

EFFECT of LSP PARAMETERS on the CORROSION and HARDNESS PROPERTIES of Ti6Al4V.

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Abstract

Because of its low density, high corrosion resistance and high strength to weight ratio, titanium and its alloys are widely used in aerospace applications. As a result, the material is exposed to detrimental environment. An enhancement in surface integrity of the alloy is believed to have the potential to prevent the materials from experiencing premature failure in these aggressive environments. One of the promising surface modification techniques for improving the properties of metals is laser shock peening (LSP), which induces compressive residual stresses in the surface of layer of alloy and results in an increase in strength and fatigue life of the part. In the present study, Laser shock peening is employed to modify the microstructure, mechanical properties and corrosion behaviour of LENS-built titanium alloy components. The surface roughness, microstructural evolution, microhardness and corrosion behaviour of the LENS-built is examined before and after laser shock peening treatment. The effect of LSP overlaps on the corrosion and hardness properties of wrought titanium alloy and LENS-built were investigated. LSP treatment with 90, 95 and 99% overlapping were chosen. The mechanical behaviour of the Ti6Al4V was characterized by material hardness measurements to assess the effect of the peening process on material properties. The microstructure of the peened samples revealed a homogeneous $\alpha + \beta$ phases. Electrochemical tests indicated that LSP improved the corrosion resistance of Ti6Al4V in sodium chloride. In addition, the hardness of Ti6Al4V increased from 375 HV before LSP to 389 HV after LSP. These results demonstrated that LSP is a promising surface modification method that can be used to improve the mechanical properties and corrosion resistance of Ti6Al4V.

Keywords: Laser Shock Peening, LENS, Microstructure, Hardness, Corrosion, passive films

Introduction

Titanium alloys are widely used materials in the aerospace field due to their high mechanical resistance combined with low density and excellent resistance to corrosion (Kanjjer et al. 2017:155). Corrosion resistance of titanium and its alloys in various environments is due to the formation of a protective passive film layer, consisting of titanium dioxide (TiO₂), which contributes to the excellent resistance of the alloy (Shahba et al. 2011:5509; Tamilselvi, Raman & Rajendran: 2006:846). Despite the excellent corrosion resistance of titanium alloys, the material is exposed to localised corrosion attack in aggressive chloride environments which usually results in pitting or crevice formation (Prando et al. 2017:302; Ferdinandov et al. 2018). Therefore it is essential to provide surface protection to the alloy. Numerous studies have been done to improve the mechanical and metallurgical properties such as fatigue life, corrosion

resistance, and wear and erosion resistance properties of titanium alloys utilizing different surface treatment techniques (Gujba & Medraj, 2014:7974, Rozmus et al. 2009). Among the recently advanced surface modification technique is laser shock peening (LSP).

Laser shock peening (LSP) is an advanced laser surface treatment technique to modify surface microstructures and enhance mechanical properties of metallic components (Liao, Ye & Cheng, 2016:24). Compared with the traditional surface modification technologies, the process applies a pulse laser with high power density (in GW/cm^2 range) and ultra-short pulse duration (in nanoseconds) onto the surface of metallic components to induce a high-temperature and high-pressure plasma through a rapidly vaporizing of an ablative layer (a black tape or an aluminium foil) (Sun et al. 2018:265). The generated plasma immediately forms a shock wave transmitting into the target material and intensively interacts with the surrounding materials (Guo et al. 2018:510). The localized plastic deformation induces residual stresses into the surface region of the material. The surface residual stresses are compressive. The induced compressive residual stresses inhibit crack growth under both static and cyclic loading, increasing the material hardness, fatigue life and resistance to stress corrosion cracking (SCC) (Shulda, Swanson & Page, 2014:652).

Laser shock peening is an important post processing technique for metal parts. The electrochemical behaviour of laser peened titanium alloys has been reported in the literatures. Kumar et al. (2019:129) confirmed an improved corrosion resistance of the titanium peened specimens in chloride environment. Geng et al. (2018) also confirmed an improvement of laser shock peening in hot corrosion of TC21 alloy. Tong et al. (2018:40) highlighted that the improved corrosion resistance of the peened specimens is due to compressive residual stresses, crystal defect and the grain refinement. Oxide protective layer of the laser shock peened specimens were noted to be beneficial in the improvement of corrosion.

In this paper, the effect of laser shock peening overlaps on the corrosion and hardness properties of titanium alloy was studied. Corrosion resistance in chloride environment was studied using linear polarization measurement. The surface morphology after corrosion test was also characterised using optical microscopy.

