use of fluoridated gels and solutions containing high levels of fluorides, as a prophylactic measure in Dentistry, has been increasingly more widespread over the last 40 years, due to the great impact on caries prevention[21]. The use of these substances on titanium surfaces, including their damaging action on implant surfaces, is a subject that has been approached in few studies. Which repercussion this damage to the implant surface will have on the mechanical behavior of implants over time is still unknown.

The study of dental metal alloys, especially of titanium, is crucial in fluoridated mediums due to a large number of high fluoride content preparations. There are few studies on the behavior of dental implants in the face of the corrosive action of fluoride containing chemical agents, and it is necessary to characterize the material and interpret its behavior after submission of the implant-abutment sets to the action of fluoride ions.

The aim of this study was to assess the action of fluoride ions on the mechanical properties of implant-abutment set by means of compression tests, mechanical cycling and metallographic characterization by scanning electronic microscopy.

MATERIAL AND METHODS

In this study, commercially pure titanium dental implants with internal hexagon implant - Connect AR treated surface (Conexão Sistema de Prótese, São Paulo, SP, Brazil; Lot 4900) were used. The implants and their components (titanium screw for internal hexagon preparation post (Conexão Sistema de Prótese, São Paulo, SP, Brazil) were removed from their original packaging and mounted according to the manufacturer's instructions.

A manual torquemeter (Conexão Sistema de Prótese, São Paulo, SP, Brazil) was used at 20 N for the internal hexagon implant-abutment set to attain the recommended torque. Once the recommended torque was obtained, the sets were divided into two groups of mechanical test. Group 1, characterized for the non-axial compression test, with 5 samples that were not submitted to ions fluoride F(-); and Group 2, characterized for the mechanical cycling test, with 5 samples that were not submitted to ions fluoride F(-) and 5 samples that were submitted to ions fluoride F(+).

After that, the tests began to be carried out on the F(-) sets that have not been submitted to the action of fluorides. The F(+) sets were placed into a 1,500 ppm sodium fluoride solution pH 6.8 (School of Pharmaceutical Science, UNESP, São Paulo State University, Araraquara, SP, Brazil) until the time the respective tests were carried out.

According to the protocol proposed by Siirilä and Könönen[19] (1991), a model that simulates the clinical conditions of fluoride use was designed. In this model, the test specimens were statically submerged in the sodium fluoride medium (1,500 ppm pH 6.8) for 184 hours (7.5 days), simulating contact with fluoride for 21 times a week during 5 years, with a mean of 2 minutes at a time (mean estimate of brushing with a fluoride dentifrice 3 times a day). The solution was changed every 12 hours, and each implant was washed in water for a 30-second interval before being submerged again in the replaced solution. This change procedure was repeated until the end of the stipulated time. Additionally, the sets were completely submerged in the solution although it is more common to find abutments exposed in the oral cavity and not the whole set. The rationale is that this is an in vitro study and can extrapolate the usual cases and simulate a screw exposition, for instance.

Two types of mechanical tests were carried out on the implant-abutment sets: static and dynamic. The static test was the non-axial compression test and the dynamic test was the mechanical cycling, also called fatigue test. The static test was carried out with the goal of characterizing the strength of the implant-abutment sets without fluoride contact. Data obtained were the basis for the dynamic test to which F(-) and F(+) sets were submitted.

The tests were carried out in a servohydraulic machine (Material Test System - MTS 810; MTS System Corporation, Minneapolis, USA), equipped with Test Star II software. The mechanical test machine was fitted with a 10 kN load cell at a constant speed of 1 mm/ min. The force was applied by means of a straight pin fixed to the load cell with a flat end with a circular cross-section.

To perform mechanical cycling, a load with a value of 60% of the compression strength of the test specimens was used, and 100,000 cycles of the mechanical test machine were set at a frequency of 15 Hz[1,7].

Surface analysis was done with a scanning electronic microscope (Model T-330 A JEOL-JSM, Tokyo, Japan) coupled to an energy dispersive analyzer and a photographic camera. Fractography analysis was further performed according to Kerins and Phillips[6] (1992).

