P. DROBNJAK, A. KOVAČEVIĆ, A. MILOSAVLJEVIĆ, Ž. RADOVANOVIĆ, I. SAMARDŽIĆ

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NIMONIC 263 MICROSTRUCTURE AND SURFACE CHARACTERIZATION AFTER LASER SHOCK PEENING

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Preliminary Note – Prethodno priopćenje

The Laser Shock Peening (LSP) is applied to the surface of Nimonic 263 alloy. The changes in microstructure and surface topography are observed and analyzed by Scanning Electron Microscopy (SEM), profilometer and microhardness tester. Various laser regimes are chosen which provoke effects of both mechanical and thermo-mechanical treatments of the sample surface. The optimal process parameters, that result in the finest microstructure, smooth and clean surface, are determined. Some wanted and unwanted phases leading to the crack formation are observed.

Keywords: Nimonic 263, laser shock peening, microstructure, profilometry

INTRODUCTION

The application of laser beams to the material surface in order to transform the material characteristics or to perform micromachining dates back to the beginning of using the laser as a device [1-3]. Laser treatments induce the changes in the microstructure of materials with the aim to improve the mechanical properties. One of the promising methods for the amelioration of the surface characteristics is laser shock peening (LSP) [4]. As a method, the LSP was introduced on the basis of the shot-peening method, using a sudden mechanical blow (pulse) to compress the surface layers, ameliorating mechanical properties of the surface and of the material in general. Rapid (explosive) expansion is enabled by the irradiation with intense pulses of a laser beam producing shock waves on the surface. The transient shock waves induce microstructure changes at the surface, and the microstructure of the material is closely associated with surface topography. Introduced residual stresses affect the fatigue resistance [5], microhardness [6] and grain size [7].

In this work, the LSP processing of Nimonic 263 is applied. By varying the laser parameters, the Nimonic 263 material was exposed to the beam with different values of pulse duration, energy density and fluence. In this way, the spots having different changes in the microstructure are obtained. The microstructure and surface topography of the spots are analyzed and discussed.

The characterization of surface topography of mechanical workpieces with intricate shapes presents a significant challenge. Among many parameters that characterize surface topography, average surface roughness is one of the most important, as it controls the friction, wear, lubricant retention and load carrying capacity, improving fatigue strength, corrosion resistance and creep life, at the same time [8,9]. In this paper, the parameters which directly affect the microstructure and initial crack forming are calculated and discussed (avarage roughness, average maximum profile valley depth, average maximum profile peak height and skewness), as they present suitable places for the stress concentrations.

EXPERIMENT

The investigations have been carried out on nickel based superalloy Nimonic 263 sheets, cold rolled and heat-treated in two stages:

(1) solid solution at 1 150 °C, hold for 1 h and cooled rapidly in the water, and (2) precipitation - treated at 800 °C, hold for 8 h and then air-cooled. In order to make the samples that will be subjected to the laser irradiation, the sheets have been cut in the form of plates, 150 × 150 × 0,7 mm. The chemical composition has been determined by gravimetrical analysis. The surface treatment has been performed by a Nd^{3+}: YAG laser type SWP 5002. The laser specifications are: wavelength 1 064 nm, mean laser power 50 W (max.), pulse peak power 6 kW, pulse energy up to 100 J (max.), pulse duration 0,5 – 50 ms, pulse repetition rate 0,5 – 10 Hz, focal diameter 0,2 – 2 mm.

The diffuse reflectance spectra have been recorded by the Labsphere RSA-PE-20 diffuse reflectance and transmittance accessory, which fits into the sample compartment of the Perkin Elmer Lambda 35 UV-VIS spectrometer.

