CLINICAL ORAL IMPLANTS RESEARCH

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The effect of oral-like environment on dental implants' fatigue performance

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Abstract

Aim and Objectives: The aim of this study was to evaluate the influence of fluid environment mimicking intra-oral conditions on fatigue performance of standard diameter, 3.75-mm implants. Dental implants placed intra-orally are repeatedly submitted to mastication loads in the oral environment, which differ substantially from room-air standard laboratory conditions. Several studies that examined fracture surfaces of intra-orally fractured dental implants have identified corrosion fatigue as the main failure mechanism. Yet, fatigue performance of dental implants has been essentially studied in room air, based on the premise that the implant material is relatively resistant to corrosion in the intra-oral environment.

Material and methods: Thirty-two 3.75-mm titanium alloy implants were tested under cyclic compressive loading. The tests were performed in artificial saliva substitute containing 250 ppm of fluoride. The loading machine stopped running when the implant structure collapsed or when it completed 5×10^6 cycles without apparent failure. The load vs. number of cycles was plotted as curve for biomechanical fatigue analysis (S–N curve). The S–N curve plotted for the artificial saliva test was compared to the curve obtained previously for the same implants tested in a room-air environment. Failure analysis was performed using scanning electron microscopy (SEM).

Results: A comparison of the S–N curves obtained in artificial saliva and in room air showed a considerable difference. The S–N curve obtained in the artificial saliva environment showed a finite life region between 535N and 800N. The transition region was found below 465N, with a probability of survival of 50%, while in room air, the transition region was between 810N and 620N and an infinite life region below 620N was identified.

Conclusions: The results of this study show that environmental conditions adversely affect implants' fatigue performance. This fact should be taken into account when evaluating the mechanical properties of dental implants.

Most dental implants used today to replace missing teeth are made of commercially pure titanium or titanium alloys. The choice of those materials is based on an excellent record of biocompatibility, high corrosion resistance rating combined with high mechanical properties. The outstanding biocompatibility is attributed to the strong surface passivation layer, mainly consisted of TiO_2 (Gonzalez & Mirza-Rosca 1999; Nakagawa et al. 1999; Virtanen et al. 2008; Fleck & Eifler 2010).

Fracture of dental implants or implants' components are relatively uncommon complications; however, once this occurs, the clinician and patient both are faced with a challenging problem. The identification of the factors responsible for intra-oral mechanical failure in dental implants is therefore of high importance. One way to address this problem relies on the use of scanning electron fractographic analysis of *in vivo* failed implants and implant parts.

Choe et al. (2004) examined six fracture surfaces of intra-orally fractured implants. The analysis identified a combination of mechanical fatigue (revealed by fatigue striations) together with the presence of corrosion products. The authors concluded that the implants failed due to corrosion fatigue. Sbordone et al. (2010) performed an SEM fractographic analysis on seven hollow implants that fractured intra-orally. Their analysis pointed out corrosion fatigue as the most likely mechanism for the implants' fracture as well. The authors postulated that the implant's metal surfaces got exposed to oral environment as a result of bone loss, while repeated loading causes the breakdown of the protective surface passivation layer. As a

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result, surface micro-cracks were created, which grew by a fatigue mechanisms finally resulting in the implant's fracture.

However, reporting of *in vitro* implant testing and characterization of the biomechanical strength performance of implant structure under cyclic loading is limited. So far, most published studies were carried out in dry air, with no reference to other environmental conditions (e.g., Huang et al. 2005; Quek et al. 2006; Shemtov-Yona et al. 2012a,b). It may be surmised that the reason for this lack of reference to oral environmental factors in these studies is the high rating of pure titanium or titanium alloys in terms of biocompatibility and strength so that oral environment has not been considered so far as a potentially harmful environment. Also, performing fatigue mechanical testing in laboratory environment is easier to perform compared to "oral-like" conditions.