Results for fracture location and screw loosening were analyzed at a 95% confidence level with Fisher's exact test.

RESULTS

During the compression test in the F(-) sets from Group 1, which were not submitted to fluoride ions, and the mechanical cycling test in the F (-) sets from Group 2, the maximum strength limit showed no dependence to the fatigue resistance (Table 1 and 2).

Regarding the mechanical cycling test (Group 2), the sets submitted to fluoride treatment, F(+)fractured before attaining the 100,000 cycles, differing from the F(-) sets. Fracture location occurred on the screw for all sets, as demonstrated in Table 2.

With regard to the screw loosening variable, Table 2 shows no frequency difference between F(-) sets and F(+) sets.

Scanning Electron Microscopy

Figure 1 shows the surface of a F(-) set and Figure 2 shows the surface of a F(+) set, from Group 2. It is possible to observe differences between them, as the first one presents some irregularities from fabrication and the second one presents signs of corrosion.

Figures 3 and 4 show the fracture surface in the screw characterized as mixed fracture and some areas of corrosion. Figure 5 illustrates the stages of fracture by fatigue.

DISCUSSION

In this study, the implant-abutment sets were exposed to a 1,500 ppm fluoridated medium for 184 hours, taking an exposure to fluoride ions of 2 minutes daily, for 5 years, with

TABLE 1- Maximum load (N) during implant compression and fracture locations

Maximum Load	Fracture Location
424.2	Center of screw
415.5	Center of screw
500.0	Head of screw
475.0	Head of screw
420.5	Center of screw

a frequency of 3 times a day, as a reference, without taking into account the residual fluoride that remains in the mouth immediately after brushing. It is known that fluoride concentrations in saliva fall drastically after brushing with a 1,250 ppm dentifrice for 90 seconds. Furthermore, fluoride concentrations even higher than 200 ppm are found in saliva, which fall during the next 30 minutes, stabilize below 29 ppm and reach 7 ppm in the following 24 hours[3]. However, the presence of fluorides is not restricted to the time of duration of oral hygiene procedures (use of dentifrice or fluoridated mouthwash) or prophylactic therapy. Then, the use of low pH fluorides may be more prejudicial to titanium[22].

To evaluate the implants and its joints with their components, as well as their stability and strength, models are widely used in research in recent times, in which eccentric loads are applied to the long axis of implants, whether at angles greater than 15 degrees or by means of devices that take the moment of flexion outside the axial shaft[8,11,12,14].

In this research, the experimental model adopted presents the previously mentioned characteristics, accepted as effective for reproducing the less favorable clinical conditions (within the limitations of the case) in the mouth.

The highest maximum non-axial compression load limit values for the group of internal hexagon implants that were not submitted to the action of fluoride ions F(-) are basically due to the stability of this system, in which the forces are distributed with greater uniformity among the implant, screw and abutment, although the maximum strength limit was shown to be practically independent of the resistance to fatigue (Table 1 and Table 2).

The simplest way of increasing fatigue resistance is by increasing implant diameters. Implants 4mm wide have 30% more fatigue resistance than implants of 3.75mm, however, a more resistant implant does not solve the problem, it only takes it to another part of the system, which may be the adjacent bone[15].

Due to the ductility of titanium, it is not possible for a c.p.Ti implant to fracture with one cycle only. With the application of cyclic load, the material starts to develop internal micro-cracks that may increase in number and size according to the number of cycles. Resistance to fatigue is given by the capacity to resist these repeated loads. In this study, low cycle fatigue was performed (up to 10^5 cycles)[4], as is noted in Table 2, and in which it is found that the implant-abutment sets that were submitted to the action of fluoride ions F(+) presented failure by fracture before attaining the cycles pre-determined in the non-axial compression test.

Under the load conditions to which the implants in this study were submitted, the fracture location, as presented in Table 2 shows that it was the screw that presented the greatest tendency to fracture, as was expected on the basis of the weak link concept for components proposed by Binon[2] (1994). In this proposal, the weak link was the gold screw that connected the gold cylinder to the titanium transgingival intermediate in the Nobel Biocare system. This screw should fracture before another component. This widely accepted concept was extrapolated to all the screws in the different implant systems.

