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Research Paper

Fatigue failure of dental implants in simulated intraoral media

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ABSTRACT

Metallic dental implants are exposed to various intraoral environments and repetitive loads during service. Relatively few studies have systematically addressed the potential influence of the environment on the mechanical integrity of the implants, which is therefore the subject of this study.

Four media (groups) were selected for room temperature testing, namely dry air, saliva substitute, same with 250 ppm of fluoride, and saline solution (0.9%). Monolithic Ti–6Al–4V implants were loaded until fracture, using random spectrum loading.

The study reveals that the only aggressive medium of all is the saline solution, as it shortens significantly the spectrum fatigue life of the implants. The quantitative scanning electron fractographic analysis indicates that all the tested implants grew fatigue cracks of similar lengths prior to catastrophic fracture. However, the average crack growth rate in the saline medium was found to largely exceed that in other media, suggesting a decreased fracture toughness.

The notion of a characteristic timescale for environmental degradation was proposed to explain the results of our spectrum tests that blend randomly low and high cycle fatigue.

Random spectrum fatigue testing is powerful technique to assess and compare the mechanical performance of dental implants for various designs and/or environments.

1. Introduction

The long term survival rate of implant supported FDP's (Fixed denture prosthesis) is of 95.6% after 5 years and 93.1% after 10 years. Along with this very high survival rate, complications in implants therapy are quite frequent, with 33.6% of the patients experiencing complications in implant therapy after 5 years (Pjetursson et al., 2012).

Complications in implant therapy are mainly divided into biological, that relate to biological processes affecting the supporting tissue of the implants (bone and surrounding mucosa), and technical or mechanical complications that include mechanical damage of the implants, abutments, and/or the supra-structures (Pjetursson et al., 2012; Papaspyridakos et al., 2012).

Mechanical complications are due to time dependent failure mechanisms, with higher complication rates for longer service time (Pjetursson et al., 2012; Dhima et al., 2014; Shemtov-Yona and Rittel, 2015). In addition, compared to biological complications, mechanical complications occur significantly later and more frequently than biological ones, some of them with limited treatment options.

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Thus, in order to prevent future mechanical complications, it is important to identify the relevant physical and mechanical causes. By examining the fracture surfaces of retrieved fracture implants and their components, it can be clearly stated that metal fatigue (Suresh, 1994) is the main failure mechanism (Morgan et al., 1993; Yokoyama et al., 2002; Choe et al., 2004; Sbordone et al., 2010; Shemtov-Yona and Rittel, 2014).

In order to evaluate the implants' mechanical reliability and their long term mechanical stability, in-vitro test should ideally be performed that can simulate the implant's service conditions. Such tests are practically impossible, mostly because of the inherent difficulty in defining and characterizing the implants' environment (Fleck and Eifler, 2010; Antunes and Lopes de Oliveira, 2012).

This environment can be divided into two main categories. The first is the mechanical environment. Implants are load-bearing devices aimed to function under the complex nature of mastication loads. Prostheses supported by dental implants are subjected to various forces, and moments, all transmitted to the implant body and its components. The force applied to an implant is extremely variable, and its magnitude depends on the patients' characteristics (age, gender, oral habits, etc.), type of prosthesis (single crown, overdenture, full partial denture (FPD), cantilever etc.), number and position of the implants, as well as type of food consumed (carrots, meat etc.) The reported magnitude of the reported bite/mastication loads ranges everywhere from 100 to 2400 [N] (Misch, 2008; Brunski et al., 2000; Gibbs et al., 1986).

The second category is the nature of the biological/chemical environment. This environment is extremely complex as the implants are exposed to a (corrosive) medium with different electrolyte concentrations and pH. The typical intraoral fluid medium contains enzymes, proteins and cells. The chemical environment is not only variable in its absolute content, but it also varies with its location around the implant. For example, when the implant's threads get exposed to the oral environment due to bone loss (perimplantitis), the implant's body might get exposed to saliva, where food contents (solids and fluids) and temperature are constantly varying.

Most dental implants today are made from commercially pure titanium (CP-Ti grade 2–4), or from titanium alloy Ti–6Al–4V ELI (extra low interstitial) (Misch, 2008; Van Noort, 1987; Elias et al., 2008). This material selection is based on the established biocompatibility and corrosion resistance, that are attributed to the native surface oxide (TiO_2) layer, 2–10 nm thick (Fleck and Eifler, 2010; Antunes and Lopes de Oliveira, 2012; Elias et al., 2008).

The fatigue properties of structures made of CP-Ti and titanium alloy, like dental implants, are strongly governed by the joint influence of the implants' environment and load transfer during mastication. In order to avoid damage caused by fatigue, one must consider all these parameters when designing a structure which will function without failure in the human body over long time durations.

