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Nanostructured severe plastic deformation processed titanium for orthodontic mini-implants

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article info abstract

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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 [\[1\]](#page--1-0). Several appliances have been developed to control undesirable movements and to allow the treatment of complex cases [\[2](#page--1-0)–5]. Nowadays, the most used device to control anchorage is the mini-implant. It was developed based on the theory of osseointegration, which is the rigid integration between bone and a biomaterial. The interaction between bone and biomaterials such as stainless steel, titanium, niobium, and vitallium has been reported over the last decades [6–[10\]](#page--1-0). 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 [\[4,7,11\],](#page--1-0) and in humans [\[1,5,12\].](#page--1-0) 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 [\[13\].](#page--1-0) 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 [\[1,14,15\]](#page--1-0).

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 [\[16\],](#page--1-0) with increase in fracture incidence and mobility of the mini-implants after the healing period [\[17\].](#page--1-0) 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 [\[15,18\].](#page--1-0)

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Ti-6Al-4 V alloy has proved to be a good alternative to cpTi [\[19\].](#page--1-0) 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 found 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 [\[20,21\].](#page--1-0)

Aluminum and vanadium have been related to diseases such as osteomalacia, anemia, neurotoxicity, pulmonary granulomatosis, and kidney lesions [\[22](#page--1-0)–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 [\[25\].](#page--1-0) 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 [\[24\]](#page--1-0).

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\].](#page--1-0) There was applied new processing technique entitled severe plastic deformation (SPD) for nanostructuring of titanium [\[29\].](#page--1-0) 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 [\[28,30,31\].](#page--1-0)

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 thermomechanical treatment (TMT) [\[32,33\]](#page--1-0) 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 crosssectional 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 [\[28\]](#page--1-0). 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 $l_{\text{basic}} = 4$ 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 ∅ 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 counterclockwise rotation to the mini-implant at a rate of $0.1\frac{o}{s}$. To obtain alignment between the mini-implant and the axis of the Instron universal test machine, a device was designed and machined [\(Fig. 2\)](#page--1-0). 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:

$P = 2 \pi r$ and $P = 360$ 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 miniimplant 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) [\[19\].](#page--1-0)

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.

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