Fleck & Eifler (2010) reviewed the corrosion fatigue properties of pure titanium and titanium alloys (Ti6Al4V); they showed that pure titanium grade 4, when loaded in Ringer's solution (saline), exhibited a distinct reduction in its fatigue performance when compared to that in room air. By contrast, the fatigue life of Ti6Al4V was not affected by that environment. These authors also pointed out that, under physiologic conditions, implants are not exposed to pure saline solutions, but to protein-containing serum, which can influence the corrosion resistance and the corrosion fatigue behavior. Likewise, Roselino-Ribeiro et al. (2007) assessed the mechanical fatigue properties of pure titanium implants that were immersed in a solution containing 1500 ppm sodium fluoride prior to the mechanical fatigue test. The test results showed how environment, containing fluoride ions, adversely influenced the implant's fatigue strength. However, this research was based on only 10 implant structures, with only one level of applied load, and the fatigue test was carried on for only 10^5 cycles, which is of limited significance and, however, might be indicative of this environmental effect.

From the above-mentioned literature, it appears that corrosion fatigue may be considered as a potential mechanism for mechanical implants failure; nonetheless, the available quantitative and systematic data are still quite scarce, with little or no relevance to the real environment surrounding implants intra-orally.

The aim of this study was to evaluate the influence of fluid environment mimicking intra-oral conditions on fatigue performance of standard diameter, 3.75-mm implants, through the construction of a curve for the biomechanical fatigue analysis (S–N curve) for implants tested in room air compared to oral-like environment. Furthermore, a detailed failure analysis was performed, using scanning electron (SEM) microscopy to identify the "signature" of this failure mechanism.

Material and methods

Tested implants

Thirty-two commercially available dental implants made of Ti6Al4V (Grade 5 ELI) were tested in this study. The experimental units consisted of tapered 13-mm long implants, with an outer diameter 3.75 mm at implant neck connected to a straight 8-mm implant abutment with an abutment screw 7 mm long. Components were tightened to 30 Ncm with a clinical torque driver.

Environment (Artificial saliva)

To mimic the intra-oral environment, a saliva substitute was used (Biotene mouthwash; SmithKline Beecham Ltd, EUCH CQ, Slough, UK). Sodium fluoride was added to the solution to generate a total fluoride concentration of 250 ppm.

Testing machine

Mechanical testing (cyclic) was performed using an MTS servo-hydraulic load frame (MTS system, Minneapolis, MN, USA) with 250 kN load capacity, driven under load control. The instruments setup and sample fixation were as described previously (Shemtov-Yona et al. 2012a,b), to ensure specimen loading at a 30° inclination angle.

Implant tested in the artificial saliva

A plastic cup was prepared to immerse the specimens in the above-described solution (Fig. 1a,b). The cup was open on its upper side to the room environment, thereby allowing the force to be applied to the implant's abutment. The bottom of the cup was sealed by a silicone rubber dam (Hygenic Fiesta Dental Dam, Coltene/Whaledent, Inc. Cuyahoga Falls, OH, USA). This seal kept the solution around the specimens at all time during the cyclic fatigue test. It was changed every time a new implant was tested.

Fatigue testing

Fatigue testing was performed under load control. Five levels of load magnitudes were selected (800N, 716N, 605N, 535N, 465N), according to the static bending strength of the 3.75-mm implant that was measured in a previous quasi-static, monotonic bending strength test (Shemtov-Yona et al. 2012a,b). Here, a vertical load was applied at a rate of 0.4 mm/min until the sample fractured or exhibited a significant amount of (permanent) plastic deformation accompanied by a load drop. Five specimens were quasi-statically tested, and the maximum applied load was recorded. This mean load was found to be of 930N ± 77N.

