



1 Article

# 2 Fracture and Fatigue of CP Titanium Narrow Dental

- Implants New Trends in Order to Improve the
- 4 Mechanical Response.
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Abstract: Sixty-four fractured (Ti-Cp) narrow dental implants (NDIs) with similar macrogeometry and connection designs were studied after different implantation times in humans in order to determine their reliability and to evaluate the causes of the fracture. These NDIs were compared with other similar implants, made with alloyed titanium with 15%Zr and with 12% strained titanium. Original implants were tested under fatigue conditions, simulating the tri-axial loads in mouth by means of Bionix hydraulic test machine. Fractography has been studied by Field Emission Scanning electron microscopy (FSEM). The results showed that Ti-cp NDI exhibits a low strength for the mechanical cycling and the Ti alloyed and strained titanium increase the mechanical strength guaranteed the long term mechanical behavior. NDIs fractured were due to fatigue and in some cases initiated by the presence of cracks in the original NDIs produce a fast propagation to fracture. These cracks were attributed to the plastic deformation during machining were found to be exalted due to acid etching in the passivation process.

**Keywords:** narrow dental implants; titanium; fatigue; fracture; plastic deformation

#### Glossary of abbreviations:

29	HVN:	Hardness Vickers Number
30	JDE®:	Bone level dental implant JDE (JDental Care, Modena, Itlay)
31	NDI:	Narrow Dental Implants
32	Ra:	Average surface roughness (µm)
33	Roxolid®:	Bone level implant ROXOLID (Straumann AGR, Basel, Switzerland)
34	Ti-cp:	Commercially-pure titanium
35	VEGA®:	Bone level dental implant VEGA (Klockner, Barcelona, Spain).
36	σ0.2:	Yield Stress (MPa)
37	σmax:	Maximum strength (MPa)
38	ε:	Strain-to-fracture (%)

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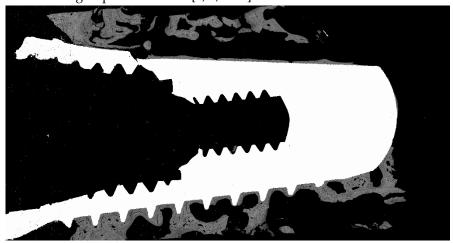
## 1. Introduction

Sometimes, the available bone tissue is insufficient to place regular diameter dental implants and it is necessary new surgical techniques for increasing and regenerating the hard tissue. The use

of NDIs is an alternative treatment because these allow dental restorations in areas with limited prosthetic space or can be inserted in places that would otherwise require grafting techniques [1-4].

Mechanical strength of Ti-cp is limited and that is used in NDIs increases the risk of fracture of the Ti dental implant [5-8]. In Figure 1, it can be observed the fracture of a osseointegrated NDI after 17 months of implantation in a patient. The small diameter and reduced wall-thickness, and the lower material bulk hamper the long-term survivor. Besides, the bending of the prosthetic components increases and decrease the restoration reliability.

It is well known, that the external hexagon connections are not adequate due to the loads with different angles which drastically reduce the strength. The internal conical or hexagonal connections improve the compromise but even in screwed internal connections can be observed abutment neck fractures, some involving implant fractures [3, 5, 9-11].



**Figure 1.** SEM micrographs of an NDI fractured 17 months after implantation in a patient. The fracture is in the connection zone where cross-sectional surface is lower.

The objective of this research is in the first place to study the cause of such premature fractures of NDIs made of Ti-cp grade 4. In second place, an evaluation and the possible improvement of the mechanical properties will be investigated following two strategies: 1) alloy the titanium with 15% of Zr and 2) straining the Ti-cp at 12%, both in order to increase the mechanical strength, hardness and the fatigue life.

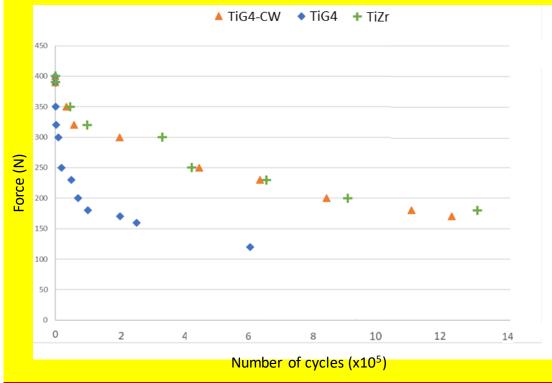
## 2. Experimental Results and Discussion.

