Optical Engineering

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Abstract. Laser peening has recently emerged as a useful technique to overcome detrimental effects associated with other well-known surface modification processes such as shot peening or grit blasting used in the biomedical field. It is worthwhile to notice that besides the primary residual stress effect, thermally induced effects might also cause subtle surface and subsurface microstructural changes that might influence corrosion resistance and fatigue strength of structural components. In this work, plates of Ti-6Al-4V alloy of 7 mm in thickness were modified by laser peening without using a sacrificial outer layer. Irradiation by a Q-switched Nd-YAG laser (9.4-ns pulse length) working at the fundamental 1064-nm wavelength at 2.8 J/pulse and with water as a confining medium was used. Laser pulses with a 1.5-mm diameter at an equivalent overlapping density of 5000 cm⁻² were applied. Attempts to analyze the global-induced effects after laser peening were addressed by using the contacting and noncontacting thermoelectric power techniques. © 2014 Society of Photo-Optical Instrumentation Engineers (SPIE) [DOI: [10.1117/1.OE.53.12.122502](https://doi.org/10.1117/1.OE.53.12.122502)]

Keywords: laser shock peening; thermoelectric measurements; Ti-6Al-4V; biomaterial.

Paper 140177SSP received Jan. 29, 2014; revised manuscript received Mar. 21, 2014; accepted for publication Apr. 8, 2014; published online Jun. 11, 2014.

1 Introduction

Titanium and its alloys are extensively used in the aeronautics, medical, and engineering industries due to their intrinsic properties, such as light weight, high strength to weight ratio, corrosion resistance, biocompatibility, and excellent high-temperature properties. Surface engineering of titanium alloy components, used as biomaterials, provides a means by which the desirable bulk properties are retained in conjunction with enhanced fatigue strength. In addition, efforts to improve the osseointegration, fixation, and stability of titanium base implants have been addressed by creating a rough topography that increases the surface area available for bone/implant apposition. Various surface processing technologies are applied in order to achieve this goal of surface treatment modification. Almost all existing methods of surface enhancement are currently available to develop a layer of compressive residual stress following mechanical tensile deformation. The methods differ primarily in how the surface is deformed (roughness) and in the magnitude and form of the resulting residual stress and cold work (plastic deformation) distributions developed in the surface layers.

For example, conventional air-blast shot peening is generally applied to steel, titanium, and nickel alloys. High velocity impact of each particle of shot produces a dimple with a region of compression in the treated zone. Typical compressive residual stress distributions reach a maximum approaching the alloy yield strength, and extend to a depth of 0.05 to 0.5 mm. The magnitude of compression achieved depends primarily upon the mechanical properties of the alloy. The depth of the compressive layer and the degree of cold working depend upon the peening parameters,

including shot size and shape, velocity, coverage, impingement angle, etc. Because each shot impacts the surface at a random location, peening for sufficient time to achieve uniform surface coverage results in many multiple impacts producing a highly cold worked surface layer.¹⁻⁵ Gravity shot peening utilizes the same mechanism as conventional air-blast but employs fewer impacts by larger shot, resulting in a less cold worked surface layer. In conventional roller burnishing, a hard cylindrical roller is pressed into the surface of an axisymmetric work piece with sufficient force to deform the near surface layers. Roller burnishing is performed with multiple passes, usually under increasing load for improved surface finish and deliberate cold working of the surface. Fatigue enhancement is attributed to improved finish, increased yield strength due to cold working, and the development of a compressive surface layer. In conventional ball burnishing, a fixed (nonrotating) ball is held in contact with the moving work piece surface under a normal force sufficient to deform the surface of the work piece. A smooth surface is achieved, but with extensive cold work and the potential for surface damage and residual tensile stress. The high friction and shear forces produced can cause surface damage even when lubricants are used.