The loads was directly applied to the implant abutment head as a sinusoidal force, with minimum-to-maximum loading ratio of $R = 0.1$. The test frequencies, chosen to minimize vibrations of the test machine, were in the range of 15–20 Hz. The machine stopped working automatically when the specimen fractured; alternatively, it was terminated when 5×10^6 cycles were exceeded without apparent failure. This limit was arbitrarily fixed as representative of very long-term

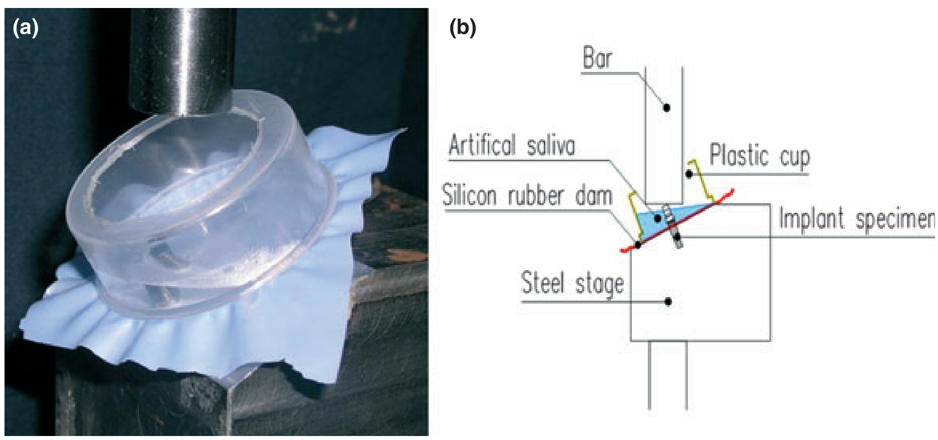

Fig. 1. Fatigue testing in artificial saliva. (a) The picture shows the specimen placed in the plastic cup filled with the saliva substitute. A rubber dam seals the lower part of the cup, while upper side is opened for load application. (b) schematic diagram of the implant abutment setup.

(infinite) service. After each test, the number of cycles and the failure mode of the specimen were recorded. Five to ten specimens were tested for each load magnitude.

Microscopic failure analysis

The fracture surfaces of the failed dental implants were examined using a scanning electron microscope (Phillips XL 30, Eindhoven, the Netherlands). To improve electrical conductivity required for SEM work, the fracture surfaces were coated with a thin layer of gold (sputtering).

Results

Fatigue test

Figure 2 shows the fatigue test results presented as load vs. number of cycles curve (S–N curve) obtained for the 3.75-mm implant in artificial saliva together with the S–N curve in room air (Shemtov-Yona et al. 2012a). The S–N curve is a descriptive method to present and evaluate the implants fatigue performance in the different environments. In this S–N curve, one can distinguish three main regions: a finite life region, which is the load range where all the specimens failed after a finite number of cycles, the transition region, which corresponds to the range of loads range where some of the specimens failed and some reached 5×10^6 cycles without apparent failure, and finally, the infinite life region, which is the load range where none of the specimens failed until 5×10^6 cycles were completed, also called "run-outs."

Table 1 summarizes the three S–N regions identified for the test performed in artificial saliva and compares to data on test performed in a room air.

***Table 1.* The three S–N regions identified for the test performed in artificial saliva and compared to data on test performed in a room air**

Environment S–N regions	Artificial saliva	Room air
Finite life region	>535N	>810N
Transition region	≤465N	620–810N
Infinite life region	Not available	<620N

The S–N curves clearly show the strong effect of the environmental conditions on the implants fatigue performance. For the same load magnitude, the number of cycles to reach failure in artificial saliva is markedly smaller than that obtained in room air. Moreover, in the transition region of room-air experiments, some implants reach an "infinite" life of 5×10^6 cycles, whereas in artificial saliva, the same range of loads causes failure of all the specimens.

Figure 3 plots the probability of fracture (the percentage of failed implant found in the same load magnitude) at each load for the two kinds of environments. It is interesting to note that the two corresponding lines have relatively similar slope; however, this line for the artificial saliva results is skewed to the left (to lower values of applied load). This observation indicates a rather "uniform" action of the environmental wet conditions in the fatigue failure process, independently of the applied load. Stated otherwise, the artificial saliva environment reduces the applicable load at a given percentage of survival by a constant value in the order of 0.2 times the normalized load.