In previous studies, it was reported that in some set systems, the first component to present failure or fracture when submitted to fatigue was not the one designed for such purpose. This occurred in the study of Basten, et al.[1] (1996), in which the titanium screw of the Estheticone system component presented fracture and not the gold screw of the cylinder, as expected.

The internal and external implant connections have been compared, and a better stability has been found in internal,

TABLE 2- Results obtained after submission of implant-abutment sets to mechanical cycling test, with F(+) and without F(-) fluoride treatment, as regards number of cycles borne by sets, fracture locations of the sets and occurrence of screw loosening during the test

	Number of cycles borne by sets	Fracture location of the sets	Occurrence of screw loosening
F(-)	> 100.000	Center screw	Yes
	> 100.000	Center screw	No
	> 100.000	Center screw	No
	> 100.000	Center screw	No
	> 100.000	Center screw	Yes
F(+)	43.890	Center of screw	No
	60.489	Head of screw	Yes
	40.000	Head of screw	Yes
	50.000	Head of screw	Yes
	46.500	Head of screw	No

Note: > 100.000: Implant-abutment set that bears the total number of fatigue
Fracture location - Fisher's exact test: p=0.024 (significant, p<0.05)
Screw loosening - Fisher's exact test: p=0.500 (non significant, p>0.05)

taper-shaped connections, in the face of the action of cyclic forces[11]. In this experiment, the internal connection model in implants was the internal hexagon, whose mechanical behavior is comparable with that of the tapered connection, as has already been referred to in a study in the year 2000[14]. This system, as was observed in this study, presented the weak link in the screw, which may reduce the possibility of an implant fracture even further. A fracture that is rare, according to the literature but, in case it does occur, may be caused due to overload by flexion of the implants. Overload by flexion may be defined as a situation in which, the occlusal forces in an implant-supported prosthesis, exercise a moment of flexion by means of the cross section of the implant body, leading to the loss of marginal bone or eventually, to fracture by implant fatigue[16].

In the present experimental model, this overload by flexion occurred due to the 30–degree angle in which the implant was placed. This produced a moment of flexion at the time of axial load application. This axial force is considered a contributing factor to increase the risk of overloading by flexion when it acts in implants that present a slope greater than 15 degrees. In other circumstances, such as in the placement of a cantilever or increase of the height of a restoration (distance from the supporting bone crest to the occlusal surface of the restoration)[17].

Comparing the frequency of screw loosening before and after fluoride application, no pattern was found that would suggest some type of positive or negative influence. Screw loosening has been reported as part of the problems that may occur in an implant-based rehabilitating treatment. For the internal hexagon systems, a very low or non-existent screw loosening index has been resported[5], a condition that did not occur in this study, as no differences were found with respect to the screw loosening variable.

Scanning Electron Microscopy

It has been reported that after a time of use in the mouth, the components may lose their shine[10] or undergo color changes[19]. This may be attributed to the changes in the oxide layer resulting from corrosion reactions. These surface reactions are more intense when the implants are submitted to the actions of fluoride ions. Macroscopically, titanium surface presented loss of shine and dark coloring in the groups submitted to the action of fluoride ions.

The scanning electronic microscopic analysis showed that the titanium surface not submitted to the action of fluoride ions exhibited the expected characteristics, with irregularities resulting from the manufacturer's surface treatment (Figure 1). When the F(+) sets were analyzed, dark stains that are responsible for the macroscopic change on titanium surface color were found, which may also be interpreted as imperfections in the oxide layer and the areas were pitting corrosion started, which is a type of corrosion mainly observed in passive metals like titanium, aluminum and stainless steel[4]. This corrosion is characterized by formation of gaps, inside of which there is no re-passivation of the oxide layer by oxygen inhibition, and the establishment of an anodic process[23] (Figure 2).