The experimental setup is shown in Figure 1. The samples have been coated with an absorptive-protective

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P. Drobnjak, High Technical School “Tehnikum-Taurunum”, Belgrade, Serbia, A. Kovačević, Institute of Physics, University of Belgrade, Serbia, A.Milosavljević, Faculty of Mechanical Engineering, University of Belgrade, Serbia, Ž. Radovanović, Faculty of Technology and Metallurgy, University of Belgrade, Serbia, I. Samardžić, Faculty of Mechanical Engineering, Slavonski Brod, University of Osijek, Croatia

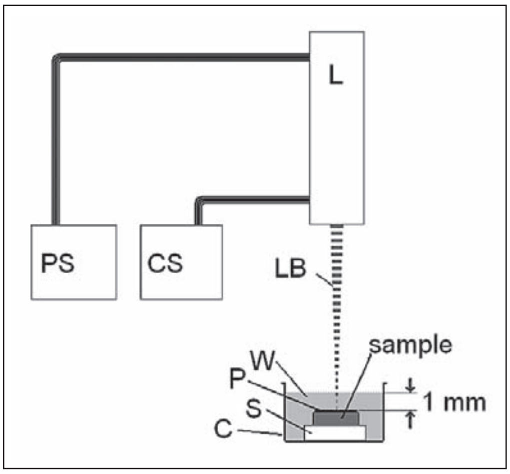

Figure 1 Experimental setup (PS: power supply, CS: cooling system, L: laser, LB: laser beam, W: water, P: protective overlay, S: support, C: container)

layer, a black paint and placed in a container with distilled water (a transparent layer) and exposed to pulsed laser beam. The beam parameters have varied (pulse duration 0,6 – 0,8 ms, energy density 120 – 492 J cm^{-2}, fluence 175 – 615 kW cm^{-2}.

During the experiment, specific values of controllable laser parameters, like voltage, duration and focus, have been chosen for each irradiation, which has yielded specific values of beam parameters at the target (energy density, fluence). The summary of the parameter values is given in the Table 1.

Table 1 **The values of laser and corresponding beam parameters**

Vol. / V	Duration / ms	Focus	Energy density / J/cm^2	Fluence / kW/cm^2
200	0,6	1	120	200,00
200	0,7	2	128	183,04
200	0,8	3	140	175,00
234	0,6	3	252	420,00
234	0,7	1	333	475,71
234	0,8	2	349	436,25
250	0,6	2	340	566,67
250	0,7	3	380	542,86
250	0,8	1	492	615,00

The damages generated by the laser beam have been observed by scanning electron and light microscopies and analyzed by electro-dispersive spectrometry with the JEOL JSM/5800 scanning electron microscope (SEM) as the implemented device. Both the circularity and the grain size of the damages have been measured by using the AutoCad 2009 program and the grain size has been calculated by the circle method [10]. The changes in the surface morphology of the irradiated samples have been determined by the Talystep profilometer and the Zygo NewView 7100 noncontact profilometer. The surface parameters (the average roughness, the average maximum profile valley depth, the average maximum profile peak height and the skewness) have been calculated according to ISO 4287-1997 standard.

RESULTS AND DISCUSSION

The composition of the samples has been determined by gravimetrical analysis and the results are given in Table 2.

The reflectivity measurements have been performed for both the Nimonic 263 surface and the Nimonic 263 surface covered with absorptive layer. The results at 1 064 nm show that, after applying the absorptive layer, the reflectivity significantly dropped from 50,2 % to 9,8 %.

Table 2 **The chemical composition of the Nimonic 263 superalloy / wt %**

Element	C	Si	Mn	Al	Co
	0,06	0,3	0,5	0,5	20
Element	Cr	Fe	Mo	Ti	Ni
	20	0,5	5,9	2,2	bal.

In Figure 2, the damage after the LSP is presented. The processing parameters have been set to: pulse energy density of 492 J cm^{-2}, pulse duration of 0,8 ms and focus “1” (corresponding to the fluence 615 kWcm^{-2}). By visual observation significant differences in the central parts of the images have been noticed. Specifically, the melted material occurred in the middle of the spot, where the pulse energy was the largest. Also, the thermo-mechanical processing created cracks for two reasons: (1) because of the pulse power density and (2) the secondary phases formed due to rapid melting and solidification of materials.

By varying the process parameters, the spots with different diameters have been obtained. The largest diameters correspond to the highest pulse energy densities, but are followed with the material melting. In addition to the pulse energy density, the diameter size is affected by both the pulse duration and focus. The largest diameter without material melting is obtained by good combination of laser process parameters.