The mechanical properties of the tested implants are shown in Table 1. The yield strength for the Grade 4 Ti-cp is lower and its value became significantly higher when the implants were composed of Ti-15Zr or presented a 12% cold worked. A similar trend was observed for the maximum strength (UTS). On the other hand, the strain-to-fracture was lower for the Ti alloy and cold worked Ti. The Ti-15Zr presented a significantly higher value of strain to the cold worked Ti. Consequently, the Ti treated by cold work presents a lower value of toughness, without any effects on other properties like biocompatibility or corrosion resistance of Ti-cp The hardness was shown to be significantly higher for the cold worked Ti than the other conditions. This fact is due to the increase of the linear defects in the hexagonal microstructure of the  $\alpha$ -titanium. The density of the slip-dislocations produces an increase in the hardness and the mechanical properties. The Ti-15Zr presented significantly higher hardness values than the Ti-cp grade 4, as well [12-15].

**Table 1.** Mechanical properties of the dental implants studied.

	Implant	σ <sub>max</sub> (MPa)	<mark>σ<sub>0.2</sub> (MPa</mark> )	<mark>ε</mark> (%)	HVN
Ti-cp (grade4)	JDE	460 (37)	357 (23)	17 (4)	104 (12)
Ti-15%Zr	Roxolid	877 (24)	678 (20)	22 (4)	199 (15)
Ti strained 12%	Vega	1100 (35)	740 (23)	8 (2)	380 (23)

Figure 2 shows the S-N curve for the different NDIs studied. The results show that the implants alloyed with <code>Zr</code> and the Ti grade 4 submitted to 12% <code>cold working</code> present more fatigue life than the <code>Ti</code> dental implants (grade 4) without hardening treatment. The implants cold work or alloyed present an asymptotic curve around 200 N. However, the <code>Ti-cp</code> grade 4 is around 100 N. These values demonstrate the importance of the static mechanical properties on the cyclic behavior. This aspect is very important in the clinical applications, the <code>Ti-cp</code> for NDIs can produce premature fractures when the loads in chewing being higher than 100 N.

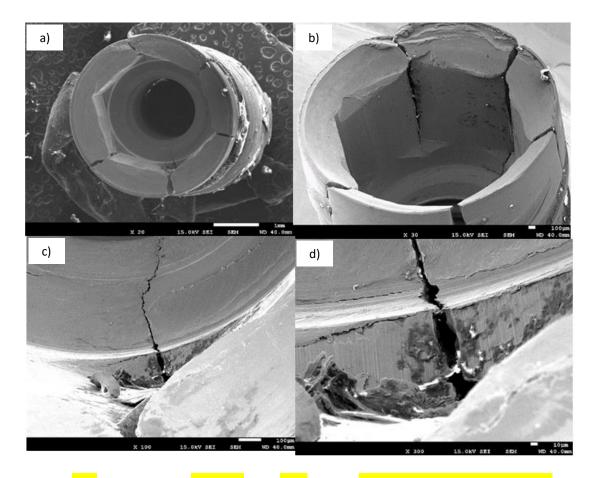


**Figure 2.** S-N curves of the different NDIs studied.

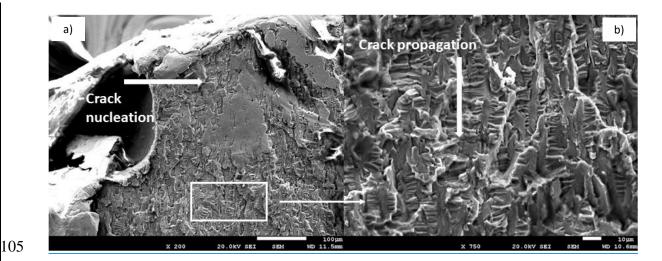
NDIs fractured after inserted in patients can observe that the fracture mechanism was fatigue in all cases. The fractography can be observed in Figure 3, with different SEM micrographs with a detailed description of both crack nucleation points and crack propagation paths. Figure 3 shows the presence of many longitudinal cracks along the practically all vertices between the walls of inner hexagonal connection.

