

Thermoelectric assessment of laser peening induced effects on a metallic biomedical Ti6Al4V

H. Carreón¹, S. Barriuso², J. A. Porro³, J. L. González-Carrasco^{2,4}, J. L. Ocaña³

¹Instituto de Investigaciones Metalúrgicas; UMSNH-IIM; Edif."U" C.U., 58000-888, Morelia, MEXICO

²Centro Nacional de Investigaciones Metalúrgicas; CENIM-CSIC, Avda. Gregorio del Amo 8, E28040, Madrid, SPAIN

³Centro Láser UPM. Universidad Politécnica de Madrid, Madrid, SPAIN

⁴Ctr. Investigación Biomédica en Red en Bioingeniería, Biomateriales y Nanomedicina: CIBER-BBN, Madrid, SPAIN

ABSTRACT

Laser peening has recently emerged as a useful technique to overcome detrimental effects associated to another well-known surface modification processes such as shot peening or grit blasting used in the biomedical field. It is worth 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. Moreover, since maximum loads use to occur at the surface, they could also play a critical role in the fatigue strength. 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 in fundamental harmonic at 2.8 J/pulse and with water as confining medium was used. Laser pulses with a 1.5 mm diameter at an equivalent overlapping density (EOD) of 5000 cm⁻² were applied. Attempts to analyze the global induced effects after laser peening were addressed by using the contacting and non-contacting thermoelectric power (TEP) techniques. 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. These results indicate that the stress-dependence of the thermoelectric power in metals produces sufficient contrast to detect and quantitatively characterize regions under compressive residual stress based on their thermoelectric power 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.

Keywords: Laser shock peening; Thermoelectric Measurements; Ti-6Al-4V; Biomaterial

1. INTRODUCTION

Titanium and its alloys are extensively used in the aeronautical industry, medicine and engineering industry 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 alloys components, used as biomaterial, provides means by which the desirable bulk properties are retained in conjunction with enhanced fatigue strength. In addition to, 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. All existing methods of surface enhancement currently available 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 axi-symmetric 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 (non-rotating) 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 pulses induces high pressure plasma (several GPa) which produces high amplitude shock waves. To protect irradiated surfaces from thermal rise, sacrificial layers can be used. Irradiating water-immersed surfaces (1–10 mm water thickness) enables a factor 5–10 intensification of the shock amplitude by 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 [8]. Laser shock processing produces a layer of compression of comparable magnitude to shot peening, but much deeper with less cold work 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 Carreon *et al.* [9-11] indicate that the stress-dependence of the thermoelectric power 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. The goal of this research work is to use two non-destructive thermoelectric techniques (NDTT), the non-contacting and contacting thermoelectric power measurements to sense the subsurface changes induced by laser peening in a metallic biomedical Ti-6Al-4V alloy.

2. THERMOELECTRIC POWER 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. Thermoelectric power (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 [9-14]. The contacting thermoelectric technique analysis consists of measuring potential differences in the micro-volt 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 Figure 1(a). Recently, this method was used in a noncontacting way by detecting the magnetic field produced by thermoelectric currents in metals called non contacting thermoelectric technique [15-21]. 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 non contacting way by a high sensitive magnetometer as shown in Figure 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 thermoelectric power of metals due to plastic deformation and the presence or residual stresses [20,21]

3. MATERIAL AND THERMOELECTRIC METHODS

In this section, the experimental setup will be described and the procedure used to detect and quantified subsurface changes on Ti-6Al-4V alloy by thermoelectric means. A surgical Ti6Al4V ELI (extra low interstitial) alloy was used in this study. Specimens of about 3x3 cm² and 7 mm thick were used. Irradiation by a Q-switched Nd-YAG laser working in fundamental harmonic 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 Figure 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 1h and 2h respectively. Such heat treatments are known to induce partial and full release of the residual stresses. For comparative purposes, samples in the as-machined condition and thermally treated (710°C/2h) were also investigated.