The circle method [10] has been used to calculate the grain size. The results confirm that the LSP treatment leads to reduction in grain size.

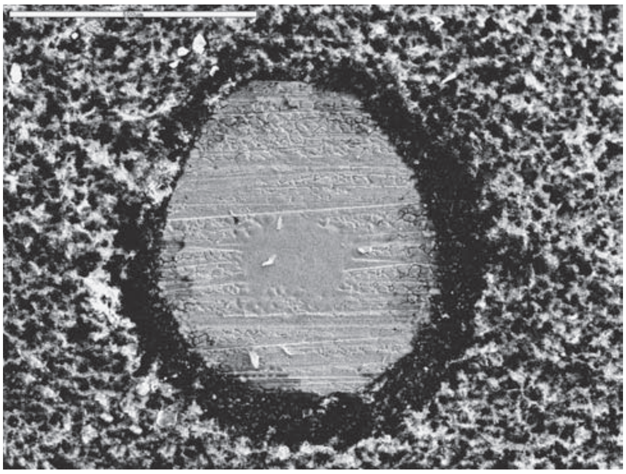

Figure 2 The exposed site after the LSP: pulse duration 0,8 ms, energy density 492 J cm^{-2}. Magnification 50 x

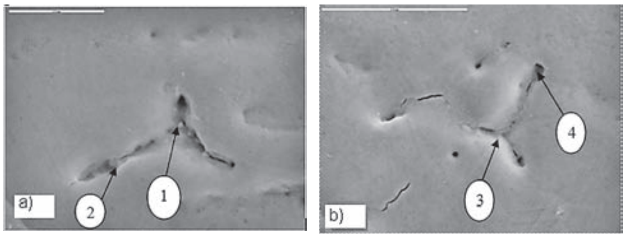

Figure 3 The secondary phases arisen after laser treatment: a) pulse energy density of 340 J cm^{-2}; Magnification 2 500 x; b) pulse energy density of 492 J cm^{-2}; Magnification 3 000 x

In Figure 3, the central parts of the treated spots are presented. Processing parameters are as follows: Figure 3a – pulse energy density of 340 J cm^{-2}, pulse duration of 0,6 ms and focus “2” (fluence ~567 kW cm^{-2}) and Figure 3b – pulse energy density of 492 J cm^{-2}, pulse duration of 0,8 ms and the focus “1” (fluence ~615 kW cm^{-2}).

In Table 3 the results of the Energy Dispersive Spectrometry (EDS) analysis in the spectra denoted in Figure 3 are shown. EDS can reveal microstructural changes indirectly, by comparing the EDS spectra of the non-irradiated area with the spectra of the irradiated area. The analysis leads to the conclusion that the laser interaction causes the formation of secondary phases in thermo-mechanically treated parts. The creation of secondary phases is due to different melting points of the superalloy’s elements.

Table 3 **Results of the EDS analysis (spots 1–4 in Figure 3).**

Elem.	EDS 1 / %	EDS 2 / %	EDS 3 / %	EDS 4 / %
O	15,98	12,93		
Al	5,34	4,92	0,33	
Si	0,52	0,57	0,14	0,22
Ti	0,8	1,03	0,53	0,75
Cr	14,65	14,81	15,13	17,32
Mn	0,47	0,49	0,57	0,62
Fe	0,48	0,45	1,43	1.17
Co	16,58	17,4	22,89	21,54
Ni	41,99	43,93	55,82	55,38
Mo	3,18	3,45	3,16	3

Examining the Table 3 (EDS 1 and 2) and spectra 3 and 4 in Figure 3b, the Al oxides formed with cracks visible around them. In this case, the Al oxides could be the initiators of the cracks and therefore are undesirable.

In Figure 3b unwanted phases are observed. Increased contents of Ni, Fe and Co suggest the creation of sigma phase. This is a typical structural element that affects the mechanical and physical properties of superalloys. However, besides the cracks that formed around unwanted phases, there are cracks caused by unfavourable laser processing parameters, primarily pulse energy and fluence.

