



Nanostructured severe plastic deformation processed titanium for orthodontic mini-implants

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ABSTRACT

Titanium mini-implants have been successfully used as anchorage devices in Orthodontics. Commercially pure titanium (cpTi) was recently replaced by Ti-6Al-4 V alloy as the mini-implant material base due to the higher strength properties of the alloy. However, the lower corrosion resistance and the lower biocompatibility have been lowering the success rate of Ti-6Al-4 V mini-implants. Nanostructured titanium (nTi) is commercially pure titanium that was nanostructured by a specific technique of severe plastic deformation. It is bioinert, does not contain potentially toxic or allergic additives, and has higher specific strength properties than any other titanium applied in medical implants. The higher strength properties associated to the higher biocompatibility make nTi potentially useful for orthodontic mini-implant applications, theoretically overcoming cpTi and Ti-6Al-4 V mini-implants. The purposes of the this work were to process nTi, to mechanically compare cpTi, Ti-6Al-4 V, and nTi mini-implants by torque test, and to evaluate both the surface morphology and the fracture surface characteristics of them by SEM. Torque test results showed significant increase in the maximum torque resistance of nTi mini-implants when compared to cpTi mini-implants, and no statistical difference between Ti-6Al-4 V and nTi mini-implants. SEM analysis demonstrated smooth surface morphology and transgranular fracture aspect for nTi mini-implants. Since nanostructured titanium mini-implants have mechanical properties comparable to titanium alloy mini-implants, and biocompatibility comparable to commercially pure titanium mini-implants, it is suggestive that nanostructured titanium can replace Ti-6Al-4 V alloy as the material base for mini-implants.

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1. Introduction

Anchorage is a known problem in Orthodontics and its control is fundamental to achieve success in the treatment [1]. Several appliances have been developed to control undesirable movements and to allow the treatment of complex cases [2–5]. Nowadays, the most used device to control anchorage is the mini-implant. It was developed based on the theory of osseointegration, which is the rigid integration between bone and a biomaterial. The interaction between bone and biomaterials such as stainless steel, titanium, niobium, and vitallium has been reported over the last decades [6–10]. Commercially pure titanium (cpTi) is the most used biomaterial in dental implantology. Due to its particular features, excellent results were described in studies in animals [4,7,11], and in humans [1,5,12]. The rigid integration between cpTi and bone allowed the development of conventional dental implants used to

replace lost teeth. Conventional dental implants were then tested for orthodontic anchor purposes [13]. Although conventional dental implants have proved to be successful for orthodontic anchorage, the distinct needs of orthodontic and prosthetics implants lead to the development of a specific system for orthodontic use, which is the orthodontic mini-implant. The mini-implant has been successfully used as an orthodontic anchorage device due to its small dimension, few limitation of insertion site, easy surgical procedure, less post-operation pain, low cost, facility to maintain oral hygiene, and uncomplicated way to attach elastics or springs [1,14,15].

The reduction of the size of conventional dental implant to mini-implant leads to a lot of benefits. Nevertheless, orthodontic mini-implants have shown more clinical failures than conventional dental implants submitted to orthodontic loads [16], with increase in fracture incidence and mobility of the mini-implants after the healing period [17]. In order to overcome the increased fracture incidence, cpTi was gradually replaced by Ti-6Al-4 V alloy as the material base of the mini-implant, due to the better mechanical properties of the alloy [15,18].

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Ti-6Al-4 V alloy has proved to be a good alternative to cpTi [19]. Serra et al. observed reduction in the fracture incidence because Ti-6Al-4 V alloy has higher mechanical properties than cpTi. On the other hand, the authors did not find improvements in the failure incidence related to the mobility of the mini-implants, because the use of the alloy may increase the metallic ion release to human body, due to the low corrosion resistance of Ti-6Al-4 V alloy in body fluids [20,21].