Experimental Setup

Materials and LSP procedures

Table 1: LENS Process Parameters

Laser Power	300 W
Scanning Speed	10.58 mm/s
Laser Energy Density	$249 \text{ J}/\text{mm}^3$

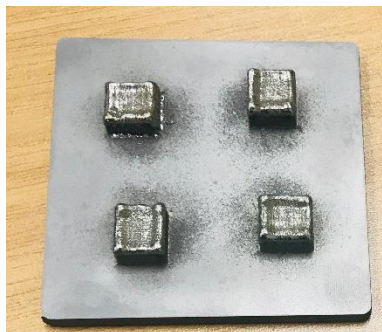


Figure 1: Fabricated LENS-built Ti6Al4V coupons

Ti-6Al-4V Grade 5 alloy was used during LENS building. Prior to fabrication, the substrate's were sandblasted and cleaned with acetone to increase the surface roughness for enhancing adsorption of the laser beam radiation. A LENS machine with a 1 kW IPG fibre laser source was utilized to fabricate the specimens. The LENS system comprised of a sealed processing chamber which is filled with argon to maintain the oxygen and moisture levels below 10 parts per million (ppm) to reduce oxidation during processing. The samples were fabricated using various process parameters as shown in Table 1. The LENS fabricated coupons are shown in figure 1. The LSP processing of the samples was done using Spectra-Physics Quanta-Ray Pro 270 Nd:YAG pulsed laser operating at wavelength of 1064 nm. The medium that was used was flowing water and no protective coating was used. The parameters selected for LSP were as follows: power intensity of 4.46 GW/cm², laser spot diameter of 1 mm and (90, 95 and 99%) overlapping. A total of three samples were laser peened while varying the overlaps. The test samples were clamped down on a XY table.

After laser shock peening treatment, specimens were sectioned on the cross section using titanium blade with the cutting speed of 50 m/s with sufficient amount of water based coolant. The samples were then mounted using hot resin. The surface of the specimens was mechanically grinded using 230, 400, 600 and 1200 grit silicon carbide grinding papers. After grinding, two polishing methods were done on the samples. The samples were first polished on MD-Largo surface with DiaPro Allegro suspension solution and final polishing was carried out using MD-Chem surface with OP-S solution as a suspension until a mirror-like surface was obtained. The samples were etched with Kroll's reagent consisting of 2-3 ml HF, 4-6 ml HNO₃ and 100 ml H₂O. The samples were etched for 10-15 seconds, rinsed with running water, sprayed with acetone and dried off. The samples for corrosion test were mounted using a cold resin while attaching the sample onto the copper wire. The exposed area of the samples was kept at 0.5 x 0.5 cm.

Microstructural Observations

The microstructural evaluation was carried out using Olympus light optical microscope equipped with Analysis ® software. Scanning Electron Microscope equipped with Energy Dispersive X-ray Spectrometry (SEM-EDS) was used for checking microstructure at higher magnification and for the identification of the elements present in the samples.

Microhardness and Surface Roughness

Micro-hardness was measured with Vickers hardness tester Matsuzawa-MMT-X, using a load of 300 g and dwell time of 10 s. Measurements were taken on the longitudinal section of the samples, 84 indents were taken from the top, center and bottom of the substrate while the space between the indentations was kept at 4000 µm.

Surface roughness measurements were done using a MahrSurf PS1 touch probe surface profilometer. The average of a total of five measurements were repeated at each sample.

Electrochemical Studies

The potentiostatic technique was used for determining polarization curves of peened and unpeened titanium alloy. The corrosion tests in 3.65% sodium chloride (NaCl) solution were carried out to express degree of corrosion attack which was verified by SEM analysis. The test was carried out at room temperature of 25°C. The electrochemical tests were performed using a three-electrode system. The fabricated and the as-received Ti6Al4V specimens were used as

the working electrode, a graphite electrode was used as a counter electrode and a silver-chloride (3M KCl) was the reference electrode using a linear polarization method. The specimens were embedded in epoxy resin with a top surface exposed area of 1 cm². The experiment was done using Autolab PGSTAT30 potentiostat equipped with NOVA software. Polarization curves were recorded at a start potential of 1.5 to -1.5 V and at a scan rate of 0.01 mV/s. The corrosion rate was determined using the Tafel extrapolation method using the NOVA software. Using the NOVA software the potentiodynamic polarization curves were plotted and both corrosion rate and potential were estimated by Tafel plots by using both the anodic and cathodic branches.

Results & Discussions

The effect of LSP overlaps on the microstructure, mechanical and corrosion properties of LENS-built titanium alloy are discussed below.