Laser shock processing (also known as laser peening), an alternative surface processing technology, has been successfully applied for surface enhancement of titanium alloys.^{6,7} Irradiation by a Q-switched high-power laser pulse induces high pressure plasma (several gigapascals), which produces high amplitude shock waves.⁸ To protect irradiated surfaces from thermal rise, sacrificial layers can be used. Irradiating water-immersed surfaces (1 to 10-mm water thickness) enable a factor 5 to 10 intensification of the shock amplitude by

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a trapping-like effect on the plasma, although this method has the practical disadvantage of possible premature water dielectric breakdown and is normally substituted by a water jet curtain method.⁹ Laser shock processing produces a layer of compression of comparable magnitude to shot peening, but much deeper with less cold works without compromising the surface roughness. Moreover, this process is not restricted by component geometry, as is the case with deep rolling.

Recently, experimental results reported by Carreón et al.^{10–12} indicate that the stress-dependence of the thermoelectric power (TEP) in metals produces sufficient contrast to detect and quantitatively characterize regions under compressive residual stress based on their thermoelectric contrast with respect to the surrounding intact material. Also, it was demonstrated that the thermoelectric method is entirely insensitive to surface topography while it is uniquely sensitive to subtle variations in thermoelectric properties, which are associated with the different material effects induced by different surface modification treatments. The goal of this research work is to use two nondestructive thermoelectric techniques, the noncontacting and contacting TEP measurements, to sense the subsurface changes induced by laser peening in a metallic biomedical Ti-6Al-4V alloy.

2 TEP Phenomena

Thermoelectricity is caused by coupled transport of heat and electricity in metals, which leads to a number of interesting phenomena, some of which can be exploited for materials characterization. Essentially, all existing thermoelectric methods are based on the well-known Seebeck effect that is commonly used in thermocouples to measure temperature at the junction between two different conductors. Microstructural characterization and mechanical properties evolution are usually monitored by destructive testing, using tensile tests, metallographic techniques, or hardness measurements. TEP measurements have recently gained a growing attention for the characterization of metallurgical properties in steels and other alloys. These measurements are sensitive to changes in the electronic structure of the material resulting from various metallurgical and mechanical processes.^{10–15} The contacting thermoelectric technique analysis consists of measuring potential differences in the microvolt range, generated by a small temperature difference

along the sample. The Seebeck coefficient, S , is defined as the ratio between the voltage, ΔV , developed along the sample and the temperature difference, ΔT , given by $S = \Delta V / \Delta T$ as shown in Fig. 1(a).

Recently, this method was used in a noncontacting way by detecting the magnetic field produced by thermoelectric currents in metals; this is the called noncontacting thermoelectric technique.^{16–22} Let us assume that we have a defect or imperfection in an otherwise homogeneous material and a temperature gradient is established throughout the specimen. Because of this temperature gradient, different points at the boundary between the defect or imperfection and the host material will be at different temperatures, therefore at different thermoelectric potentials. These minor thermoelectric potential differences will drive local thermoelectric currents around the affected area producing a flux magnetic density given by $\mathbf{B}$, which can be detected in a noncontacting way by a high sensitive magnetometer as shown in Fig. 1(b). This technique was originally suggested for the detection of metallic inclusions in the material but was subsequently shown to be sensitive enough to sense subtle changes in the TEP of metals due to plastic deformation and the presence of residual stresses.^{21,22}

3 Material and Thermoelectric Methods

In this section, the experimental setup and the procedure used to detect and quantify subsurface changes on Ti-6Al-4V alloy by thermoelectric means will be described. A surgical Ti6Al4V ELI (extra low interstitial) alloy was used in this study. Specimens of about $3 \times 3 \text{ cm}^2$ and 7-mm-thick were used. Irradiation by a Q-switched Nd-YAG laser working at the 1064 nm fundamental at 2.8 J/pulse and a 9.4-ns pulse length was performed. Laser pulses with a 1.5-mm diameter at an equivalent overlapping density (EOD) of 5000 cm^{-2} were applied. A continuous film of water was used as confining medium as shown in Fig. 2. The laser power density was about 3 GW/cm^2 . Taking into consideration the intended application and to avoid surface contamination, no sacrifice coating was applied. In order to establish how the laser induced effects individually and collectively affect the thermoelectric measurements, a set of samples was heat treated at 595°C and 710°C for 1 and 2 h, respectively. Such heat treatments are known to induce partial and full release of the residual stresses. For comparative

Fig. 1 Schematic diagram of the thermoelectric power (TEP) measurements by contacting detection using (a) the hot tip technique and (b) the noncontacting detection by magnetic monitoring of thermoelectric currents.