The crack nucleation is generated inside of the implant body, specifically in the inner hexagonal connection walls just in the bottom horizontal ground plane. After crack nucleation, progressive crack growth produced under cyclic masticatory multiaxial loading seems to describe a propagation path along the vertices of hexagonal connection as well as through wall thickness, as can be seen in Figure 3b.

Further fractography analysis performed on fatigue tested NDIs showed the same fracture patterns, as well as crack nucleation points and fracture paths, described in retrieved implants.



**Figure 3.** SEM Fractography of explanted NDIs Ti-cp grade 4: a) SEM micrograph of an explanted implant top view, b) SEM micrograph of hexagonal inner connection (side view) with detail of longitudinal fracture cracks, c and d) SEM micrographs with detail of crack propagation at different magnifications.



**Figure 4.** SEM micrographs with detail of crack nucleation and propagation: a) SEM micrograph with detail of crack nucleation starting point, b) SEM micrograph with detail of crack cyclic propagation by fatigue.

From Figure 4A, site of crack nucleation can be observed, as well as a place with crushing, this is due to the friction between the surfaces of fracture in the crack propagation. This place is the less

effective surface which the load is applied and consequently this is a point of the stress exaltation which provokes the crack nucleation in a load cycle. After, the crack propagation is fast due to the cyclic loads to fracture. In Figure 4B can be observe the typical striation or fatigue marks which indicate the direction of the crack propagation [15-18].

One cause of the premature fracture of the Ti-cp Ti NDIs is the low mechanical properties, especially in the connection zone in relation to the others implants with strength and fatigue limits higher. On the other hand, in the manufacture process, the conformation of the hexagonal connection is made by plastic strain giving the connection shape. The plastic strain produces small defects, as can be observed in Figure 5. These defects present a higher residual energy and are points of crack nucleation. Besides, the implants are treated with acids in order to increase the roughness for improving the osseointegration and for passivate the dental implant. Sometimes, the acid etching is used to color the connection for the facilitating at the surgeon the identification of the pieces for the prosthesis. These acids etch the defects and produce cracks before the insertion of the implant in the patient. When the implant is inserted, the crack propagation starts due to the cyclic loads for chewing until fracture [19-21].

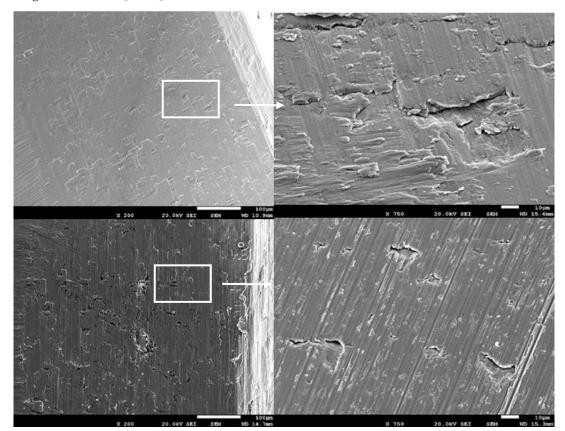
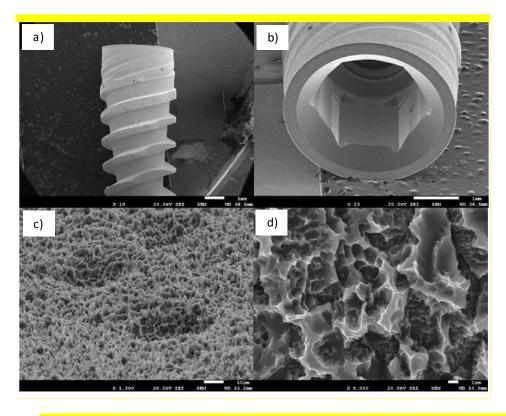


Figure 5. SEM micrographs of inner implant connection with details of some plastic deformation and crack formation on the connection surface due to the conformation methods like "broaching"