Microstructural Observations, Surface Roughness and Hardness

The optical micrographs in Figure 2 are longitudinal views showing the microstructure of LENS-built titanium alloys components before and after laser shock peening. The microstructure of the samples consists of a refined martensite microstructure consisting of α -phase lamellae contained in β -phase. Columnar grain are the predominant microstructure of the deposited specimens. The increase of laser energy density causes an increase in cooling rate, which results in refinement of the microstructure (Arthur & Pityana, 2018:36). The homogeneous microstructure will guarantee high corrosion stability to the alloy. Both the laser shock peened samples show similar microstructures. However the top region of the peened microstructure is different to the unpeened microstructure. The top region of the peened samples is due to the effect of shock waves which was induced on the sample.

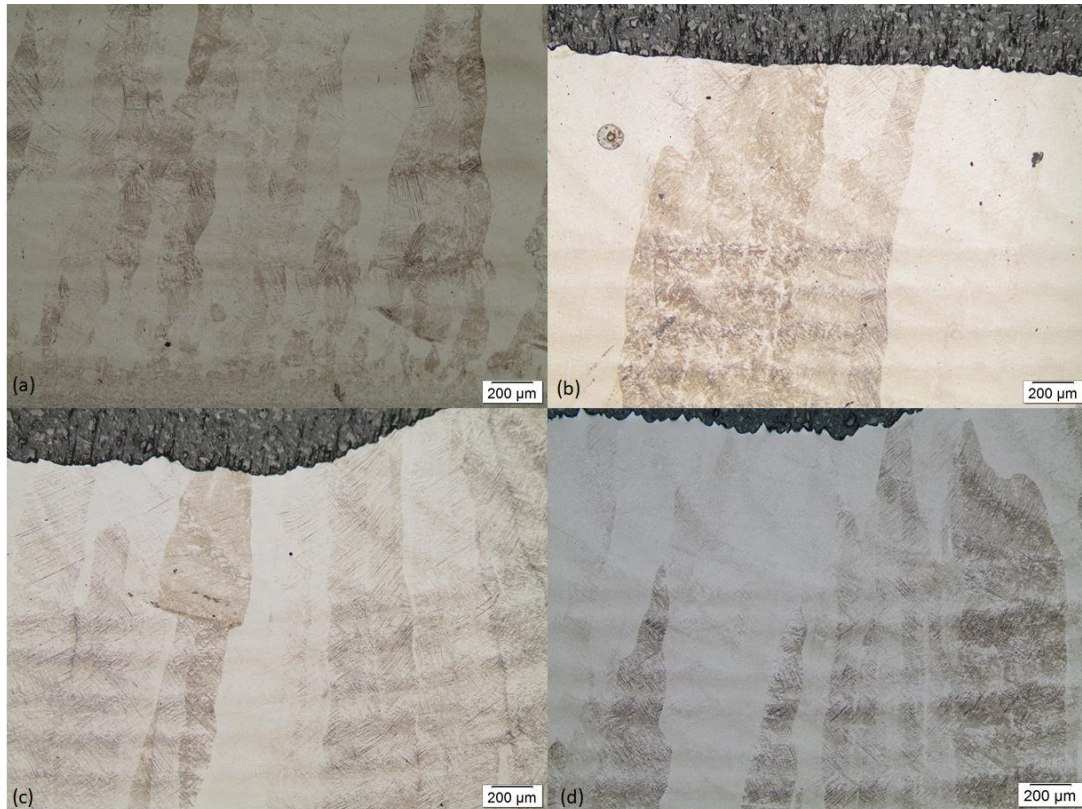


Figure 2: Optical micrographs peened titanium alloys (a) 90%, (b)95% & (c) 99%

Figure 2 represents the surface roughness values measured for the laser peened and unpeened Ti6Al4V samples. As shown in the graph, the arithmetic-mean value (Ra) is considered the representative parameter for measuring roughness. In comparison to the unpeened surface, the laser peened surface registered a small increase in mean surface roughness, Ra from 3.8 μm to 4.6 μm. The improved surface roughness will be beneficial in environment where corrosion is taking place (Newby et al. 2018:102)