Aluminum and vanadium have been related to diseases such as osteomalacia, anemia, neurotoxicity, pulmonary granulomatosis, and kidney lesions [22–24]. These problems may not be relevant in the orthodontic context due to both the small size of mini-implant, and the short time inside the bone. However, metallic ion release can impair the osseointegration and the biocompatibility of the device. Corrosion resistance in body fluids plays an important role in the biocompatibility process and in the implant maintenance in human tissues. Moreover, metallic particles can be lost by abrasion during torque insertion and removal of the mini-implants, and can be deposited in the surround bone tissue, impairing the osseointegration [25]. Thus, cpTi is more biocompatible, less toxic, and more resistant than Ti-6Al-4 V alloy. Nevertheless, its mechanical properties are unsuitable for orthodontic applications, which require load support in a device with small dimensions [24].

Recently, nanostructured titanium (nTi) has been developed and proposed for biomedical applications. The higher mechanical resistance is pointed out as the main improvement to the use as biomaterials for fixation in medicine [26–29]. There was applied new processing technique entitled severe plastic deformation (SPD) for nanostructuring of titanium [29]. The rods were produced from high-strength titanium by means of equal channel angular pressing combined with drawing. As numerous studies revealed, SPD makes it possible to form nanostructural state in titanium and significantly enhance its mechanical properties to the level exceeding those of titanium processed by conventional methods [28,30,31].

We hypothesize that nTi could replace cpTi and Ti-6Al-4 V alloy as the material base for orthodontic mini-implants, since it has higher strength properties than cpTi, better biocompatibility, higher corrosion resistance, and less toxicity than Ti-6Al-4 V alloy. Hence, the aims of this work were (1) to process nanostructured titanium, (2) to mechanically compare cpTi, Ti-6Al-4 V, and nTi mini-implants by torque test, and (3) to evaluate both the surface morphology and the fracture surface characteristics of them by SEM.

2. Materials and method

2.1. Nanostructured titanium processing

The effort was conducted using commercially pure Grade 4 titanium [C – 0.052%, O₂ – 0.34%, Fe – 0.3%, N – 0.015%, Ti-bal. (wt. pct.)]. Nanostructuring involved severe plastic deformation (SPD) processing by equal-channel angular pressing (ECAP) followed by thermo-mechanical treatment (TMT) [32,33] producing 7 mm diameter bars with a 3 m length.

The rod production process comprised a few stages. First, the billets were subjected to ECAP. In this process, the billets were punched in a special equipment through two intersecting channels of equal cross-sectional area lying at 90° to each other. The deformation was conducted at 450 °C. There were four passes and the billets were rotated about the longitudinal axis by 90° after each pass [28]. The obtained billets were finally subjected to forging and drawing with cumulative deformation equal to 80%. The last stage was to anneal them at 300–350 °C for 1 h.

The billets microstructure analysis was conducted by the methods of scanning and transmission electron microscopy. Standard billets notched out of the rod central part were used for the stretching tests (GOST 1498-84). Mechanical tensile tests were performed at room temperature on the tensile machine with a strain rate of 10^{-3} s^{-1} on

the flat samples with the following sizes: gage length $l_{\text{basic}} = 4 \text{ mm}$, width = 1 mm and height = 0.5 mm. At least three samples were tested for each state.

2.2. Torque test (TT)

Nanostructured Ti, cpTi, and Ti-6Al-4 V alloy mini-implants were machined by turning, cleaned, and passivated with nitric acid (HNO₃). The mini-implants had a cylindrical screw design and a hexagonal shaped head (6.0 mm x Ø 2.0 mm; Conexão Sistemas de Próteses, SP, Brazil). A total of 15 mini-implants were used in this study (Fig. 1).

The test was measured with an Instron universal test machine with a 50 N load cell. It was performed by applying a counter-clockwise rotation to the mini-implant at a rate of $0.1^\circ/\text{s}$. To obtain alignment between the mini-implant and the axis of the Instron universal test machine, a device was designed and machined (Fig. 2). Hence, there was only torsional load during the torque test, avoiding bending moment.