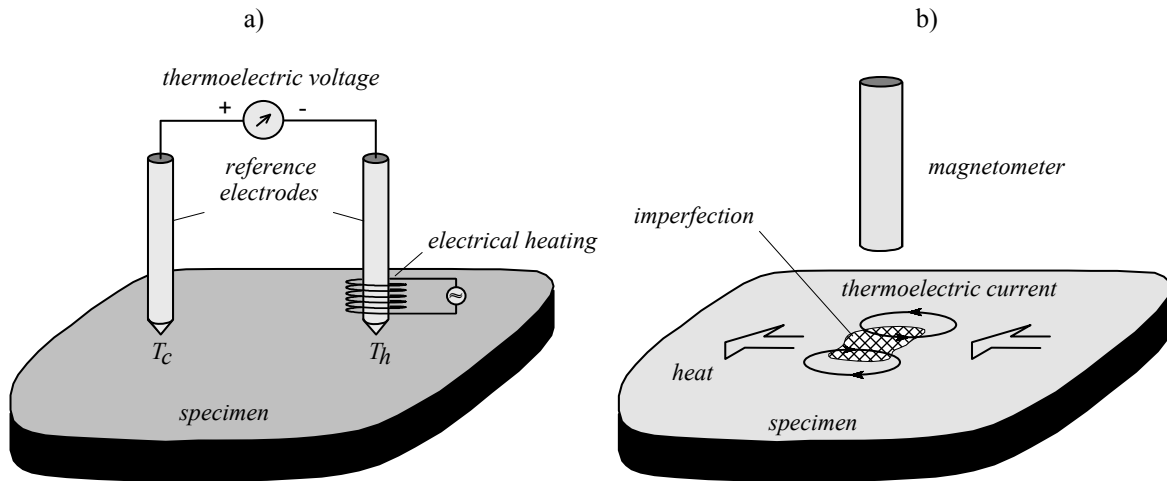


Figure 1. Schematic diagram of the TEP measurements by contacting detection using a) the hot tip technique and b) the noncontacting detection by magnetic monitoring of thermoelectric currents.

3.1 Thermoelectric measurements

The contacting TEP measurements were performed using a calibrated Alloy Thermo-Sorter. 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.

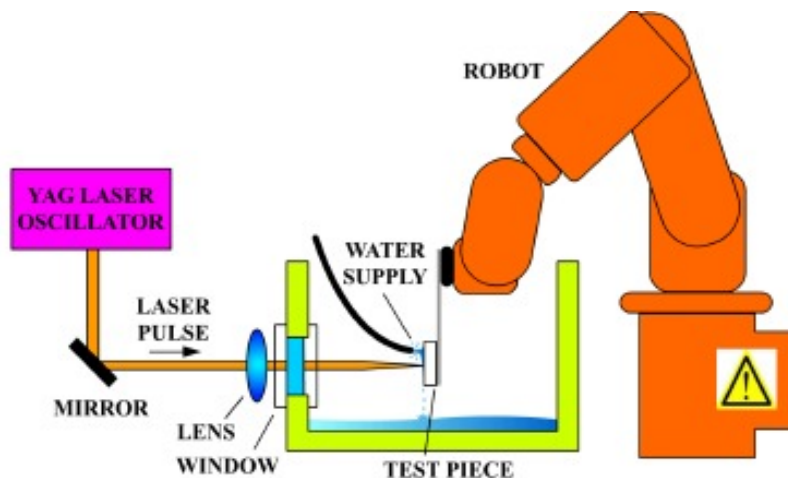


Figure 2. Schematic diagram of the laser peening process.

On the other hand, in the non-contacting 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, while the other is at 50°C. The temperature gradient is kept at ~ 1.5 °C/mm in all non-contacting 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. The inspection of the specimen is realized at the horizontal sensor polarization. The lift-off distance between the primary sensor and the laser treated Ti-6Al-4V sample surface is ~ 2 mm.

4. RESULTS AND DISCUSSION

All the laser treated Ti-6Al-4V samples were tested using the contacting and non-contacting thermoelectric techniques. In the case of TEP measurements with the contacting technique, the TEP value of the laser peened samples, Fig. 3a, is 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 1h and at 710°C for 2h. 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 [11]. This means that only lattice defects were developed during processing and they are eliminated after annealing. In the case of magnetic measurements, Fig. 3b, 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 nT (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 / 1h). Finally, on the second stress relaxation treatment (710°C / 2h), 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 [19].