The deterioration of fatigue properties in engineering materials can be caused by an external medium in the form of a solid, liquid or gas. While the most common generic term for a chemically caused attack of a material is "corrosion", the latter becomes more specific when it comes to a combined loading with surrounding atmosphere. The deterioration of mechanical properties of alloys resulting from their environment is commonly referred to as environmental assisted cracking (EAC). Corrosion fatigue is another term which is commonly used to denote the damage and failure of a material under the combined action of cyclic stresses and any embrittling medium, mostly in the context of aqueous environments.

The corrosion fatigue behavior of CP-Ti and Ti–6Al–4V is markedly affected by the metal's environment. CP-Ti grade 4, when loaded in Ringer's solution, exhibited a clear degradation of its fatigue performance when compared to that in room-air. By contrast, the fatigue life of Ti–6Al–4V was not affected by that environment (Fleck and Eifler, 2010).Yet, one must remember that under physiological 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.

Moreover, despite the titanium's passive oxide film, highly acidic solutions with high hydrogen content were shown to directly cause a reduction of the metal fracture toughness (K_{IEAC}), a measure of the mechanical threshold for fracture to occur under corrosive conditions (Suresh, 1994; Hertzberg, 1989). In addition an increase in crack propagation rates were reported (Kim, 2006; Tal-Gutelmacher and Eliezer, 2004).

Papakyriacou et al. (2000) assessed the corrosion-fatigue properties of CP-Ti and Ti–6Al–7Nb alloy (an alpha-beta titanium alloy, similar to the Ti–6Al–4V used in dental implants), in a physiologic saline solution (0.9% NaCl). Lactic acid was added to the saline solution in order to stimulate the aggressive environment that might be generated in the oral cavity specifically related to the pH. This study showed (as evident in the S–N (stress–number of cycles) curve that was constructed) a decrease in fatigue lives of both metals tested in the simulated environment, and a significant shift of the curves towards lower stress values in comparison to room-air. These findings suggest that the oral environment can be regarded as an aggressive environment that might degrade the fatigue properties of CP-Ti and titanium alloy alike.

Evaluation of the corrosion-fatigue behavior of CP-Ti and Ti–6Al–4V in saliva-like environment is seldom addressed in the literature. Shemtov-Yona et al. (2014) showed, by means of S–N curves, that saliva substitute with the addition of 250 ppm fluoride acts as an aggressive environment for commercial dental implant fatigue performance. The results obtained showed a significant reduction of (high cycle) fatigue life compared with that reported for dry laboratory-air. The analysis of the test results suggested that the shorter fatigue life in the saliva-like environment is the result of accelerated crack growth (rather than initiation time) in this environment.

All the aforementioned studies were based on constant load cyclic testing (S–N curves) or crack-growth rate measurements, which are the most common accepted test method to assess the structural response when repeated loads are anticipated. Therefore, while such curves clearly revealed trends, the quantitative effect of the environment of the fatigue performance could not be clearly assessed.

The aim of this paper is to evaluate the dental implants reliability and long term fatigue performance using a functional fatigue testing in selected environments. For that purpose, random spectrum loading is applied, as a more "natural" type of loading that mimics to some extent the mastication dynamics. Three different media were selected, that are integral part of the actual chemical environment sustained by implants, namely: 0.9% saline solution, saliva substitute, and same with 250 ppm fluoride.

2. Materials and methods

2.1. Tested implants

The investigated dental implants are commercial Ti–6Al–4V, 3.6 mm diameter, 11.5 mm length monolithic implants, manufactured by "Sigdent Dental Implants" (Israel). Monolithic implants were selected in order reduce the multiplicity of potential failure sites in the different components, and to focus solely on the effect of the environment on the implant failure. A representative implant is shown in Fig. 1. As mentioned, the implant is monolithic and consists of two main parts, the abutment and the threaded part (body). The exact surface roughening treatment was not disclosed by the manufacturer, except for the fact the implants underwent grit-blasting and etching, a fact that was later confirmed during scanning electron examination of the implants' surfaces.

2.2. Mechanical tests

2.2.1. Testing rig and setup

The testing rig and setup were previously described by (Shemtov-Yona and Rittel (2016), so that they will only be briefly described here. Mechanical testing (monotonic and cyclic) was performed using an Instron (model 1342) servo-hydraulic machine, operated under compression load control. A programmable controller (Shimadzu 4830) was used to drive the loading apparatus. The Instron machine was equipped with a load-cell of 3000 N full capacity.

The instruments setup and implant fixation block were those described previously in Shemtov-Yona and Rittel (2016). The specimens were flush mounted such as to leave only the abutment protruding with a 30° inclination angle with respect to the applied load.