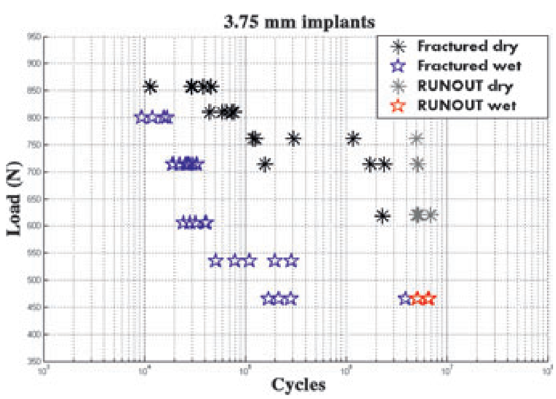

Fig. 2. Load vs. number of cycles in fatigue testing for the 3.75-mm implant in artificial saliva, together with the result obtained in room air. The results obtained for the test in artificial saliva are marked by stars. The results obtained in room air are marked by asterisks. A "Fractured dry" symbol means that the specimen failed after a finite number of cycles in room air. "Fractured wet" symbol means that the specimen failed after a finite number of cycles in artificial saliva. "Run-out" means that the specimen did not fail after 5×10^6 cycles. [Correction added on 8 January 2013, after first online publication: Fig 2 has been replaced with the corrected version: percentages on the right derived from all implants have been removed.]

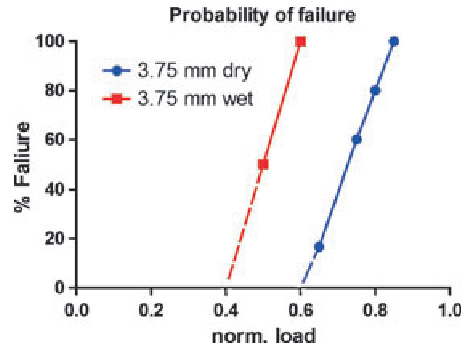

Fig. 3. Probability of failure. The plot compares the fatigue behavior of the 3.75-mm implant in the artificial saliva environment to the room-air environment and describes according to the S–N results the percentage of failed implants at each load. The normalized load refers to the test load divided by the static failure load, to allow for the comparison between the two batches.

Modes of fracture

Two distinct fracture locations were identified. One is at the implant body – neck (six fractured specimens), and the second was at the implant body – thread 2 (22 fractured specimens). In the oral-like environment, the dominant fracture location is implant body – thread 2 (78.5% of the fractured specimens).

Scanning electron microscopy (SEM)

The implant surface was examined using SEM to identify the effect of the artificial saliva environmental condition on the implant. Fig. 4 shows a SEM photograph of the implant surface tested in artificial saliva; it reveals a high degree of homogeneity. The overall texture's topography results from the manufacturing surface treatments. No evidence of corrosion products or localized corrosion attack (e.g., pitting) was observed on the surface of this implants.

Typical macro- and micro-fractographs of fracture surfaces of implants fractured in artificial saliva are shown in Fig. 5. The implants' surfaces of specimens that are shown (both at room air and liquid conditions) were broken at a similar load level to

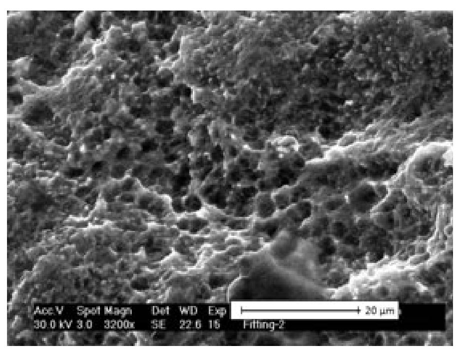

Fig. 4. 3.75-mm implant surface showing a homogeneous surface with no typical corrosion marking.

allow for comparison. This figure clearly shows that the typical characteristics of fatigue fracture, namely transgranular fracture with secondary cracking, can be identified using 1 and 3 K magnifications, irrespective of the testing environment. However, the classical fatigue striations that are characteristic of fatigue in the room-air environment, usually observed at higher magnifications (×13 K), cannot be clearly observed in the artificial saliva case regardless of the magnification.