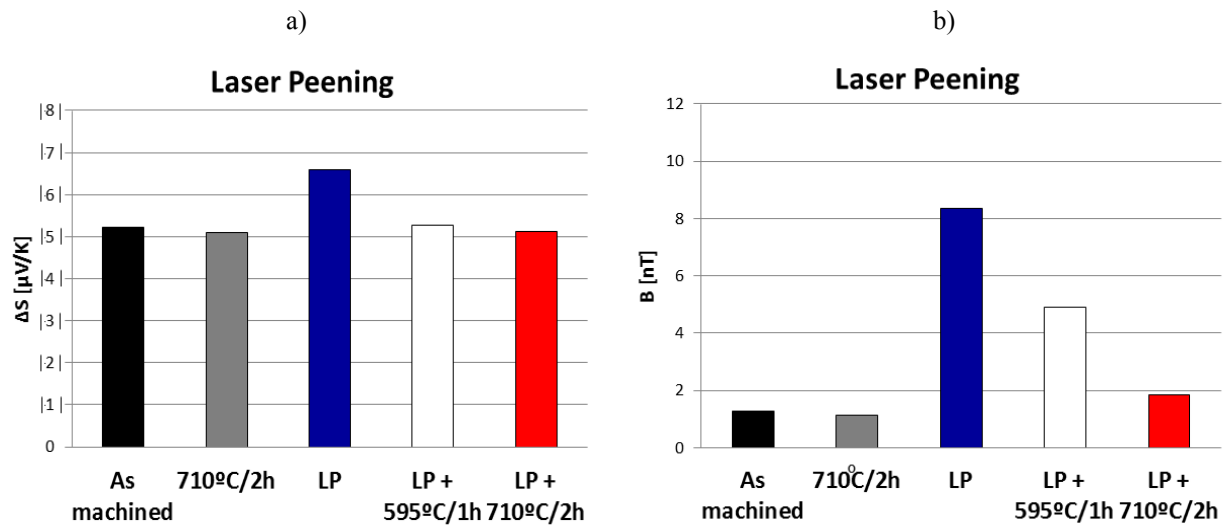


Figure 3. TEP measurements of surface treated Ti-6Al-4V samples by a) contacting and b) no contacting means.

In general, the thermoelectric background signature produced by non-contacting TEP measurements depends on the intrinsic anisotropy and inhomogeneity of the material [15]. 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-to-case basis by comparing the signatures recorded after rotating the specimen around its principal axes, in our case with respect to the rolling direction (0° and 90°). Since anisotropic properties are invariant for 180° rotations, the true source of the magnetic signature can always be established by repeated measurements at different orientations of the sample. In 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° , which clearly indicates that the observed signal originates from the anisotropy of the specimen as shown in Figure 4a. 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° , which clearly indicates that the observed signal cannot originate from the anisotropy of the specimen unless it is also inhomogeneous as shown in Figure 4b. In this research work, the inhomogeneity of the material was represented by the plastically deformed region due to 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.

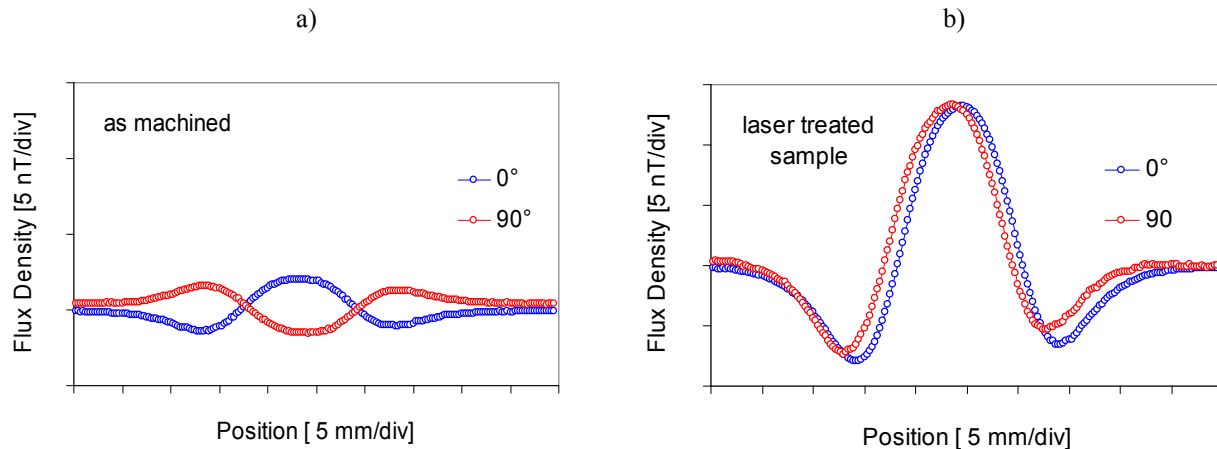


Figure 4. Axial magnetic profile obtained from a) machined Ti-6Al-4V sample and b) laser treated Ti-6Al-4V sample at a temperature gradient of $1.5^\circ\text{C}/\text{mm}$ and 2 mm lift-off distance.