## 3. Materials and Methods

Sixty-four Ti-cp grade 4 fractured NDIs were studied. The dental implants presented internal connections screwed. The surface had been treated by sand-blasting, using  $Al_2O_3$  particles as a ...... material, as well as by acid-etching in order to achieve an average roughness (Ra) around 1.5  $\mu$ m. In Figure 6, it can be observed an original NDI in state of fabrication. Roughness was evaluated for the test surfaces using a white light interferometer microscopy (Wyko NT1100, Veeco) was used. The surface analysis area was  $459.9 \times 604.4 \mu$ m2 for all the microrough surfaces. Data analysis was performed with Wyko Vision 232TM software (Veeco, USA). A Gaussian filter was used to separate waviness and form from the roughness of the surface. Cut-off values,  $\lambda c = 0.8$  mm, for micro-rough

surfaces were applied. The measurements were made in three different surfaces of each type of surface treatment to characterize the Ra (the average roughness), which is the arithmetic average of the absolute values of the distance of all points of the profile to the mean line.



**Figure 6.** SEM micrographs of an NDI in state of fabrication: a) Longitudinal view with detail of self-tapping coils, b) Upper view with detail of inner hexagonal connection, c) Surface SEM micrograph showing micro roughness topography produced by sand blasting, d) SEM micrograph showing nano roughness topography produced by acid-etching.

For comparison with other NDIs which have been submitted to a hardening processes, such as alloying with 15% Zr or cold working at 12%, Roxolid® by Straumann and Vega® by Klockner, were used respectively. In Figure 7, the designs and main characteristics are presented in Table 2. As it can be observed, the macrodesign is very similar and the surface treatment followed is the same, achieving roughless of Ra around 1,7 µm for Roxolid and 1,9 µm for Vega.







**Figure 7.** NDIs studied. TiG4: cp titanium grade 4. TiZr: Titanium alloyed with 15% Zr and TiG4CW: cp titanium grade 4 cold worked at 12%.

Table 2. Implants used distributed by groups.

<b>GROUP</b>	GROUP 1	GROUP 2	GROUP 3	
	<mark>Cp-Ti grade 4</mark>	Ti alloyed with 15%Zr	Ti-cp grade 4 hardened	
			by 12% cold working	
<b>IMPLANT</b>	JDE (Ø3.2 mm, h	Bone level Roxolid (Ø3.3	Bone Level Vega (Ø3.5	
<b>TYPE</b>	8mm) (JDental Care,	mm, h=8mm) (Straumann	mm, h=8mm) (Klockner,	
	Modena, Itlay)	AGR, Basel, Switzerland)	Spain).	
CONNEECTION	conical internal	Cross-fit internal	<mark>hexagon</mark>	
TYPE			external / internal	

Initially, static uniaxial compression tension tests were conducted in order to determine the yield strength of the material, the ultimate strength and the strain to fracture. The hardness of the specimens was measured on polished cross-sections by using a Vickers microhardness tester (Akashi, Matsusawa, Japan) with a Vickers diamond indenter under a load of 0.98 N (100 gf) and 15 s of indentation. Fifteen data points were collected and averaged for each hardness value. Ten implants were analyzed for static tension and the same number for hardness testing (n=10).

Samples used in hardness testing were first embedded in methyl-methacrylate resin (Technovit 7200; Kulzer-Heraus GmbH, Wehrheim, Germany), and subsequently photo-polymerized in a light control unit (Histolux; Kulzer GmbH, Wehrheim, Germany) in order to obtain a solid transparent blocks to allow samples cutting and polishing, and avoiding any kind of deformation and/or fracture during these procedures. Resin blocks were cut in cross-section by using a diamond saw EXAKT 310 CL (EXAKT Advanced Technologies GmbH, Norderstedt, Germany) with continuous water irrigation at a maximum rotation speed with minimum load. Finally, cross-section samples were polished by means of an automatic grinding machine Exakt-400CS (EXAKT Advanced Technologies GmbH, Norderstedt, Germany), with parallelism, load and speed controls, first using SiC progressively abrasive papers (600, 800, and 1200 grit) and subsequently polished by 1 μm Al<sub>2</sub>O<sub>3</sub> abrasive suspension, following the recommendation of ASTM-E3 standard [22].