The tests were done with the mechanical traction on the vertical axis of the device resulting on the torque force. The arithmetic conversion from distance to degrees was calculated based on the diameter of the rotation pivot as:

$$P = 2.\pi.r \quad \text{and} \quad P = 360 \text{ degrees}$$

To set up the mechanism and to ensure correct alignment for torque test measurements, a holding fitting the head of implant was attached to the left grip of the device. Then, the end of the mini-implant was attached to the right grip. In each torque test, the curve was recorded and the maximum value (N.mm) was considered the maximum torque resistance (MTR) [19].

The statistical analysis for reporting mean and standard deviation of data from MTR was performed for all groups. For significance of differences, the data were evaluated by one-way analysis of variance test (ANOVA) followed by the post hoc Tukey test ($p < 0.05$).

2.3. SEM

The surface morphology and the original design of all mini-implants were qualitatively analyzed. Afterwards, the fractured mini-implants

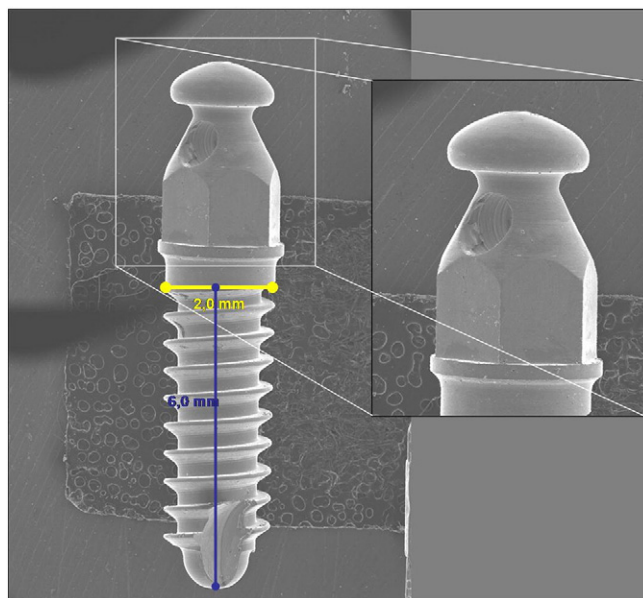


Fig. 1. Machined mini-implants design. Hexagonal head, 6.0 mm in active length, and 2.0 mm in diameter.

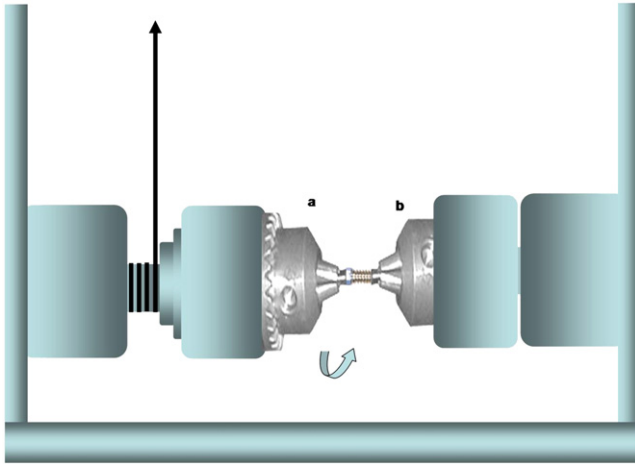


Fig. 2. Torque test diagram: (a) rotational left grip where the mini-implants head was attached; (b) fix right grip.

resulting from the torque test were evaluated in the fracture area. The SEM images were obtained by secondary electrons incidence.

3. Results

3.1. Nanostructured titanium processing

The SPD processing resulted in reduction of the grain size. The original titanium rods presented a 25 μm equiaxed grain structure. It was reduced to 150 nm after combined SPD and TMT processing, as shown in Fig. 3. At selected area of the nanostructured Ti microstructure (Fig. 3c), the electron diffraction pattern further suggests that the ultra fine grains contained predominantly high-angle grain boundaries with non-equilibrium distorted structure leading to their excess energy [28].