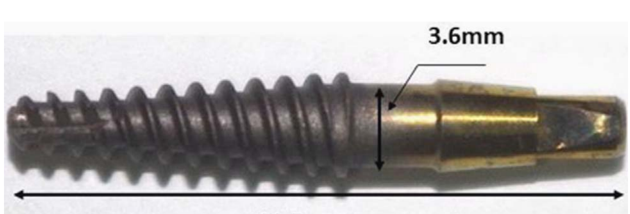

Fig. 1 – Tested implant.

2.2.2. Fatigue tests in liquid oral-like media

In order to mimic as much as the possible the chemical intra-oral environment, fatigue tests were performed at room temperature in 4 different media, namely:

1. Saline −0.9% Sodium-Chloride (NaCl).
2. Protein-free saliva substitute (Biotene mouthwash, SmithKline Beecham Ltd, EUCH CQ, Slough, UK). The listed ingredients of this substitute are: Purified water, glycerin, xylitol, sorbitol, propylene glycol, poloxamer 407, sodium benzoate, hydroxyethyl cellulose, methylparaben, propylparaben, flavor, sodium phosphate and disodium phosphate.
3. Saliva substitute with 250 ppm sodium fluoride.
4. Dry – room air environment.

All three media had a neutral pH in purpose, to eliminate a possible effect of pH which would have added one variable to the research.

2.2.3. Random spectrum loading

To evaluate the functional fatigue performance of dental implants, the applied loads consisted of random spectrum loading, aimed at mimicking the mastication dynamics as a more "natural" alternative to constant load cyclic loading.

Random spectrum loading, was designed as follows. The signal was comprised of a succession of sinusoidal blocks, each of which consists of a repetition of negative (compression) half-cycles. Each block was randomly assigned a number of 10–100 such cycles. The frequency of the block was randomly assigned to vary between 1–3 Hz. The maximum duration of the random block was determined by the frequency of the specific block and the number of cycles it contains. The amplitude of the signal was randomly assigned to vary between 0 and 1. The value of 1 represents the maximum load that may be applied to the implant. A maximum representative load was selected to be 1000 N, according to the previously measured quasi-static bending strength of the implant (1136±57.1 N), as in Shemtov-Yona and Rittel (2016). Pauses were randomly applied during the spectrum, with a probability of occurrence of 0.1. In that case, the block was randomly assigned a 0 amplitude, and its duration determined the duration of the pause. Pauses are supposed to represent "dead times" during which the implant is not actively loaded, but still subjected to the liquid environment.

A total of 10 implants were tested for each medium, when all of which underwent identical spectrum loading.

The outcome of the test was the total time to fracture, expressed in seconds, including the pauses. This parameter cannot be directly converted into cycles due to the random nature of the applied loads. A typical random spectrum is shown in Fig. 2 as a time series.

2.3. Microstructural and mechanical characterization

2.3.1. Microstructure assessment

Metallographic longitudinal and cross sections were prepared of a new implant. After embedding and polishing the sectioned specimens using conventional techniques, the latter were etched using Kroll's reagent for a few seconds.

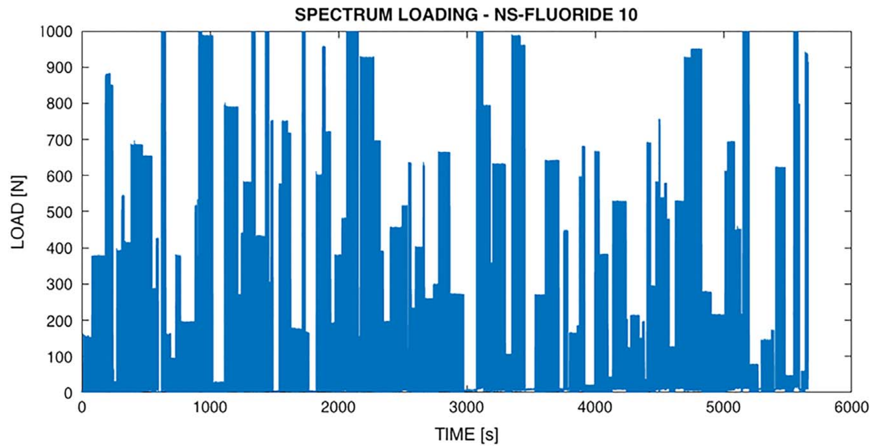

Fig. 2 – A typical random spectrum loading.

Table 1 – Time to fracture for the different tested groups.

Tested medium	N	Mean (s)	Std	Median	p value[a]
Dry	10	3411	1513	3220	0.0045
Saline	10	2266	695	2093	
Saliva	10	4723	1352	5597	
Saliva and fluoride	10	3834	1784	3258	

[a] Kruskal–Wallis test.