Discussion

The exposure of dental implants to a liquid medium mimicking oral environmental conditions resulted in a significant reduction in fatigue life compared with that reported for dry laboratory air conditions as seen in the S–N curve.

The term corrosion fatigue indicates damage caused by the combined action of repeated mechanical loading and corrosive attack. The fatigue strength of a material in a corrosive environment will be less than that in room air, meaning that the material will fail at a smaller number of load cycles in the wet environment, while its infinite life range will correspond to smaller loads, when compared to inert conditions. (Hertzberg 1989; Bundy & Zardiackas 2006)

Likewise, Papakyriacou et al. (2000) assessed the corrosion fatigue properties of Ti-6Al-7Nb alloy, which is an α+β titanium alloy, similar to the Ti-6Al-4V of our tested implants, in a physiologic saline solution (0.9% NaCl). Lactic acid was added to the saline solution in order to stimulate similar conditions in the oral cavity. This study showed (as evident in the reported S–N curve) a decrease in fatigue life of the artificial saliva specimens and a significant shift of the curves toward lower stress values in comparison with room air. These findings suggest that the oral environment can be regarded as an aggressive environment, which might hinder the fatigue properties of titanium-made dental implants.

Titanium and its alloys are highly rated as a biocompatible and corrosion resistance material due to its TiO_2 passivation film (Gonzalez & Mirza-Rosca 1999; Nakagawa et al. 1999; Virtanen et al. 2008 & Fleck & Eifler 2010). The strongly adherent dense surface TiO_2 passivation film is known to be a thermodynamically stable. (Virtanen et al. 2008). When damaged, this layer can spontaneously rebuild itself, even in solutions with low oxygen content (Fleck & Eifler 2010). However, dissolution of the passivation film was identified in complex chemical environments, such as solutions containing fluoride ions (Virtanen et al. 2008) and by unique mechanical load transfer such as bending (Niinomi 2007). In an electrochemical study evaluating the corrosive behavior of pure Ti and Ti6Al4V alloys in artificial saliva, it was made clear that the corrosion resistance of Ti alloys is influenced by the saliva PH, in which acidic saliva aggravated the breakage of the passivation layer (Barão et al. 2011).

When evaluating the chemical characterizations of the oral environment, one can conclude that the oral environment is aggressive (corrosive) for such dental implants made of Ti alloys. During the day, the pH of the saliva and fluoride concentration varies in areas around dental implants, influenced by the types of food consumed during the day, oral hygiene habits and oral hygiene regimen that is being used by the patient (Nikolopoulou 2006). Plaque, soft tissues, calculus and tooth structure itself can be regarded as fluoride intra-oral reservoir, which maintain a constant presence of fluoride ions (Yardeni et al. 1963; Campus et al. 2003).

The fractographic analysis revealed that the identification of corrosion fatigue failure mechanism, mostly for the purpose of a failure analysis of *in vivo* failures, might not be straightforward. This is because corrosion products, which are the obvious characterization of the environmental effect, might not be present. This issue will be addressed in detail in a forthcoming article.