Fig. 2 Schematic diagram of the laser shock peening process.

purposes, samples in the as-machined condition and thermally treated (710°C/2 h) condition were also investigated.

3.1 Thermoelectric Measurements

The contacting TEP measurements were performed using a calibrated ATS-6044T Alloy ThermoSorter (Walker Scientific, Inc., Auburn Hills, Michigan). The operation of this instrument is based on the well-known thermoelectric principle. The thermoelectric instrument induces the temperature difference in the sample by means of a dual-tipped reference probe. One tip is at room temperature and the other is heated to a specific temperature $\approx 50^\circ\text{C}$. In our case, a copper hot tip (standard probe) was used in order to measure the TEP of the laser peened Ti-6Al-4V samples. The dual-tipped probe is placed on the sample, an electric circuit is completed, and a signal is generated. This signal is then processed to obtain a peak reading, which is displayed on the microvolts digital display. The variation in this reading on the laser treated sample is representative of the microstructural changes induced by the plastic deformation process.

On the other hand, in the noncontacting TEP measurements, each sample is mounted into two pure copper supporters, which are perforated by a series of holes and equipped with sealed heat exchangers to facilitate efficient heating and cooling and then mounted on a nonmagnetic translation table for scanning. In order to get a better heat transfer between the specimen and the copper heat exchangers, a layer of silicone heat sink compound was applied. One of the copper supporters is at 15°C , whereas the other is at 50°C . The temperature gradient is kept at $\sim 1.5^\circ\text{C}/\text{mm}$ in all noncontacting TEP measurements, which is more than sufficient to produce detectable magnetic signals on surface treated Ti-6Al-4V samples. A pair of fluxgate sensors is used in a gradiometric arrangement in order to detect the thermoelectric signals from the laser treated zone as shown in Fig. 3. Since the generated magnetic field is perpendicular to both the heat flux in the specimen and the gradient of the material property, the magnetometer was polarized in the tangential direction. The lift-off distance between the primary sensor and the laser treated Ti-6Al-4V sample surface is $\sim 2\text{ mm}$.

4 Results and Discussion

4.1 Microstructural Characterization

Thermal treatment (710°C/2 h) of the as-machined samples was aimed to relieve any subsurface residual stresses associated with the sample preparation. Surface scanning electron microscope (SEM) examination, Fig. 4, reveals that annealing yields the formation of a continuous outer oxide layer consisting of submicron crystals of TiO_2 , as determined by grazing x-ray analysis. This feature is consistent with the well-known oxidation behavior of the alloy.¹²

Interestingly, laser treatment of this surface induces a significant change in the topography that is characterized by different items. At first, SEM evidences the formation of a continuous Ti-dioxide layer that looks like a pancake of melted drops rounded by isolated cavities of small size, likely associated with places where sublimation took place as seen in Fig. 5. Average size of these drops is $2\text{ }\mu\text{m}$, which discards any correlation with the size of the laser spots ($\sim 1.5\text{ mm}$) or grain size of the metal substrate ($\sim 10\text{ }\mu\text{m}$). Occasionally, such cavities are linked by small cracks. Such oxidation is consistent with the short but severe thermal effect associated with the laser processing, which in this case is enhanced due to the absence of the sacrificial layer.

X-ray diffraction of these surfaces reveals the formation of TiO , which is a metastable oxide structure likely

Fig. 3 Diagram of the noncontacting thermoelectric method used for the characterization of laser treated samples.