2.3.2. *Fractographic analysis*

The fracture surfaces of the failed dental implants were examined using a scanning electron microscope (Phillips XL 30, Eindhoven, Netherlands). The fatigue crack length was measured almost for each tested implant, and an average crack propagation rate was calculated by dividing the crack length (a) obtained from the fracture surface by the total time to failure (t). Here, the final overload duration was neglected.

3. Results

3.1. *Mechanical tests*

Table 1 presents the random test results. For each tested medium, the mean time to failure, standard deviation and the median are presented.

The random test result shows a statistical difference in time to failure between the tested media ($p<0.05$).

The mean time to failure was highest in saliva substitute and lowest in saline medium. The distribution of time to failure according to the tested group is shown in Fig. 3.

In order to test this result, a Wilcoxon two-Sample test was carried out for all possible pairs of groups.

Table 2 shows the p-values that were obtained from the Wilcoxon two-Sample test.

Table 2 indicates a significant difference between saline and dry media ($p=0.0257$), between saline and saliva ($p=0.0010$), and between saline and saliva with fluoride ($p=0.0376$). No other statistical difference was found between the other groups. In other words, the saline medium is the only significantly different medium.

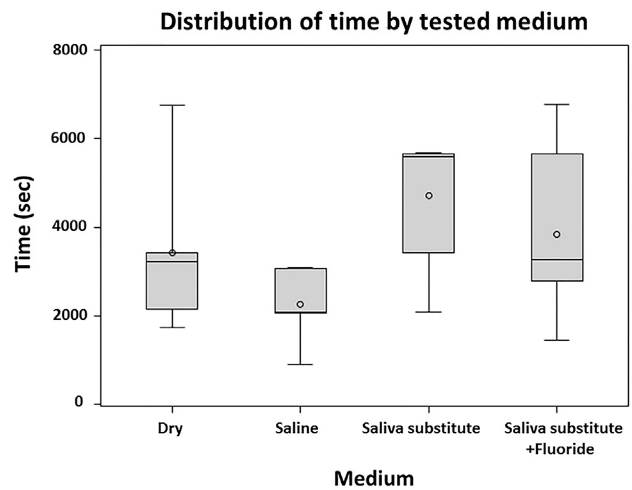

Fig. 3 – The distribution of time to failure according to the tested group. For each medium the colored square indicate the values between the 25 and 75 percentiles. The line inside the square is the median and the circle indicates the mean value. The two extreme lines are the maximum and minimum values obtained.

Table 2 – Results of Wilcoxon two-Sample tests for the lifetime of the tested implants in different media. The boldfaced p values indicate the media that are significantly different. In this case, the saline medium is the only different medium.

	Saline	Saliva substitute	Saliva substitute and fluoride
Dry	**0.0257**	0.0587	0.7913
Saline	–	**0.0010**	**0.0376**
Saliva substitute	–	–	0.2730

3.2. *Microstructural and mechanical characterization*

3.2.1. *Microstructure assessment*

Typical micrographs are shown in Fig. 4(a) and (b) for the cross sections and longitudinal and, respectively. The microstructure

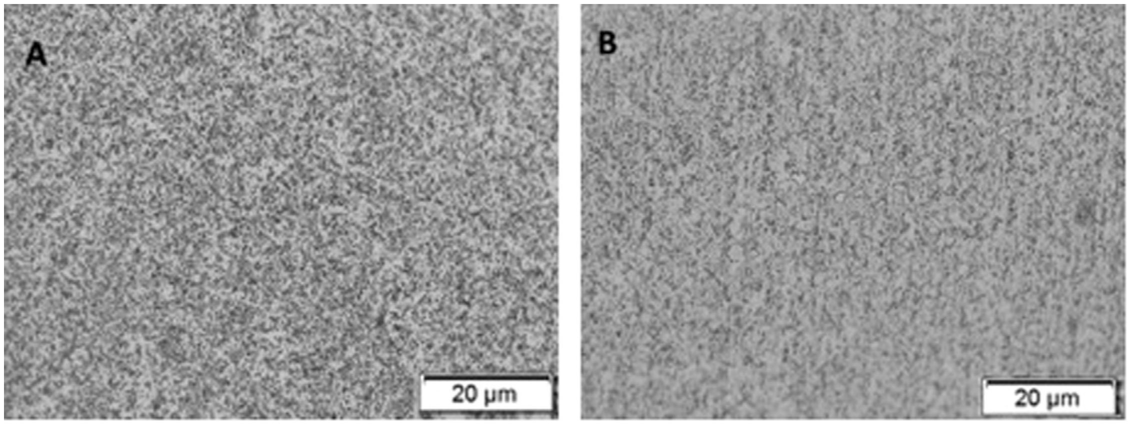

Fig. 4 – Implant's metal microstructures A) cross sections B) longitudinal.

