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Significance of the contacting and no contacting thermoelectric power measurements applied to grit blasted medical Ti6Al4V

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ABSTRACT

Grit blasting is a surface plastic deformation technique aimed to increase the surface area available for bone/implant apposition, which contributes to improve fixation and mechanical stability of Ti–6Al–4V implants. Besides roughening, grit blasting also causes surface contamination with embedded grit particles and subtle subsurface microstructural changes that, although does not challenge their biocompatibility, might influence other surface dominated properties like corrosion and ion release. Additional benefits are expected due to the induced compressive residual stresses, hence enhancing fatigue strength. The net effect depends on the type of particles used for blasting, but also on the amount of the subsurface cold work associated to the severe surface plastic deformation. In this work we study the potential of the non-contacting and contacting thermoelectric power (TEP) measurements in the analysis of the global changes induced in the Ti6Al4V when blasting the alloy with Al_2O_3 or ZrO_2 particles, which yields a coarse and a fine rough surface, respectively. To reveal the effect of residual stresses, a set of specimens were thermally treated. The study proves that the non-contacting technique is more sensitive to the presence of residual stresses, whereas the contact technique is strongly influenced by the grain size refinements, work hardening and changes in solute.

1. Introduction

316 LVM (low carbon vacuum melt) and Ti6Al4V alloys are the materials of choice for many structural implantable device applications and there is no reason to expect a change in the short and medium term. Despite their good corrosion resistance and reasonable biocompatibility, many efforts have been addressed to enhance their biofunctionality through surface modifications. Roughening of the implant by grit blasting is a low-cost solution to increase the surface area available for bone/implant apposition [1–3], which contributes to improve fixation and mechanical stability of implants. Typically examples for Ti-base alloys are dental implants or hip components. Severe surface plastic deformation by grit blasting is also considered an attractive modification of 316 LVM to improve fatigue strength of intramedullary nails for the proximal femur and diaphysary fractures, providing an optimal combination between high resistance during the consolidation period and a minimal invasive geometry. Additional benefits are expected due to the induced compressive residual stresses at the subsurface that could contribute to delay crack nucleation and further propagation, hence enhancing fatigue strength. The net effect depends on the roughness and in turn on the type of particles used for blasting, but also on the amount of the subsurface cold work associated to the severe surface plastic deformation [4]. Besides the primary residual stress effect, grit blasting also causes surface contamination with embedded grit particles and subtle subsurface microstructural changes that, although does not challenge their biocompatibility, might influence corrosion resistance and ion release [5,6]. Moreover, since maximum loads use to occur at the surface, they could play a detrimental role in the fatigue strength by acting as stress concentrators. Characterization of the global blasting induced effects becomes then of paramount importance for the intended medical applications.

Residual stresses developed by blasting are usually evaluated by X-ray diffraction (XRD) or synchrotron radiation X-ray diffraction (SR-XRD) [7,8]; whereas subsurface work hardening and the associated microstructural changes are assessed by combining scanning electron microscopy (SEM) and microhardness testing [9,10]. Thermoelectric power (TEP) measurements have recently been performed using the contacting and noncontacting techniques to detect the global blasting induced effects in the medical 316 LVM stainless steel [11–13]. Development of small amounts of α′-martensite phase at the subsurface, however, was found to limit the applicability of the noncontacting thermoelectric technique. Elimination of the magnetic phase by appropriated thermal treatments after blasting revealed that both techniques offer complementary information useful for monitoring the global blasting induced effects.

In this work, we study the potential of the non-contacting and contacting thermoelectric power measurements in the analysis of the

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global changes induced by grit blasting on the Ti6Al4V alloy, where magnetic phases are not expected to form [4,7]. The attractiveness of this blasted alloy is primarily based on an excellent combination of corrosion resistance and biocompatibility, especially after thermal oxidation treatments [14–16].

2. Thermoelectric power techniques

Most existing thermoelectric techniques are based on the well-known Seebeck effect that is commonly used in thermo-couples to measure temperatures across junctions made of different materials. A variety of material properties can contribute to change the TEP of materials. The fact that metal sorting is a fundamental application of thermoelectric materials characterization stems from the fact that chemical composition exerts the strongest effect on the thermoelectric properties of the material [17]. However, it is known that under special conditions, different heat treatments, hardening, texture, fatigue, etc. can also produce an effective thermoelectric voltage between contacting materials of identical chemical composition [18,19]. Consequently, by using a reference electrode that is similar to the material being tested, we can reliably estimate small differences in TEP, thereby characterizing material property of interest.