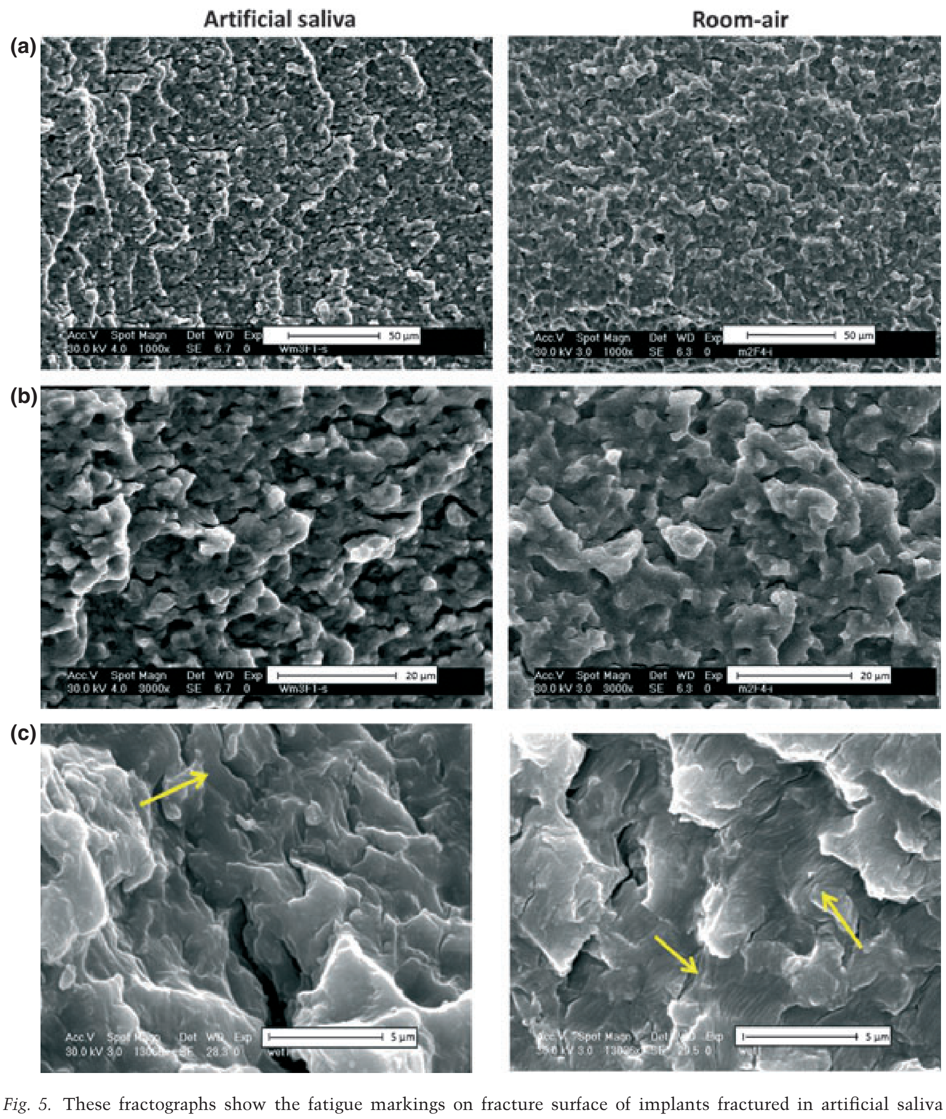

Fig. 5. These fractographs show the fatigue markings on fracture surface of implants fractured in artificial saliva and in room air using magnification of 1 K (5A), 3 K (5B) and 13 K (5C). In both the artificial saliva and room air, transgranular fracture with secondary cracking is visible. In the 13 K magnification, clear fatigue striations in room air are visible (two yellow arrows), while only faint possible striations are seen in the artificial saliva (yellow arrow).

The examination of the implants' surfaces did not reveal any evidence of localized corrosion attack, such as pitting. Such evidence would mean that the time (number of cycles) required to initiate a crack is reduced. However, in the absence of such evidence, it appears that the shorter fatigue life in the saliva-like environment is the result of accelerated crack growth (rather than initiation time) in this environment where metal dissolution might be promoted by the high local stresses at the crack tip.

According to the recommendations of the ISO standard for dynamic fatigue testing of dental implants (ISO 14801 2003), the tests shall be carried out in water, normal saline or physiologic medium, and testing is limited to at least two specimens until failure or 2 million cycles reached. (Lee et al. 2009) (ISO 14801 2003). The results of the present study tend to suggest that the ISO fatigue protocol might not be ideal for this purpose.

Conclusions

The results of this study show that artificial saliva acts as an aggressive environment for dental implant fatigue performance. Transgranular fracture and secondary parallel microcracks are the main fracture micro-mechanisms, which indicate fatigue as the failure mode. Those can be identified at relatively low SEM magnifications. The classical fatigue striations cannot be clearly observed. It is recommended to include testing in artificial saliva into the existing requirements for implants testing in order to evaluate the fatigue performance of dental implants in conditions that mimic better the oral environment.

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