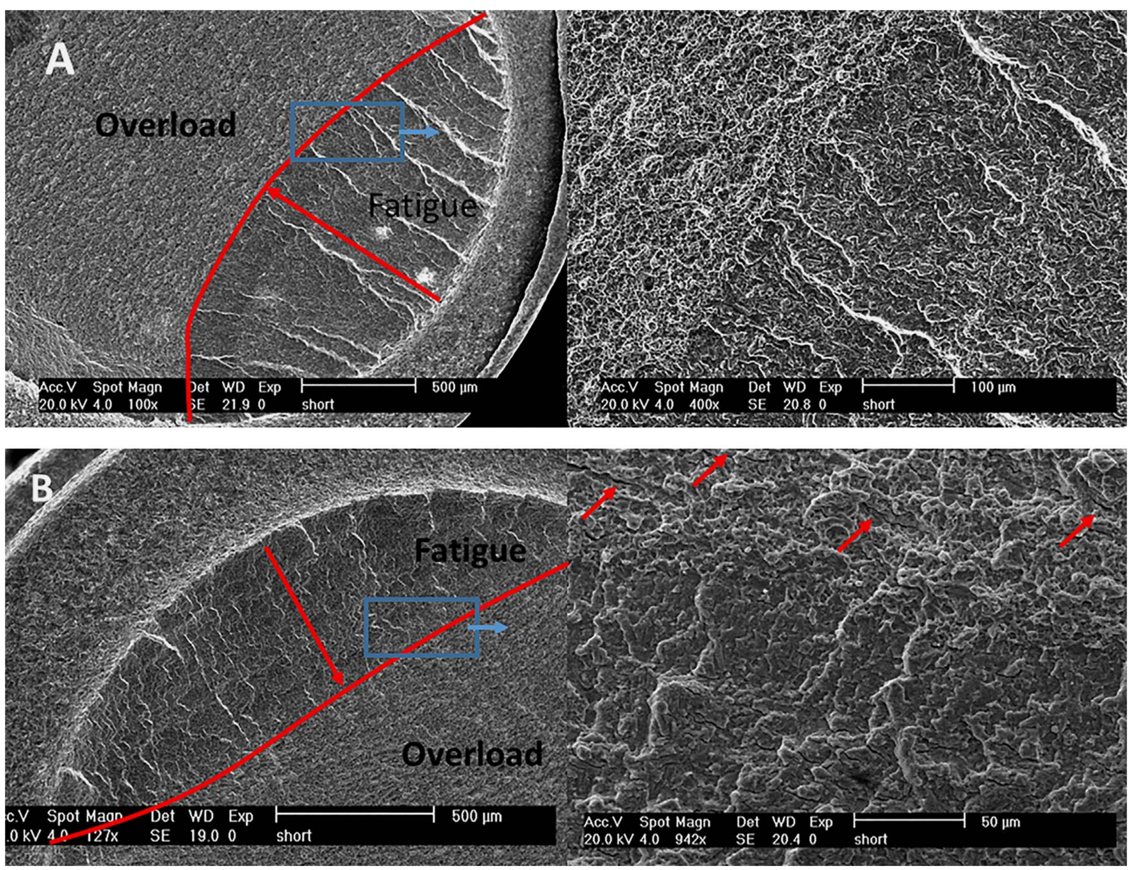

Fig. 5 – Fracture surfaces of specimens tested in saline (A) and in saliva (B). The fracture surfaces are comprised of the fatigue and the overload regions. The fatigue crack length is marked with a red arrow. The blue rectangle indicates the transition between fatigue and overload regions. Pictures on the right panels are magnifications of the same area. Monotonic modes that consist of overload dimples and secondary cracks together can be identified only on the saliva fracture surface (red arrows). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

is characteristic of the mill-annealed condition. I addition, one can note the slightly elongated structure in the longitudinal specimen as opposed to that of the cross section. This indicates a certain degree of anisotropy resulting from the forming process of the raw material (probably a rod). This issue will not be developed further as it is not deemed to be central to the results reported herein.

3.2.2. *Fractographic analysis*

A first observation is that all the specimens failed by fracture at the same location, namely in the second thread, similar to the failure pattern reported in Shemtov-Yona and Rittel (2016) for room air tests. This similarity illustrates the insensitivity of the fracture location to the composition of the test medium.