The ability of the contacting thermoelectric technique detecting small variations in material properties can be limited by the existence of spurious thermoelectric signal related to the inherently imperfect contact between the specimen and the reference electrode [19]. Clearly, conventional thermocouples based on Seebeck effect cannot be used in a noncontacting manner since they require direct electrical and thermal coupling with the specimen to be inspected. However, thermoelectric measurement can be conducted in an entirely noncontacting way by using high-sensitivity magnetic sensors to detect the thermoelectric current caused by inclusions and other types of inhomogeneities when inspected specimen is subjected to direct heating and cooling [20]. Assuming the existence of a defect or imperfection in an otherwise homogeneous material and that a temperature gradient is established throughout the specimen, different points at the boundary between the defect or imperfection and the host material will be at different temperatures, and therefore at different thermoelectric potentials. These potential differences will drive local thermoelectric currents around the affected area, which can be detected in a noncontacting manner by a high sensitive magnetometer. This technique was originally developed for the detection of metallic inclusions in metals, but has shown to be sensitive enough to a series of material imperfections that are currently no detectable or very challenging by any other inspection methods, including cold work, localized texture, residual stress, excess heat, fatigue damage, etc. [21].

3. Material and experimental procedure

A Ti6Al4V ELI (extra low interstitial) alloy was used in this study. Rectangular specimens of about 8×25 mm^2 and 2 mm thick were machined and grit blasted with different types of particles under a pressure of 350 kPa for 2 min and with a distance between the nozzle and the target surface of 20 cm. A first set of samples, hereafter BL-ZrO samples, was blasted using ZrO_2 microspheres sized between 125 μm and 250 μm. The second set of samples, hereafter BL-AlO samples, was blasted with Al_2O_3 angular particles of $\approx$750 μm. Blasting was manually performed in such a way that all surfaces become equally modified. For comparative purposes, polished samples, hereafter PL samples, were ground with consecutively finer SiC papers, and finely polished with diamond paste and colloidal silica (0.5 μm) to remove the outer surface layer of deformed metal during the sample preparation. Roughness of the as-processed specimens was determined with a mechanical profilometer averaging 3 measurements of 4 cm in length.

It is important to mention that one of the main blasted induced effects is the cold work of the outer zone, which should contribute to change the TEP of blasted samples. Thus, in order to establish how it affects the TEP measurements, a set of blasted Ti6Al4V samples were annealed at 595 °C and 710 °C for 1 h and 2 h respectively. Such heat treatments are known to induce a partial and a fully release of the residual stresses, respectively [22].

A schematic representation of the contacting thermoelectric technique is given by Fig. 1. The sample is pressed between two blocks of pure copper. One of the blocks is at 15 °C, while the other is at 25 °C to obtain a temperature difference, ΔT. A potential difference, ΔV, is generated at the reference metal contacts. The apparatus does not give the absolute TEP value of the sample (S^*), but a relative TEP (S) in comparison to the TEP of pure copper ($S_0{}^*$) at 20 °C. The relative TEP value (S) is given by $S = S^* - S_0{}^* = \Delta V/\Delta T$. The measurements are performed very quickly (<1 min) and precisely ($\pm$0.5%), with a resolution of about 1 nV/K [23].

On the other hand, in the noncontacting thermoelectric technique each sample is mounted into two pure Cu supporters which are perforated by a series of holes and equipped with sealed heat exchangers to facilitate efficient heating and cooling. The system is mounted on a nonmagnetic translation table for scanning. In order to get a better heat transfer between the specimen and the Cu heat exchangers, a layer of silicone heat sink compound was applied. One of the Cu supporters is at 15 °C, while the other is at 25 °C. The temperature gradient is kept at ~1.2 °C/mm in noncontacting TEP measurements, which is more than sufficient to produce detectable magnetic signals in the grit blasted Ti6Al4V alloy samples. A fluxgate magnetometer is used to detect the thermoelectric signals from the grit blasted zone. Since the resulting magnetic field is perpendicular to the heat flux in the specimen (parallel to the surface) and the gradient of the material property (normal to the surface), the magnetometer was polarized in the tangential direction as shown in Fig. 2. The lift-off distance between the magnetometer and the sample surface is ~2 mm.

Determination of the residual stresses and identification of phases present at the outer surface were performed with a conventional X-ray diffractometer equipped with a tube of Co. For the calculation of residual stresses, the sen^2 ψ method and the (203) reflection of Ti was applied. Measurements were performed by tilting the specimen

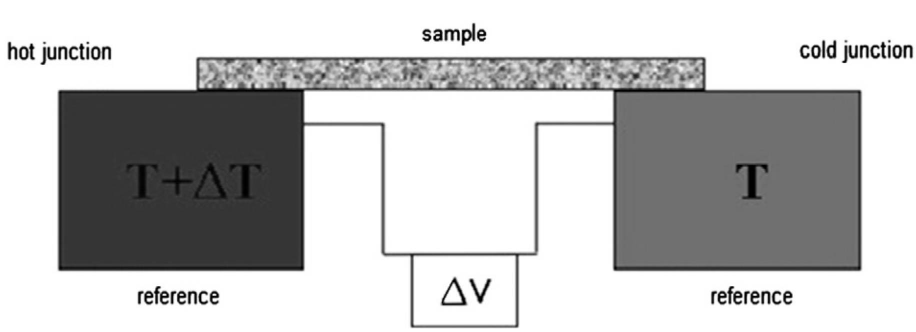

Fig. 1. Schematic diagram of the contacting thermoelectric technique as used for the characterization of grit-blasted samples.