A similar microstructure can be produced in small discs of cpTi using other SPD methods, for example – high-pressure torsion (HPT). In the present work, it was essential to produce homogeneous ultrafine-grained structure throughout a 3 m length rod. It allowed the mini-implants production and supplied the material needs for thorough mechanical and biomedical tests of nanostructured titanium.

Table 1 illustrates the mechanical property benefits attainable by nanostructuring cpTi. The strength of the nTi has almost doubled vis-a-vis conventionally processed cpTi. Notably, this improvement has been achieved without the drastic ductility reductions normally seen after rolling or drawing.

Laboratory fatigue studies of nanostructured and conventional cpTi were performed according to ASTM E 466-96 at $R (\sigma_{\min}/\sigma_{\max}) = 0.1$ and 20 Hz. Fig. 4 shows that the fatigue strength of nanostructured Ti at 10^6 cycles is almost 80% higher than conventionally processed cpTi and approaches that of the Ti-6Al-4 V alloy [27].

Table 1

Mechanical properties of conventionally processed and nanostructured cp Grade 4 titanium.

State	Processing	UTS, MPa	YS, MPa	Elongation, %	Reduction in area, %
1	conventional Ti warm-rolled	700	530	25	52
2	nTi ECAP + TMT	1240	1200	12	42

3.2. Torque test (TT)

The MTR means and standard deviations for cpTi, Ti-6Al-4 V, and nTi mini-implants were $114.8 \text{ N.mm} \pm 11.5$, 153.0 ± 24.5 , and 160.1 ± 18.8 , respectively (Table 2). The statistical analysis demonstrated that Ti-6Al-4 V and nanostructured Ti mini-implants had significantly higher MRT than cpTi mini-implants (Table 3). When Ti-6Al-4 V and nanostructured Ti mini-implants were compared between each other, they did not show significant statistical difference ($p < 0.05$) (Fig. 5).

3.3. SEM

The original surface morphology (Fig. 6a and b) and the fracture morphology after the torque test (Fig. 6c and d) of cpTi, Ti-6Al-4 V, and nTi mini-implants were compared at low and high magnification.

The mini-implants of different base materials were machined in order to have the same shape (Fig. 1). The SEM evaluation demonstrated that all of the experimental mini-implants presented a cylindrical screw design, a hexagonal head, 6.0 mm in active length, 2.0 mm in diameter, a total of 9 pitches, and 0.51 mm between the top of the pitches (Figs. 1 and 6a).

The 6a and 6b micrographs series exhibited the mini-implants surface in macroscale. Comparing the superficial finishing, nTi mini-implants presented the smoothest surface, and Ti-6Al-4 V mini-implants presented the roughest surface (Fig. 6b). The 6c and 6d SEM micrographs series presented a torsional pattern fracture in all material base groups. The shear deformation was concentrated out of the center of the mini-implants long axis. It is possible to observe that the fracture surface of the mini-implants is smooth and relatively flat, perpendicular to the axis of the mini-implant, and characterized by a “center of twist” surrounded by concentric circular flow lines.

4. Discussion

The rigid bone anchorage with mini-implants was developed in Orthodontics based on the application of conventional dental implants to replace lost teeth [13]. The conventional dental implants were successfully used to anchor purposes in the orthodontic treatment. However, the big size, the large bone area required to insertion, the high post-operative pain, the difficult to attach springs and elastics, and the high cost were important limitations [1]. Mini-implants were