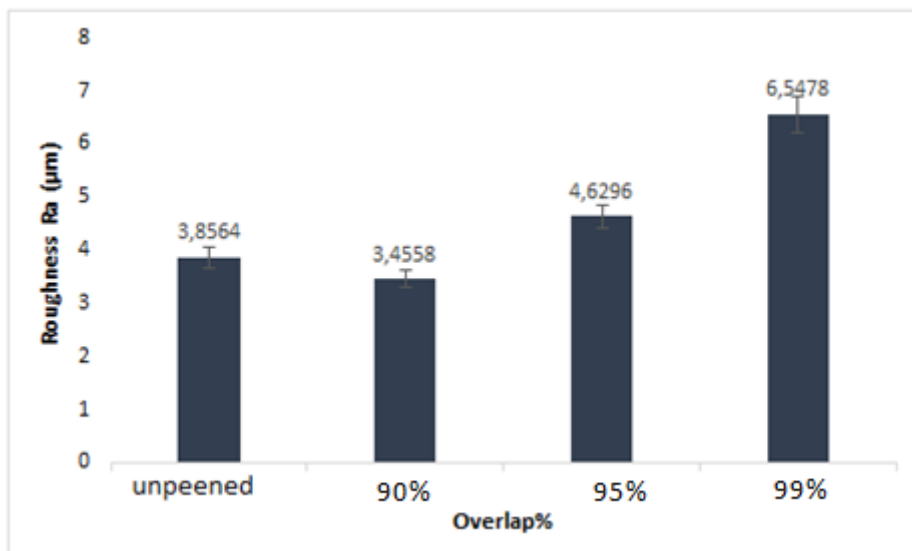


Figure 3: Surface roughness of laser peened samples

Figure 3 shows the microhardness of LSP-treated and untreated Ti6Al4V alloy with increase of the depth from the laser shock peened surface. The hardness profiles depend on laser processing parameters, material types and microstructures of material (Montross et al. 2002:1036). The sample treated at 99% overlap exhibited the highest microhardness value of 389 HV as compared to all the other samples which were 375 HV, 384 HV and 386 HV for the unpeened, 90% and 95%; respectively. Table 1 shows the hardness values of unpeened and peened samples. The peened samples increase with an increasing overlaps; therefore it can be concluded that an increase in peening overlaps increases hardness. According Guo et al. (2018:510) the large enhancement of microhardness after LSP is due to the increase of dislocation density

Table 2: Unpeened and peened samples hardness values, HV 0.3

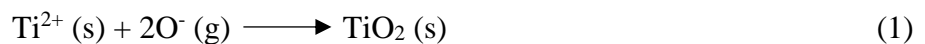
Samples	LENS-built	90%	95%	99%
Max	399	438	427	425
Min	303	356	359	352
Average	375	384	386	389
STD Dev	17	16	12	16

Corrosion studies

Figure 3 shows the polarization curves of LSP-treated and untreated samples immersed in 3.65% NaCl solution. The corrosion potential and corrosion current density were estimated by the Tafel extrapolation method as shown in Table 1. It is observed that peening slightly improves the corrosion resistance of titanium specimen. The roughness and homogeneity of surface are very effective in passive layer formation and also in corrosion resistance of material (Azar et al. 2010:355). According to Cremasco et al. (2008) the type of microstructure in the alloy plays a role on the corrosion behaviour of the material.

Degradation degree of the metallic materials is indicated by the corrosion current densities (i_{corr}). Table 1 shows a decrease of i_{corr} for the peened samples as compared to untreated titanium specimen, which can be the result of formation of passive film on the specimen surface.

The polarization resistance is a quantitative parameter that represents the protection degree of the passive layer (Mareci et al. 2007:856, Vasilescu et al. 2015). From the linear polarization graph it can be observed that the peened specimens registered a higher polarization value of 40932 Ω , 119730 Ω and 122220 Ω for 90%, 95% & 99%, respectively, which according to Rosalbino et al. (2012:37) is due to the formation of passive films such as TiO₂ on the surface. The TiO₂ forms on the surface from the cathodic-anodic reaction cited by Equation 1. Titanium is very resistant in the presence of chlorides due to its protective native TiO₂ oxide film (Vasilescu et al. 2015). The TiO₂ passive films are reported by researchers to be strong mitigation against degradation of the specimen surfaces, such results correlate to other researchers results for passivation films of titanium alloys (Shahba et al. 2011:5509; Tamilselvi, Raman & Rajendran: 2006:846). TiO₂ film is dominant as shown in Figure 5 which confirms why the corrosion resistance of peened samples slightly improved.