Fig. 4 Secondary electron images of the surface of (a) as machined surface, and (b) after thermal treatment (700°C/2 h) to release the associated residual stresses.

developed as a consequence of the local high temperature achieved during laser processing and further fast cooling due to the low temperature of the thicker metal substrate. Further annealing of the laser treated samples allow the approach of this oxide to the stable forms, likely to rutile. Such transformation occurs at 595°C for the development of additional features at the surface that resembles the nanocrystals developed prior to the laser treatment. Annealing at 710°C causes the development of a platelets-like oxide decorating the initial oxide drops.

These changes in the topography are also manifested by changes in roughness, Table 1. Roughness of the specimens was determined with a profilometer Mitutoyo Surftest 401 averaging four measurements of 3 cm in length. As can be seen, thermal treatment (710°C/2 h) of the as-machined samples hardly modifies the roughness, which is consistent with the expected thin and continuous oxide layer (<1 µm). Interestingly, laser peening increases the roughness up to about a Ra of 5.5 µm. It worth noticing that Ra is not affected by further annealing. However, secondary electron images (Fig. 6) reveal a roughness increase that is not detected by the profilometer, but that would be of interest for the intended application.

4.2 TEP Measurements

All the laser-treated Ti-6Al-4V samples were tested using the contacting and noncontacting thermoelectric techniques. In the case of TEP measurements with the contacting technique, the TEP value of the laser peened samples, Fig. 7(a), is

Fig. 5 Secondary electron images of the surface of laser peened Ti6Al4V.

higher than that for the as-machined condition without and with thermal treatment (710°C). The TEP values decreased after the stress relaxation treatments at 595°C for 1 h and at 710°C for 2 h. Interestingly, annealing of the laser-treated samples yields values that are similar to those in the as-machined condition, which correlates well with the absence of defects.¹² This means that only lattice defects were developed during processing and they are eliminated after annealing. In the case of magnetic measurements, Fig. 7(b), it was found that the thermal treatment of the as-machined sample hardly influenced the flux density. However, the TEP increased significantly from ~1.5 (as-machined) to ~8 nT (laser peened), which agrees with the expected increase in the residual stresses induced by laser peening. Then, the flux density decreased gradually after the first stress relaxation treatment (595°C/1 h). Finally, on the second stress relaxation treatment (710°C/2 h), the flux density was reduced to a value that it is somewhat higher than the as-machined condition, thus it may be because some residual stresses still remain after the second relaxation treatment or because of the presence of other microstructural changes, such as texture associated to anisotropy of the material.²⁰

In general, the thermoelectric background signature produced by noncontacting TEP measurements depends on the intrinsic anisotropy and inhomogeneity of the material.¹⁶ Ti-6Al-4V specimens often exhibit both anisotropy and inhomogeneity; therefore, the measured magnetic signature is due to a combination of both effects. Whether the actual signature is dominated by anisotropy or inhomogeneity of the specimen can be established on a case-by-case basis by comparing the signatures recorded after rotating the specimen around its principal axes, in our case with respect to the

Table 1 Roughness of the investigated materials.

Sample	Ra (µm)
As machined	1.84 ± 0.05
710°C/2 h	1.70 ± 0.10
Laser Peened (LP)	5.43 ± 0.40
LP + 595°C/1 h	5.67 ± 0.29
LP + 710°C/2 h	5.33 ± 0.31

Fig. 6 Secondary electron images of laser modified samples thermally treated at (a) 595°C/1 h and (b) 710°C/2 h.