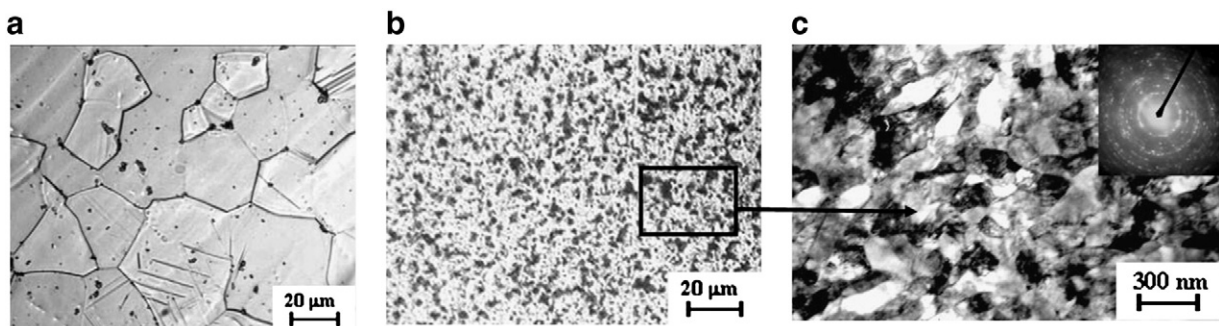


Fig. 3. Optical and transmission electron microscopy illustrating microstructure of CP Grade 4 titanium: (a) Conventional; (b,c) ECAP + TMT.

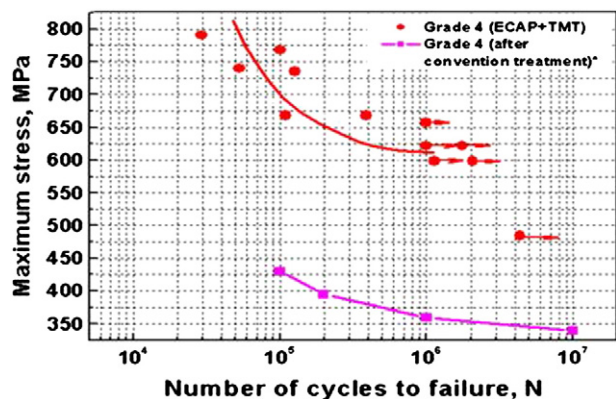


Fig. 4. Fatigue study in room temperature for conventional and nanostructured CP Grade 4 titanium ($R = 0.1$ at 20 Hz).

the most useful orthodontic system developed to overcome these limitations.

CpTi grade 4, commonly used in the conventional dental implants, was the first material choice for mini-implants [1,14]. However, due to the required small size of the mini-implants, fractures were reported during insertion and removal surgical procedures [4,17]. Hence, cpTi was gradually replaced by Ti-6Al-4 V alloy as the mini-implant material base. In spite of the poor biocompatibility and the lower corrosion resistance, Ti-6Al-4 V alloy became widely used for this orthodontic application [18,19,34–36]. This replacement solved the problems of mechanical resistance, but impaired the osseointegration process.

The carried investigation revealed the possibility to produce nanostructured Ti from cpTi grade 4, by equal-channel angular pressing (ECAP) followed by thermo-mechanical treatment (TMT). The nTi produced presented higher ultimate strength and yield strength than conventional cpTi grade 4, without an important ductility reduction. These increased strength properties, added to the higher biocompatibility and higher corrosion resistance in body fluids of cpTi, when compared to Ti-6Al-4 V alloy, made nanostructured Ti useful to implants in medical applications [26].

The strength properties enhanced by the SPD processing allow the indication of nTi in place of Ti-6Al-4 V alloy as material base of mini-implants. Moreover, this replacement would result in a relevant discussion issue related to the biocompatibility, the corrosion resistance and the mechanical resistance of the mini-implants.