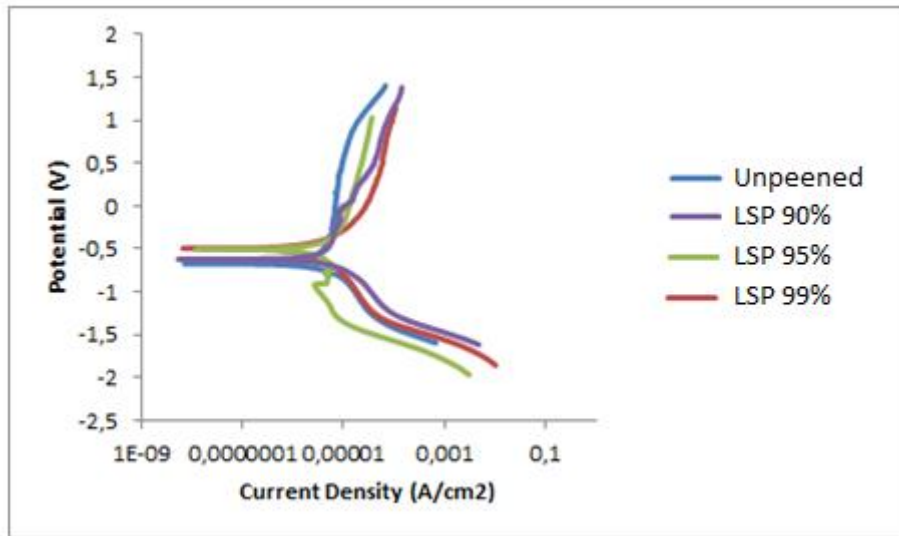


Figure 4: Polarization curves of peened and unpeened titanium alloy samples

Table 3: Linear Polarization Tafel Data plots

Samples	E _{corr} (V)	j _{corr} (A/cm ²)	Corrosion rate (mm/year)	Polarization resistance(Ω)
Unpeened	-0,67224	4,73E-05	0,063	29239
90%	-0,61981	5,11E-06	0,003	40932
95%	-0,59463	3,08E-06	0,004	119730
99%	-0,40481	4,09E-06	0,005	12220

Post-electrochemical microstructure and phases formation

Figure 4 shows optical micrograph images of the surface of the test samples after linear polarization. The unpeened Ti6Al4V specimen shows the presence of pits, whereas in the peened specimen the presence of localized corrosion was not observed. Therefore it can be concluded that the mechanism by which the alloy degrades is due to pitting corrosion.

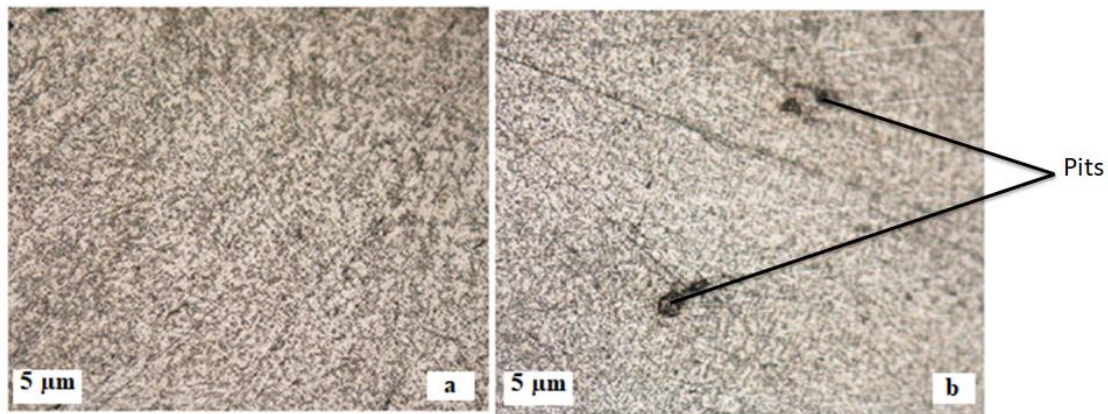


Figure 5: Optical micrograph of the sample surface (a) Peened and (b) unpeened Ti6Al4v samples after linear potentiodynamic polarization

Conclusions

From the above results and discussions, the following conclusions are made:

- Optical microstructures revealed a homogeneous α - β phase observed from LENS fabrication of Ti6Al4V alloy. Very homogeneous microstructures assure high corrosion stability.
- Laser shock peening was demonstrated to have a positive influence on the material properties, as it increased material hardness and was associated with an increase in corrosion resistance.
- Localised pitting corrosion was successfully reduced through the use of laser peening as a post-processing technique. This promoted an increase in surface roughness, which was seen to be beneficial in improving corrosion resistance of the material. Formation of oxide films provided further strength and resistance to corrosion attack on laser peened samples.
- The sample peened using an overlap of 99% had better corrosion and hardness properties.

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