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 $\nabla T \approx 1.5^\circ\text{C}/\text{cm}$ temperature gradient, a) before stress release non heat treatment, b) after stress release heat treatment ($710^\circ\text{C}/2\text{h}$) and for comparative purposes c) as-machined condition were presented in Figure 5. 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 (laser damage zone) as shown in Figure 5a. 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 (Figure 5c). In the two phases ($\alpha+\beta$) Ti-6Al-4V alloy the manufacturing process used to fabricate stock materials (bar, billet, plate ect.) tend to induce a preferred crystallographic orientation due to the restricted nature of mechanical slip (dislocations), leaving the material with a remarkable intrinsic anisotropy. On laser treated sample, after the second stress release heat treatment (Figure 5b), the magnetic flux density of the base line is $\sim 1.86\text{ nT}$, 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 non-contacting technique is more sensitive to the presence of residual stresses, whereas the contact technique is strongly influenced by the work hardening [11].

5. CONCLUSIONS

In the current research, TEP measurements were applied as a non-destructive, 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 so sensitivity 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 non-contact TEP.

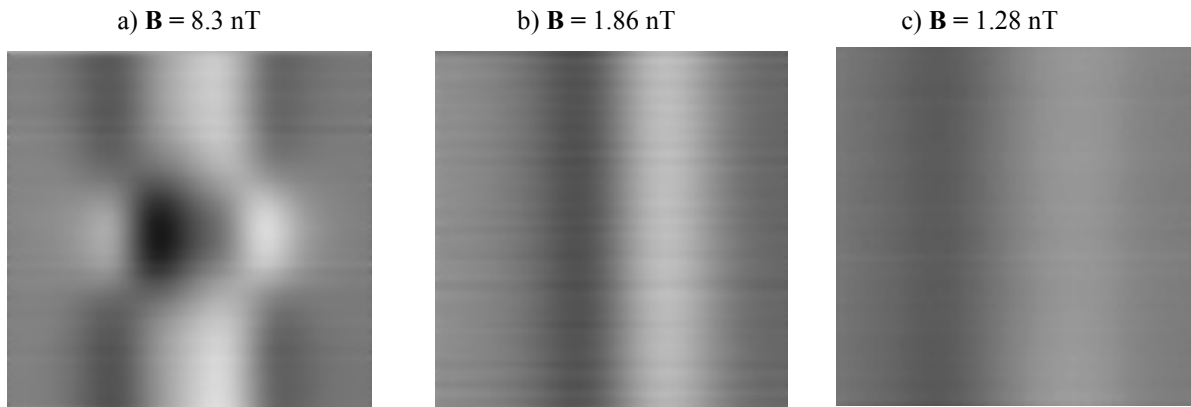


Figure 5. Magnetic signatures recorded on laser treated Ti-6Al-4V samples a) before stress release non heat treatment, b) after stress release heat treatment (710°C/2h) and for comparison c) as-received condition.