The biocompatibility and the corrosion resistance are strongly related to the surface oxide layer that covers the metallic materials. The cpTi oxide layer is mainly composed of TiO_2 with small amounts of TiO and Ti_2O_3 [35]. In Ti-6Al-4 V alloy, the surface oxide layer is composed by a variety of oxides (TiO , Ti_2O_3 , and TiO_2) with amounts of Al_2O_3 , hydroxyl groups, and water [37]. When the alloy elements Al and V are added to cpTi to produce the Ti-6Al-4 V alloy, the mechanical resistance is improved, but the corrosion resistance decreases. This occurs because the alloy elements destabilize the surface oxide of the Ti-6Al-4 V alloy, making it vulnerable to corrosion. When the alloy is implanted into the human body, it contacts the body fluids and the metal elements present in the alloy (Ti, Al, and V) are released to local and remote tissues [38]. Titanium is considered biocompatible since there is no sign of local or systemic reaction when this material is used in the human body [39]. On the other hand, the low percentages

Table 2
Torque test results: maximum torque resistance means and standard deviations.

Groups	Valid <i>n</i>	Mean (N.mm)	S.d. (N.mm)
Cp Ti	5	114.8	11.5
Ti-6Al-4 V	5	153.0	24.5
Cp nanostructured Ti	5	160.1	18.8

Table 3

One way ANOVA and post hoc Tukey for comparison means of MTR values.

Analysis of Variance	<i>P</i> value = 0.0056		
Term	Probabilities for Post hoc tests: material base mini-implants		
Post hoc test	Cp Ti	Ti-6Al-4 V	Cp nanostructured Ti
Cp Ti (114.8)	–	*0.0202	*0.007
Ti-6Al-4 V (153.0)	*0.0202	–	0.826
Cp nanostructured Ti (160.1)	*0.007	0.826	–

* Difference statistically significant (*p* value < 0.05).

of Al and V are potentially toxic [26,35]. Aluminum has been associated to encephalopathy, Alzheimer's disease, osteomalacia, pulmonary granulomatosis, and fibrosis, while high concentrations of vanadium are associated to kidney lesions [26,40]. Thus, under the biocompatibility point of view, cpTi and nTi mini-implants are safer to be used than Ti-6Al-4 V mini-implants.

In the clinical situation, mini-implants are submitted to torque during insertion and removal surgical procedure. Clinical fractures of mini-implants have been mostly described during these procedures [4,15,17,19]. In this way, torque test was used in this work to compare the fracture resistance of the three types of mini-implants due to the correlation with the clinical procedure and due to be the most used test to evaluate the mini-implant fracture resistance [4,7,15,17,35]. Buchter et al. [4] tested 200 cpTi mini-implants with 1.1 and 1.6 mm in diameter. The authors described a total of 11 fractures during insertion or removal procedures. Miyawaki et al. [17] tested cpTi mini-implants with the diameter of 1.0, 1.5 and 2.3 mm. All the cpTi mini-implants with 1.0 mm in diameter failed due to fractures or excessive mobility. Park et al. [15] suggested using cpTi mini-implants with 2.0 mm or more in diameter to avoid the fractures and Huang et al. [18] described that Ti alloy use could overcome this disadvantage. In this manner, Serra et al. [19] tested Ti-6Al-4 V alloy mini-implants. They did not describe any fracture in a total of 72 mini-implants tested. However, they described failure rate of 10% related to the osseointegration process. Freire et al. [41] did not described fractures in a total of 78 Ti-6Al-4 V alloy mini-implants tested, but the success rate of the experimental group varied between 66.67 and 88.89%. On the mini-implants development, the change of the material base solved the fracture limitation, while the failure rate still remained high. It could be related to the lower biocompatibility of the Ti-6Al-4 V alloy. The torque test result of the present work did not show statistical difference between nTi and Ti-6Al-4 V mini-implants. It allows us to suggest