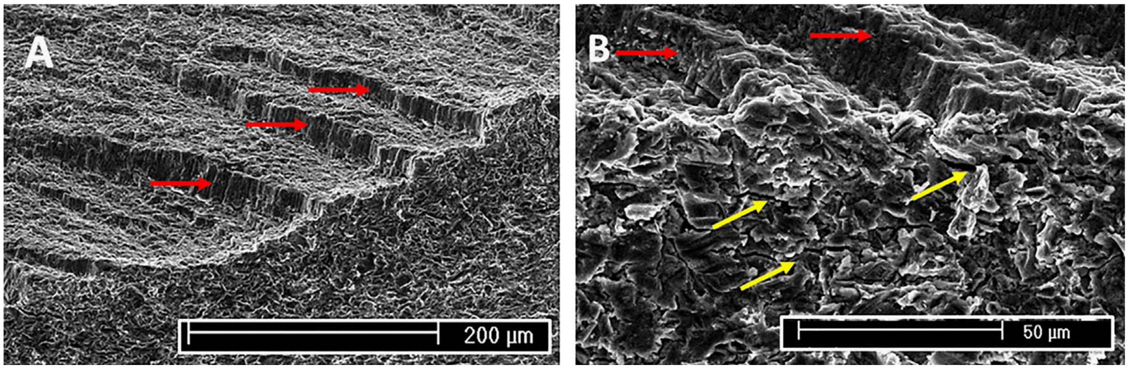

Fig. 6 – Fatigue crack initiation location starting from the implant surface A: tear ridges as cracks origin (red arrows) B: Secondary cracks on the implants surface parallel to the fracture surface (yellow arrows), indicating multiple crack initiation on the implant's surface. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Fracture surfaces of specimens tested in saline (a) and in saliva substitute (b) are shown in Fig. 5. The fracture surfaces are comprised of the fatigue and the overload regions. In both cases, the fatigue cracks have relatively similar lengths. Looking at a transition area between fatigue and overload regions (Blue square, pictures on the right panels) at higher magnifications reveals further the effect of the test medium on the fracture surface. For the saline solution, a clear and sharp transition exists between the fatigue markings with secondary cracks and fatigue striations, and overload markings (dimples). For all the other media (air, saliva substitute, and substitute with fluoride) monotonic modes that consist of overload dimples and secondary cracks together can be identified as a "transition" area between the fatigue and overload regions.

Fatigue crack initiation location starting from the implant surface is shown of Fig. 6. Clear tear ridges starting from the surface of the implant that indicate multiple crack initiation sites are observed (Milella, 2013; Kerlines and Phillips, 2006). These tear ridges are extremely marked and long. They extend through the whole length of the fatigue area for the saline fracture surface. For all the other media, those ridges fade out gradually as the crack propagates towards final overload failure.

Secondary cracking, perpendicular to the main fracture plane, can be identified at higher magnifications, (Fig. 7) which are the dominant indication of fatigue crack growth process in this titanium alloy. At higher magnifications, fine striations can be observed on both tested groups.

3.2.3. *Quantitative fractographic results*

Table 3 presents the mean value and the range of fatigue crack lengths for each medium, as measured on the SEM fracture surfaces. The rightmost two columns show the calculated crack growth rate and its range.

Crack lengths that were measured from the fracture surfaces were all of the same length scale. The Kruskal–Wallis statistical test did not show a statistical difference between the groups (p.value$>$0.05). By sharp contrast, the average calculated crack growth rate showed a statistical difference ($p=0.0044$) between the tested media. The distribution of crack length according to the tested group (A), and of average crack growth rate (B) are shown in Fig. 8.

In order to further refine this result, paired t-tests were carried out for all possible pairs of groups as to the average crack growth rate.

Table 4 shows the p-values that were obtained. There seems to be again a significant difference between saline and dry media ($p=0.0114$), between saline and saliva substitute ($p=0.001$), and between saline and saliva substitute and fluoride ($p=0.01$). No other statistical difference was found between the groups. Saliva substitute, saliva substitute and fluoride, and dry media did not show a statistical difference between them.

4. Discussion

The results of this research have systematically characterized the effect of different environments on dental implants' mechanical reliability. The study relies on a new testing method for dental implant fatigue performance, simulating its working conditions through a combination of a potentially aggressive environment and random spectrum mastication-like loading. The results presented herein are not limited to the implants themselves, but apply more generally to the material they are made of, namely Ti–6Al–4V. It is also worth noting that, contrary to constant load amplitude testing, for which the measured life varies for each load level, the spectrum tests are characterized by a unique value of the fatigue life and its standard deviation.

The first important outcome of this work is that under spectrum loading conditions, the only medium that was found to be aggressive is the saline. Aggressive is to be understood here as a medium causing a noticeable reduction of the implant's fatigue life. The time scale of the random spectrum tests carried out in the present study is relatively short (between 1000–6700 s). Pauses were inserted into the spectrum, not only to simulate mastication habits, but also to enable the time-dependent mechanisms, if any, to operate. The time scale of the present experiment blends randomly low and high cycle fatigue testing.