By analyzing the all input parameters and conditions under which the secondary phases (and above all – the cracks) formed, it can be concluded that the crack creation is more affected by pulse fluence than the pulse energy density.

The highest average roughness of the laser treated surfaces is obtained by following process parameters: the pulse energy density of 340 J cm^{-2}, pulse duration of 0,6 ms and focus “2” – fluence ~567 kW cm^{-2}. The lowest average roughness is obtained by: pulse energy density of 140 J cm^{-2}, pulse duration of 0,8 ms and focus “3” – fluence ~175 kW cm^{-2}. Pulse duration has the greatest impact, the higher pulse duration the lower average roughness, and increasing the pulse energy density increases the average roughness.

Our results show that laser treatment reduces the surface roughness, and in some cases generates a positive effect on the mechanical properties of materials. The characteristics of the base material, calculated by the Gwyddion software are: average roughness 1,287 μm, average maximum roughness valley depth 3,766 μm, average maximum roughness peak height 3, 317 μm, skewness |0,163|. Calculated characteristics of the material treated with the fluence of ~567 kW cm^{-2} (340 J cm^{-2}, 0,6 ms and focus “2”) are: average roughness 0,394 μm, average maximum roughness valley depth 1,123 μm, average maximum roughness peak height 1,043 μm, skewness |0,201|.

It can be noticed that the surface improved its cleanness, with more regular structure and smaller grains compared to the structure of untreated surface.

As an illustration, Figure 4 shows the surface profiles of the areas after the LSP with parameters: pulse duration 0,7 ms, fluence 380 J cm^{-2}, power density ~543 kW cm^{-2}. The centres of the spots are melted and smoother than the rest of damaged material. In Figure 4, intensity maps and surface profiles in 2D and 3D are included. On both axes distance from the reference point is given in μm.

Microhardness was measured by Vickers HV 0,5 method. The results for the base metal were 305 HV, and for the surface after the LSP 350 – 380 HV. Different laser surface treatment parameters do not play important role for microhardness values.

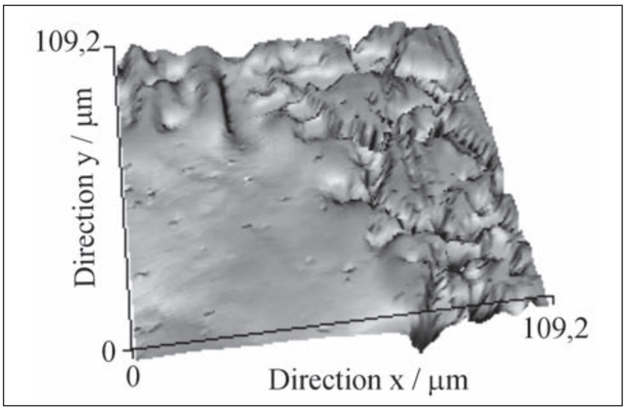

Figure 4 The surface profile taken by the Zigo NewView 7100 profilometer. The laser parameters: pulse duration 0,7 ms, fluence 380 Jcm^{-2}, power density ~ 543 kW cm^{-2}

CONCLUSION

Based on the results presented in this paper, different outcomes have been noticed: as opposed to the laser mechanical treatment, the laser thermo-mechanical treatment has created cracks due to higher power density of the laser beam and forming of unwanted secondary phases. The crack creation is more affected by pulse fluence than the pulse energy density. We have shown that the grain size decreases due to applied LSP. The finest grain structure is produced by lowest energy density with longest pulse durations; the circularity of the spots is most influenced by pulse duration: increasing the pulse duration decreases the circularity. The treatment improved the surface cleanness – the structure became more regular and the size of the grains decreased. Among all the beam parameters, the pulse duration has the greatest impact – the higher the duration, the lower the average roughness. Another beam parameter that influences the average roughness is the pulse energy density. The average roughness increases when the energy density increases. Laser shock peening increases the microhardness of material.

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Note: For English language translation responsible is prof. Martina Šuto, University of Osijek