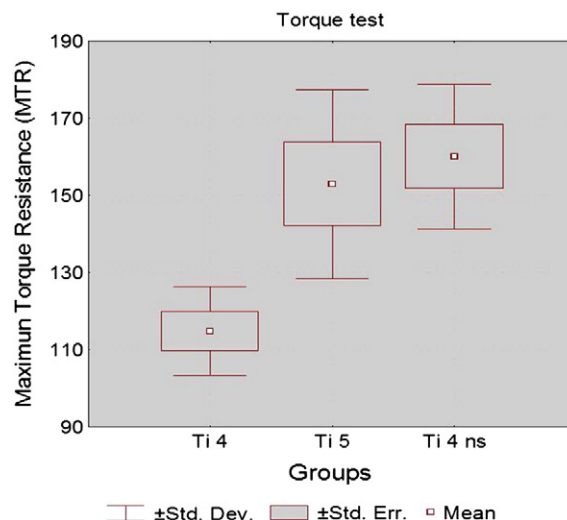


Fig. 5. Box plot graphic: the MTR means and standard deviations for cpTi, Ti-6Al-4 V, and nTi mini-implants.

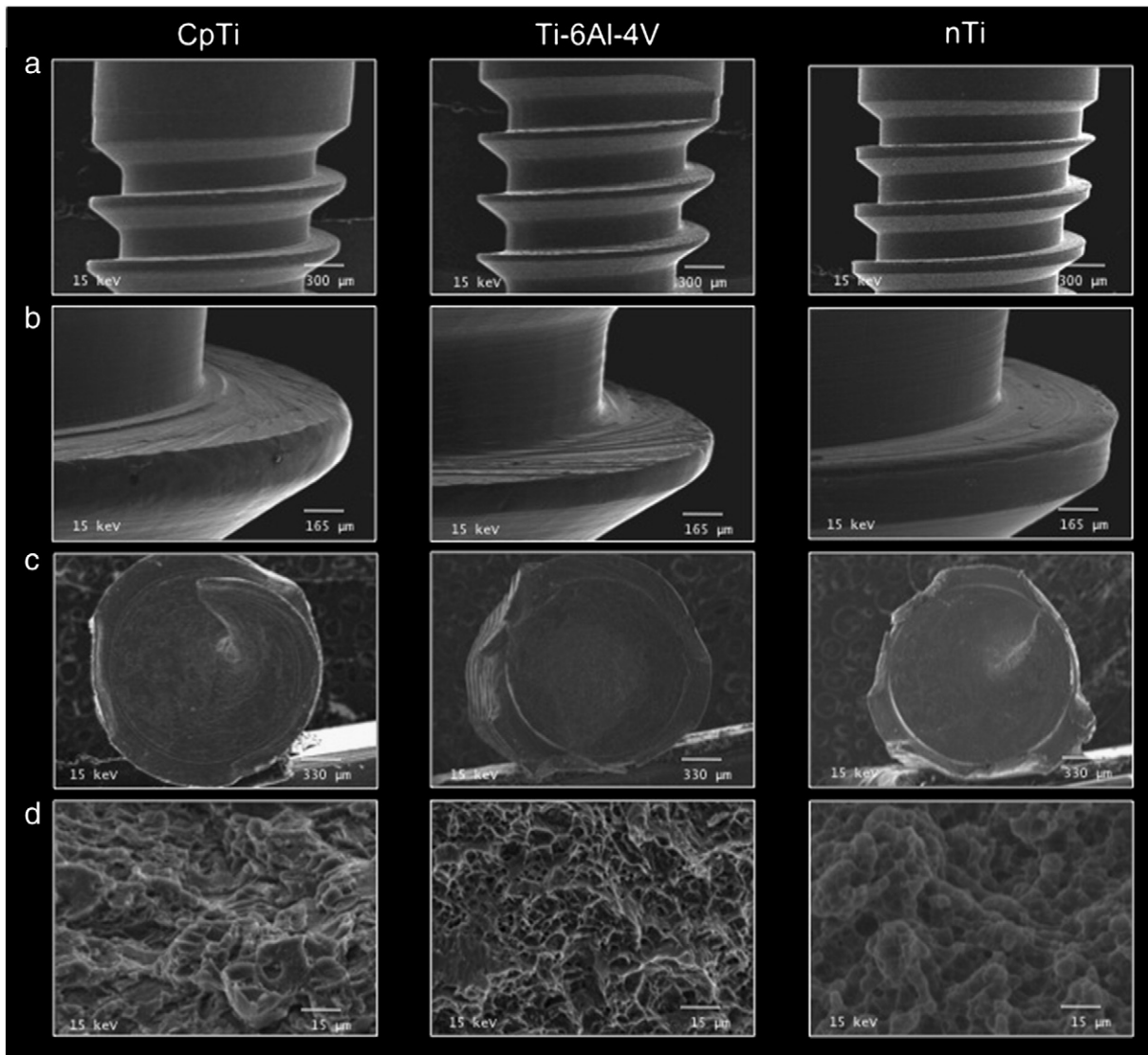


Fig. 6. CpTi, Ti-6Al-4 V and nTi mini-implants (a) (b) screw thread view with surface machined marks; (c) (d) post fracture.

that the nanostructured Ti could overcome the Ti-6Al-4 V alloy clinical performance due to its better biocompatibility properties and the equal torque resistance [26,35,39].

The SEM analysis showed the smoothest surface in nanostructured Ti mini-implants. The difference of the pattern of surface finishing observed in all samples was not significant to the healing or to the osseointegration process. Surface treatment has been proposed in mini-implants in order to increase the bone-mini-implant contact area. Porous-surfaced implants were compared to machined implants by Oyonarte et al. [42]. It was demonstrated that porous-surfaced mini-implants presented higher bone-mini-implant contact area after the healing process [42]. Chaddad et al. [43] tested sandblasted/acid etched surfaces in order to compare the effect of the increased surface area in the osseointegration process. The survival rate of the sandblasted/acid etched mini-implants was 93.4% and the machined mini-implants was 82.4% [43]. The superficial area increase obtained by different surface treatment has been described as a good alternative to decrease the failure rate. However, surface finishing changes observed in the present work did not result in a significant increased area. On the other hand, ECPA titanium microroughened surfaces were compared with Ti-6Al-4 V and conventional coarse-grain pure titanium microroughened surfaces by morphology of spread

cells, cell attachment, viability, alkaline phosphatase (ALP) activity, quantitative analysis of osteoblastic gene expression and mineralization nodule formation. The authors related superior osteoblast cell compatibility and greater mineralization nodule formation on ECPA Ti surface [44]. This results anchor the possible higher osseointegration even in machined surfaces.