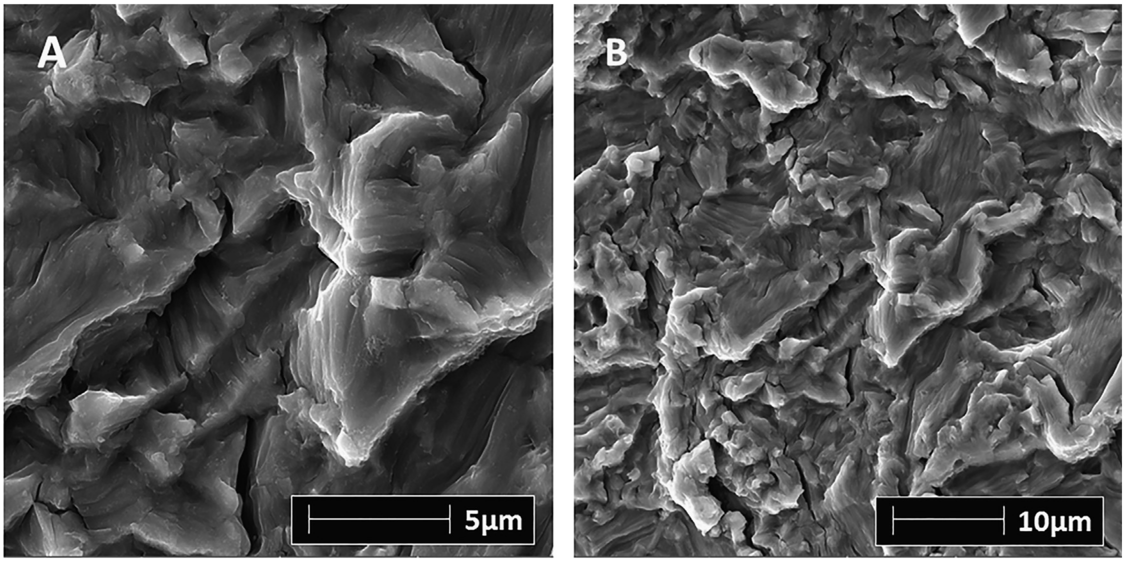

Fig. 7 – Fatigue markings on an implant tested in saline A: fatigue striations B: secondary cracking, perpendicular to the main fracture plane and fatigue striations.

Table 3 – The mean value and the range of fatigue crack lengths for each medium. The rightmost two columns show the calculated crack growth rate and its range.

Medium	mean a [µm]	Range [µm]	p-value	mean a/t [µm/s]	a/t range [µm/s]	p-value
Dry	645±154	491–799	0.7531	0.20±0.06	0.14–0.26	0.0044
Saline	726±69	657–795		0.31±0.08	0.23–0.39	
Saliva substitute	678±128	588–852		0.18±0.07	0.11–0.25	
Substitute+fluoride	720±132	550–806		0.17±0.07	0.10–0.24	

The seemingly conflicting results, as to the influence of the saline medium with respect to the other media, might suggest that different, environment-related characteristic time scales exist for environmentally assisted cracking of titanium and alloys. Such a notion has been mentioned earlier by Dawson and Pelloux (1974) who reported a frequency (thus time) dependent behavior of titanium alloy. The study showed that, depending on the specific environment tested, among which distilled water and 3.5%NaCl, a clear increase in fatigue crack growth rate was observed for the same stress intensity level (ΔK) with respect to room air conditions. This dependence is further complicated in the current context by the fact that the spectrum comprises a random combination and sequence of load levels, each of them having a different influence on the passive oxide layer. Translated into a physical mechanism, the rebuilding of the oxide protective layer competes with its cyclic mechanical attrition. This mechanism was not directly and quantitatively investigated here.

It can nevertheless be surmised that the saline medium, which was previously reported to be harmless in the high cycle fatigue regime, becomes quite aggressive when low cycle fatigue is increasingly involved. Likewise, the saliva substitute-fluoride medium, that seems to be innocuous in the present low cycle fatigue tests, has indeed been reported to be deleterious in the high cycle fatigue regime (Shemtov-Yona et al., 2014). Such observations point out again to the existence of a characteristic time scale for each combination of medium and load level, which should be further investigated to delineate the domain in which an apparently innocuous medium can turn into potentially aggressive for the implant.