Fig. 7 TEP measurements of laser treated Ti-6Al-4V samples by (a) contacting and (b) no contacting means.

rolling direction (0 and 90 deg). Since anisotropic properties are invariant for 180-deg rotations, the true source of the magnetic signature can always be established by repeated measurements at different orientations of the sample. In the as-machined condition Ti-6Al-4V sample, it was found that the shape of the background signature essentially remained the same but flipped its sign when the specimen was rotated by 90 deg, which clearly indicates that the observed signal originates from the anisotropy of the specimen as shown in Fig. 8(a). On the other hand, in laser treated Ti-6Al-4V samples, it was found that the shape of the background signature and its sign essentially remained the same when the specimen was rotated by 90 deg, which clearly indicates that the observed signal cannot originate from

the anisotropy of the specimen unless it is also inhomogeneous as shown in Fig. 8(b). In this research work, the inhomogeneity of the material was indicative of the plastically deformed region due to the laser peening process. The results of this assumption can be used to optimize thermoelectric inspection procedures for flaw detection as well as to develop techniques capable of quantitatively assessing the thermoelectric inhomogeneity of metals.

Investigations were conducted to determine whether the remnant signal of laser treated sample after annealing might be caused by the intrinsic crystallographic anisotropy of the titanium alloy. The magnetic scans of the laser treated Ti-6Al-4V samples, taken at $\nabla T 1.5^\circ C/cm$ temperature gradient, (a) before stress release heat treatment, (b) after stress

Fig. 8 Axial magnetic profile obtained from as (a) machined Ti-6Al-4V sample and (b) laser-treated Ti-6Al-4V sample at a temperature gradient of $1.5^\circ C/mm$ and 2 mm lift-off distance.

Fig. 9 Magnetic signatures recorded on laser-treated Ti-6Al-4V samples (a) before stress release non heat treatment, (b) after stress release heat treatment (710°C/2 h) and for comparison (c) as-received condition (∇ 1.5°C/cm, 2-mm liftoff distance, 28.3×28.3 -mm² scanning dimension)

release heat treatment (710°C/2 h) and for comparative purposes (c) as-machined condition are presented in Fig. 9. The measured peak magnetic flux density is also listed for comparison in all cases. As we expected, the main lobes (thermoelectric currents) get stronger and the magnitude of the magnetic flux increases as work hardening is presented and the spatial distribution of the field become well defined and localized as indicated schematically by two dashed lines outlining the laser treated region [see Fig. 9(a)]. The magnetic image recorded from the as-machined condition Ti-6Al-4V titanium alloy sample exhibits a substantial background thermoelectric signature due to the crystallographic anisotropy [Fig. 9(c)]. In the two phases ($\alpha + \beta$) Ti-6Al-4V alloy, the manufacturing process used to fabricate stock materials (bar, billet, plate etc.) tends to induce a preferred crystallographic orientation due to the restricted nature of mechanical slip (dislocations), leaving the material with a remarkable intrinsic anisotropy. On the laser treated sample, after the second stress release heat treatment [Fig. 9(b)], the magnetic flux density of the base line is ~ 1.86 nT, which is actually larger than the signals produced by as-machined condition sample, which indicates that the background signature is due to residual stresses and/or axial texture caused by the laser peening process. Overall this study reveals that the noncontacting technique is more sensitive to the presence of residual stresses, whereas the contact technique is strongly influenced by the work hardening.¹²

5 Conclusions

In the current research, TEP measurements were applied as a nondestructive, fast and qualitative method to detect subtle material variations produced by laser shock peening in a metallic biomedical Ti-6Al-4V alloy. The TEP measurements were sensitive to the work hardening and residual stresses induced by plastic deformation in the laser treated zone and the contribution of these two effects can be distinguished by the application of the two methods, contact and noncontact TEP. These results indicate that the stress-dependence of the TEP in metals produces sufficient contrast to detect and qualitatively characterize regions under compressive residual stress based on their TEP contrast with respect to the surrounding intact material. However, further research is needed to better separate residual stress effects from secondary material effects, especially in the case of low-conductivity engineering materials like titanium alloys.

Acknowledgments

The authors acknowledge financial support from CONACYT (Mexico) under project CB-80883, MINECO (Spain), under projects MAT2009-14695-C04, MAT 2012-37736-C05-01 and MAT 2012-37782 and CSIC for the scholarship JAE-I3P of S.B.

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