The mini-implants fractures had the center of twist out of their geometric center due to the shape of their thread. This condition shows that the shear stresses were developed in the plane of fracture and the thread is a stress concentration area. To accomplish this, the center of twist is displaced away from the geometric center, along a diameter perpendicular to the direction of the applied force. Helical dimples followed the twist deformation on the cylindrical portion of the mini-implant. These dimples are similar to the dimples typically observed in a ductile material subjected to pure torsional loading. All of the mini-implants had similar fracture process and the torsion resistance was higher in nTi and Ti-6Al-4 V mini-implants.

The failure incidence, the presence of potentially toxic elements, and the mechanical resistance of the mini-implants have been stimulated the interest to adopt others alternatives than Ti-6Al-4 V mini-implants. The nanostructured Ti as the mini-implant material base has shown potential use due to demonstrate mechanical resistance

compatible for clinical use, due to be corrosion resistant in body fluids, and due to be biocompatible without the presence of the vanadium and aluminum elements.

5. Conclusion

The association of the biocompatibility of cpTi mini-implants and the mechanical resistance of the Ti-6Al-4 V mini-implants is concentrated in nTi mini-implants. On the present investigation, nTi mini-implant has shown torsion resistance compatible with Ti-6Al-4 V mini-implants and better than cpTi mini-implants. Similar surface finishing and fracture process among the 3 types of mini-implants were found. Based on these results, it is suggestive that nanostructured Ti could be useful as the material base for orthodontic mini-implants. We thank Brazilian National Council for Scientific and Technological Development (CNPq) for the financial support of this study.

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