The fractures by titanium compression are characterized by mixed ruptures, as they present regions with macroscopic plastic deformation. This plastic deformation occurs both in the fragile fracture and in the ductile fracture. The difference is that cracking in the fragile fracture needs minimal
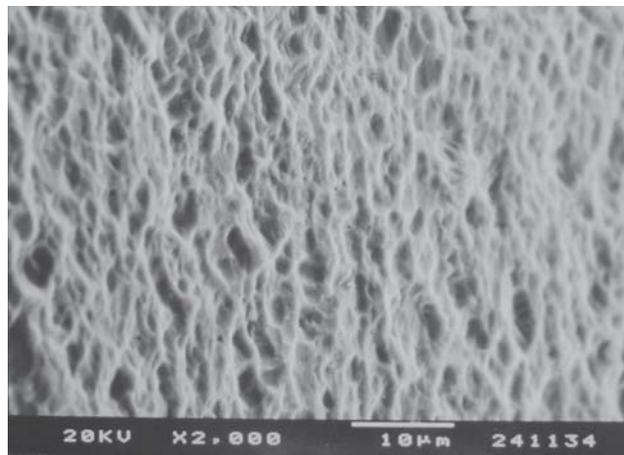

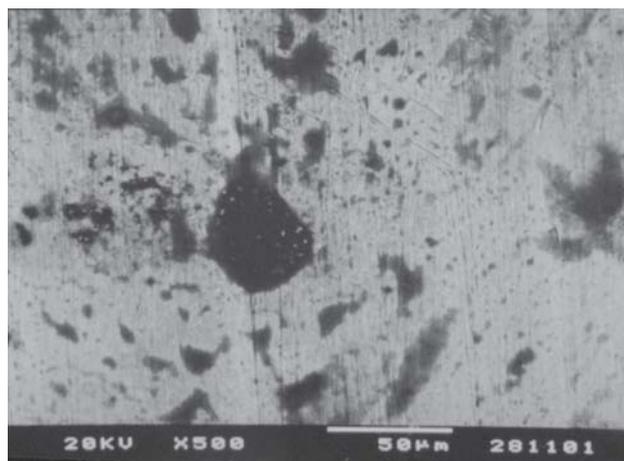**FIGURE 1-** Implant not submitted to the action of fluoride ions F(-) 2000x

FIGURE 2- Implant submitted to the action of fluoride ions F(+). Corrosion by pitting 500x
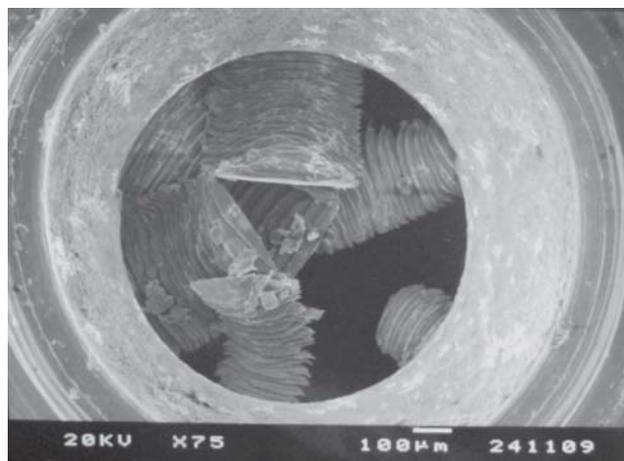

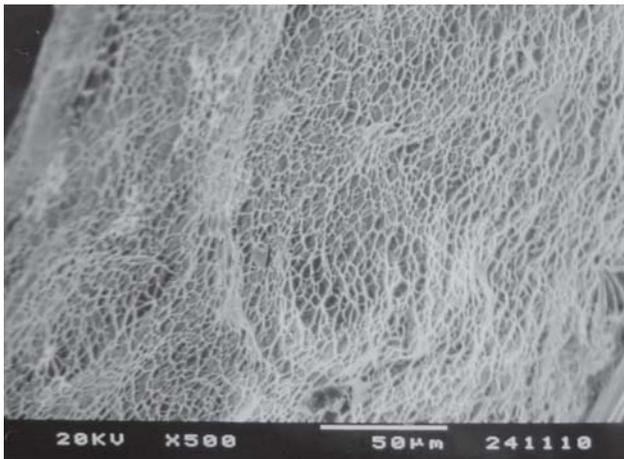**FIGURE 3-** Fracture on the head of screw after mechanical cycling F(+) 75x

plastic deformation to propagate itself, whereas in the ductile fracture this deformation is necessarily greater[20].