Implants used for mechanical testing were embedded into polymeric resin to mimic oral conditions before performing any mechanical assay in order to provide a stable support. NDIs were placed perpendicularly at 3.0mm±0.1mm above the nominal bone level described by the different manufacturers in a cold auto-curing polymeric resin (Mecaprex MA2+, Presi, France), as can be seen in (Figure 8).

All NDIs systems used for mechanical testing were assembled according to different surgical protocols, using the correct original components (abutments, screws and implants) as well as using proper torques defined by each manufacturer by using self-adjustable precision surgical tools.

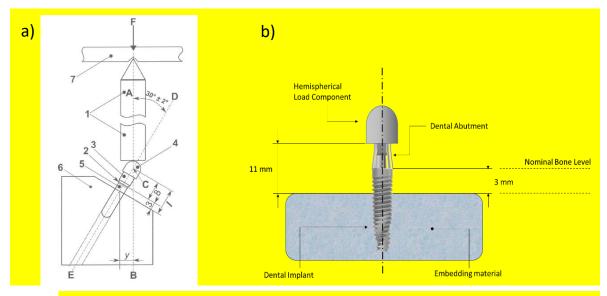
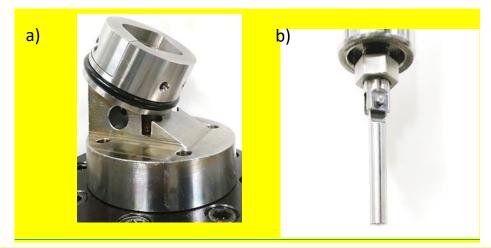


Figure 8. a) Overall scheme of the mechanical testing described by ISO 14801 standard and, b) Front view of representative drawing with detail of lengths and distances respected by the testing samples.

Following the static compression-to-fracture tests, fatigue tests at various percentages of the obtained yield strength were performed following the recommendations of the ISO 14801:2016 standard [23], which allowed determining the number of cycles before fracture. The aim was to find the stress value at which the sample supported a total of 10<sup>7</sup> cycles, which is considered the fatigue limit. The assays were performed with the servo-hydraulic testing machine (MTS Bionix 858, Minnesota, USA) equipped with the software TestStar II (MTS, USA). This machine was equipped with a load cell MTS of 25 kN. The implants were loaded with a sinusoidal function of fatigue at a frequency of 15 Hz and 10% stress variation.

The dynamic cyclic fatigue assay was carried out in a triaxial compression-flexion-torsion mode, placing the NDI implants fixed with an inclination of 30°±2° at from the compression stress application axis with the z axis of the tensile-compression machine (Figure 9), under sinusoidal load at 30° and 15Hz at room temperature and dry conditions. The data was represented as the number of cycles reached at fracture for different applied forces. The deformed and fractured specimens were observed by means of Field Emission Scanning Electron Microcopy (JSM 7100, Jeol, Japan).

Statistical significant differences among test groups for mechanical evaluation were assessed using statistical software (MinitabTM 13.1, Minitab Inc., New York, USA). ANOVA tables with multiple comparison Fisher test were calculated. The level of significance was established at p-value<0.05.



**Figure 9.** Detail of the fatigue testing grips used by servo-hydraulic testing machine: a) Lower clamping grip and, b) Upper articulated grip.

Sixty-four fractured implants remained in position for a period of time ranging from six weeks to 20 months, after which the dental implant fractured in all cases in the connection. After the dental implants were removed. The patients were from 35 to 74 years old being 58% women and 42% men. All patients were treated with calcium phosphate granules in order to regenerate the bone. Once bone formation was obtained a new dental implant was implanted.

#### 4. Conclusions

Mechanical properties of Ti-cp NDIs should be improved in order to guarantee long-term success of the treatment. Different premature fractures have been observed by fatigue due to the low mechanical properties and by the defects in the surfaces produced in the conformation process. Two methods for hardening the Ti-cp have been studied: 12% cold work straining or alloying with 15% Zr. In both cases, the mechanical properties were improved and specially the fatigue response of the NDIs. Ti-cp should be used with caution by the clinicians and this study will help clinicians to select a better NDI system for a more predictable treatment.

**Acknowledgments:** The authors kindly acknowledge the collaboration of many individuals and institutions in the present manuscript. The authors are grateful to the Spanish Government and European Union FEDER by the concession of the project RTI2018-098075-B-C22.

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