The next important observations are related to the examination of the fracture surface and the quantitative data that could be extracted. It was found that, irrespective of the test medium, the average fatigue crack length was identical (within statistical limits) for all tested specimens. While air, saliva substitute, and saliva substitute with fluoride were not found to differ statistically in terms of lifetime, the saline solution medium was found to significantly shorten the spectrum fatigue life of the implant. Translated into an "average" crack growth rate, this result shows (with statistical significance) that the fatigue cracks of our tests grew faster in the saline solution medium.

McEvily and Wei (Suresh, 1994) presented a simple classification for corrosion fatigue behavior of metals in terms of K_{max} and K_{Iscc}, the maximum stress intensity factor and the stress corrosion cracking fracture toughness, respectively. According to those authors, when $K_{max}<K_{Iscc}$ the environment has no effect on fracture, but if $K_{max}>K_{Iscc}$, the crack growth rate increases markedly towards final fracture. This suggestion is corroborated by the fractographic results presented earlier. For all media except the saline solution, for the final stages of fatigue crack propagation where K_{max} is close to K_{IC} (fracture toughness), we identified monotonic modes that consist of overload dimples and secondary cracks. By

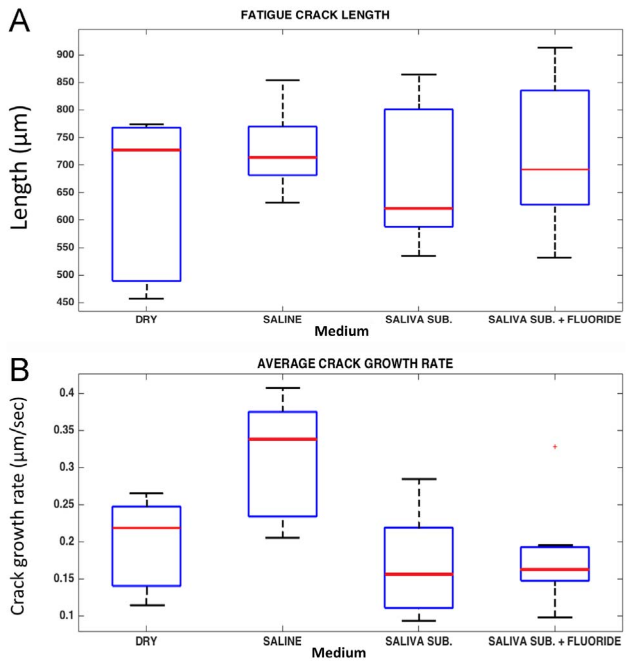

Fig. 8 – The distribution of crack length (A) and average crack growth rate (B) according to the tested group. For each medium the square indicate the values between the 25 and 75 percentiles. The line inside the square is the mean. The two extreme lines are the maximum and minimum values obtained.

Table 4 – Results of *t* tests carried out for the average crack growth rate between the different tested media. The boldfaced *p* values indicate the media that are significantly different. In this case, the saline medium is the only different medium.

	Saline	Saliva substitute	Saliva substitute and fluoride
Dry	**0.0114**	0.4751	0.6558
Saline	–	**0.001**	**0.001**
Saliva substitute	–	–	0.748

sharp contrast, the saline solution fatigued implants exhibited only steady fatigue crack growth without those monotonic modes.

One should note again that all the specimens tested here underwent the same loading spectrum and all had comparable fatigue crack lengths. Therefore, the specimens that failed at later stage actually accumulated more fatigue damage. This leads to the conclusion that Ti–6Al–4V in saline solution medium failed at $K_{ISCC} < K_{IC}$. Likewise, in other media, failure occurred most likely at K_{IC}. This conclusion applies strictly to the current random spectrum tests.

As mentioned in the introduction, the available information on the environmental behavior of dental implants was so far limited to constant amplitude cyclic loading or to crack propagation rate measurements. Extending such information to functional conditions is not straightforward but quite important to the mechanical reliability of the dental implants.

While from a purely clinical point of view, the media examined in this work can be considered as "model media" with respect to a highly complex reality (Doi et al., 2016), it is believed that the current methodology has clearly shown its potential for providing rapid and accurate estimates of the degree of aggressiveness of any medium of interest, without resorting to extensive statistical analyses applied to concept of fatigue strength.

Overall, the present work which addresses a mechanical issue of clinical relevance, illustrates the need for combining engineering and clinical sciences to achieve the sought after progress.

5. Conclusions

Random spectrum loading is a powerful technique to investigate the effect of intraoral media on the mechanical reliability of dental implants.

Of the 4 investigated media, the saline solution was the only aggressive medium observed for the reported tests. This medium operates for short duration random loading tests.

Saline solution medium accelerates the fatigue crack-growth rate, thereby apparently reducing the fracture toughness of Ti–6Al–4V to a lower environmentally assisted value.

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