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REFERENCES

- [1] H. Hanagarth, O. Vöringer, E. Macherauch, Relaxation of Shot Peening Residual Stresses of the Steel 42 CrMo by Tensile or Compressive Deformation, Shot Peening, Editor K. Iida, The Japanese Society of Precision Engineering, 337-345 (1993).
- [2] W. Zinn, B. Scholtes, Mechanical Surface Treatments of Lightweight Materials - Effects on Fatigue Strength and Near-Surface Microstructures," J. Mat. Eng. & Perf. 8, 145-151 (1999).
- [3] I. Altenberger, Cyclic Deformation and Near Surface Microstructures of Shot Peened or Deep Rolled Austenitic Stainless Steel AISI 304, Mat. Sci. & Eng. A264, 1-16 (1999).
- [4] A. Drechsler, Mechanical Surface Treatments of Ti-10V-2Fe-3Al for Improved Fatigue Resistance, Mat. Sci. & Eng. A243, 217- 220 (1998).
- [5] S.A. Martinez, S. Sathish, M.B. Blodgett, S. Mall, Residual Stress relaxation due to fretting fatigue in shot peened surfaces of Ti-6Al-4V, in: review of progress in quantitative NDE, AIP 22, 1531–1538 (2003).
- [6] I. Altenberger, R.K. Nalla, Y. Sano, L. Wagner, R.O Ritchie, On the effect of deep-rolling and laser-peening on the stress-controlled low- and high-cycle fatigue behavior of Ti-6Al-4V at elevated temperatures up to 550°C, Int. J. Fatigue 44 , 292-302 (2012).
- [7] P. Peyre, L. Berthe, R. Fabbro, X. Scherpereel, E. Bartnicki, Laser shock processing of materials: Characterisation and application of the process, Proc. SPIE 3097, 558-569 (1997).
- [8] J.L. Ocaña, M. Morales, C. Molpeceres, J.A. Porro, A review of the physics and technological issues of high intensity laser shock processing of materials as a method for mechanical properties modification, Proc. SPIE 6346, XVI International Symposium on Gas Flow, Chemical Lasers, and High-Power Lasers, 63461P (April 26, 2007); doi:10.1117/12.738836.
- [9] H. Carreon, P.B. Nagy, Detection of surface imperfections in metals by a noncontacting thermoelectric method, in: 7th ASME NDE Topical Conference, 20, 209–215 (2000).
- [10] H. Carreon, S. Barriuso, G. Barrera, J. L. González, F. G. Caballero, Assessment of blasting induced effects on medical 316 LVM stainless steel by contacting and non-contacting thermoelectric power techniques, Surf. & Coat. Tech. 206, 2942-2947 (2012).

- [11] H. Carreon, S. Barriuso, M. Lieblich, J. L. González, J.A. Jimenez, F. G. Caballero “Significance of the contacting and no contacting thermoelectric power measurements applied to grit blasted medical Ti6Al4V” *Mater. Sci. Eng. C*, 33, 1417-1422 (2013).
- [12] F.G. Caballero, C. Capdevila, L. F. Alvarez, C. García de Andrés, Thermoelectric power studies on a martensitic stainless steel, *Scr. Mater.* 50, 1061-1066 (2004).
- [13] J.P. Ferrer, T. Cock, C. Capdevila, F.G. Caballero, C. Garcia, Comparison of the annealing behaviour between cold and warm rolled ELC steels by thermoelectric power measurements, *Acta Mater.* 55, 2075-2083 (2007).
- [14] X. Kleber, Detection of surface and subsurface heterogeneities by the hot tip thermoelectric power method, *NDT & E Int.* 41, 364-370 (2008).
- [15] H. Carreon, A. Medina, Non-destructive characterization of the level of plastic deformation by thermoelectric power measurements in cold-rolled Ti-6Al-4V samples, *Nondestruct. Test. Eval.* 22, 299-311 (2007).
- [16] W. Morgner, Fundamentals of nondestructive materials characterization, *NDT&E Int.* 27, 263–268 (1994).
- [17] H. Carreon, Thermoelectric detection of the magnetic field by fluxgate gradiometer on subsurface tin inclusions embedded in a copper bar, *NDT&E Int.* 39, 22–28 (2006).
- [18] F. Yu , Detection of rectangular tin inclusion in copper using laser-based self-referencing thermoelectric technique, *NDT & E Int.* 43, 182-189 (2010).
- [19] H. Carreon, Thermoelectric detection of hard alpha inclusion in Ti-6Al-4V by magnetic sensing, *J. Alloys Compd.* 427, 183–189 (2007).
- [20] H. Carreon, P.B. Nagy, M.P. Blodgett, Thermoelectric nondestructive evaluation of residual stress in shot-peened metals, *Res. Nondestr. Eval.* 14 , 59–80 (2002).
- [21] B. Lakshminayanan, H. Carreon, P.B. Nagy, Monitoring of the level of residual stress in surface-treated specimens by a noncontacting thermoelectric technique in: review of progress in quantitative NDE, *AIP* 22, 1523–1530 (2003).