The mechanical behavior of the test specimens changed after being submitted to the action of fluoride ions. Their fatigue strength, as previously reported, decreased.
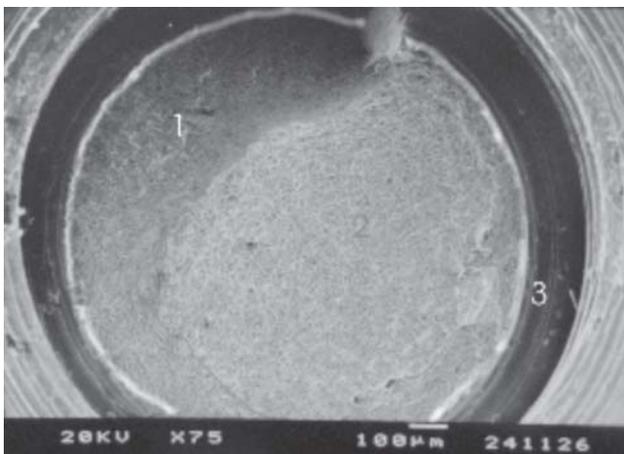
Scanning electron microscopic analysis showed characteristics of mixed fracture, with predominance of fragile fracture, and the screw fracture surfaces showed regions that indicated corrosion (Figure 3), by which crack propagation may have occurred. Therefore, there was influence of the medium, with predominance of fragile fractures (Figure 4).

There is a great deal of controversy about fracture by fatigue due to the variety of mechanisms that determine the rupture of the metal.

Rupture by fatigue, according to Souza[20] (1974), may occur in three stages: 1) crack nucleation, 2) crack propagation and 3) test specimen rupture. The first two stages take up practically all of the test time and, when the crack length reaches a size that leaves the tensioned section relatively small, the remaining portion can no longer resist the load and the rupture occurs suddenly. Figure 5 shows a characteristic fracture by fatigue that presents three zones, zone one is produced by the gradual and progressive crack development, zone two is produced by the sudden fracture, this interface being better observed in zone 3.

A point of concentration of tensions and/or of less resistance in the implant is the level at which the internal threads end, giving way to the beginning of the solid body of the implant (where the screw that holds the pillar is placed), due to the thin implant walls, is the place where fracture most likely occurs[17].

Siirilä and Könönen[19] (1991) showed that the main deterioration factor for titanium, which may be present in the mouth, would be the abrasive action of brushing, but recommended avoiding prolonged exposure to the action of fluoride ions. The results of this study differ from those of these authors, as evidence was found of the modification of the titanium surface after contact with the fluoride ions, as well as of this modification acting on the mechanical performance of the materials. With regard to this aspect, the results of this investigation are in agreement with the findings of Toumelim-Chemla, et al.[22] (1996), who pointed to high concentrations of fluoride ions and prolonged contact, as possible harmful agents to the durability of metal prostheses and rehabilitations with implants, which are present in the oral environment.

Additionally, Reclaru and Meyer[18] (1998) reported that titanium and other dental alloys showed a corrosive process only when the pH dropped below 3.5. This findings differ from those of our study and the study by Nakagawa, et al.[9] (1999) which revealed that titanium could be corroded by the existence of a small amount of NaF if pH was considerably low or, in conditions of high pH if NaF concentration was also considerably high.

FIGURE 4- Mixed fracture with predominance of fragile fracture F(+) 500x

FIGURE 5- Zones of fracture development by fatigue in the screw center F(-) 75x. 1) crack nucleation; 2) crack propagation; 3) test specimen rupture

CONCLUSION

It may be concluded that fluoride ions negatively influenced the resistance to fatigue of implant-abutment sets, and that fracture occurred before reaching the minimum number of cycles established in this study. The scanning electron microscopic analysis showed evidence of pitting corrosion, which confirms the action of fluorides on the surfaces of the studied biomaterial.

ACKNOWLEDGEMENTS

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303

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