



Surface EBSD analysis and strengthening mechanism of AISI304 stainless steel subjected to massive LSP treatment with different pulse energies



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ABSTRACT

The effects of massive laser shock peening (LSP) treatment with different pulse energies on surface roughness and microstructural evolution in the surface layer of AISI304 stainless steel were investigated. The deformation-induced grain subdivision processes under two LSP treatment conditions of 3 and 6 J pulse energies were characterized and presented, respectively. Subsequently, EBSD characterization was conducted to analyze the peaks of misorientation angle for as-machined sample and LSPed samples with different pulse energies. Furthermore, a novel MT–MT intersection with four directions was found for the first time in the plastic deformation layer of AISI304 stainless steel, and the generation mechanism was completely presented according to the inherited crystal structure of austenite stainless steel. The formation process of the surface roughness and the formed microstructure subjected to massive LSP impact treatment with different pulse energies were compared and revealed.

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1. Introduction

Austenitic stainless steels cannot be hardened by heat treatment because of low carbon content, and the amount of plastic strain induced strongly depends on the stress required to further deform the material [1,2]. Laser shock peening (LSP), which is also known as laser shock processing, is a surface strengthening technology that uses the mechanical effect of laser shock wave to generate a deeper compressive residual stress layer at the surface of metallic materials and alloys, and thus significantly improves the fatigue lives, wear, and corrosion resistance of metallic components [3–6].

Over the past two decades, studies have reported the effects of LSP on the mechanical properties and microstructures of metal materials. For example, the effects of a single LSP impact on residual stress relaxation and hardness of the LSPed AISI 304 stainless steel (AISI 304SS) were investigated, and the results showed that LSP can improve the distributions of residual stress relaxation and hardness through the generated parallel mechanical twins (MTs) in one direction [7,8]. Similarly, the distribution of micro-hardness and micro-structural morphology for ANSI 321 stainless steel in depth direction were investigated before and after LSP, and the formation mechanism of the dislocation–cell structure in these stainless steels was established [9]. The increase in micro-hardness of the stainless steel subjected to LSP may also be attributed to increasing average dislocation density [10]. In fact, for metallic materials

with low stacking fault energy, deformation twin dominates during plastic deformation [11,12], and the MT thickness and the spacing between adjacent MTs increase with increasing strain rate [13]. Microstructure and fatigue life of dual-phase spring steel subjected to multiple laser peening have been investigated, and results showed that the ultra-high strain induced by laser shock wave was an important factor of grain refinement and plastic deformation in the surface layer of dual-phase spring steel [14]. Moreover, there was a transformation from interlath-retained austenites to martensites during warm laser peening [15].

The abovementioned studies concentrated on mechanical properties and deformation method by severe plastic deformation induced by laser shock wave. MT–MT intersection is the important form to refine the coarse grains of austenitic stainless steel suffering from evident surface treatment technologies, such as ultrasonic surface rolling [16], shot peening [17], and surface mechanical attrition treatment [18]. Considering quantitative research on multiple LSP impacts, we found MTs in the third direction for the first time in our previous work [19,20] and reported the finding in the recent work [21]. We speculated whether MTs aligned in the fourth direction or the *n*th subdivided submicron triangular block into irregularly shaped submicron blocks with large misorientation would appear if the top surface of AISI 304SS was subjected to four or more LSP impacts [19]. Furthermore, the generation mechanism of MTs aligned in three directions is still pending. These proposed problems are worth investigating.

Tri-direction MT–MT intersection dominates during the grain refinement of AISI 304SS, which is induced by multiple LSP impacts. However, during LSP treatment, the relationship between MT direction and slip

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system in austenitic stainless steel was not found in previous reports. In engineering application, massive LSP treatment is an effective method to achieve uniform residual stress field, and different pulse energies result in different areas generating plastic deformation at the surface of metallic components. In addition, LSP treatment causes a coarser surface for austenitic stainless steel [8,22], and surface roughness meeting the requirement of engineering is another issue to consider.

This paper aims to investigate the effects of massive LSP treatment with different pulse energies on the surface roughness and grain refinement process of AISI 304SS. Special attention was given to the subdivision method by MT–MT intersection with multiple directions, and a relationship between MT direction and slip system was established. We revealed the formation of surface roughness and the formed microstructure subjected to massive LSP impact treatment with different pulse energies.

2. Experimental Procedures

2.1. Sample Preparation

The AISI 304SS sample was cut into a rectangular plate with dimensions of 60 mm × 20 mm with a thickness of 3 mm. The chemical composition of AISI 304SS is listed in Table 1. Prior to LSP treatment, all samples were grinded using SiC paper with different grades of roughness (from 150# to 1500#), thoroughly degreased with ethanol, and then rinsed with deionized water by ultrasonic vibration.

2.2. LSP Experiment

Massive LSP treatment was performed with a Q-switched Nd:YAG laser operated by a wavelength of 1064 nm. The focused laser beam presents a diameter of 3 mm and pulse width of 10 ns. Two kinds of pulse energies of 3 and 6 J were selected. During massive LSP treatment, a layer of flowing water with 1–2 mm thick was used as confinement layer to enhance the peak pressure induced by laser shock wave, and the 3M professional aluminum foil (made in USA) with a thickness of 100 μm was selected as the absorbing layer to prevent the thermal effect on the sample surface.

The peak power density can be calculated according to the following formulas:

$$Q_p = \frac{E_p}{(A\tau)} \quad (1)$$

$$A = \frac{\pi d^2}{4} \quad (2)$$

where A is the beam shot area (7.07 mm²), τ is the laser pulse duration (10 ns), and E_p is the pulse energy. Hence, the peak power densities are equal to 4.24 and 8.48 GW/cm² when the pulse energies are 3 and 6 J, respectively.

As shown in Fig. 1, Region A (in gray) and Region B (in light blue) on the AISI 304SS sample surface were treated using massive LSP treatment with pulse energies of 3 and 6 J, respectively. The red point denotes the starting point, and the blue point denotes the end point on the top surface. The scanning path and overlapping rate of massive LSP treatment can be found in the literature [23]. After massive LSP treatment, the treated sample was uniformly cut into three thin plates in depth direction, and the bottom part is regarded as substrate (as-received sample) S1. Subsequently, the top part is divided into two pieces, i.e., the LSPed

region with a pulse energy of 3 J (S2) is on the left, and the LSPed region with a pulse energy of 6 J (S3) on the right. Three kinds of samples are schematically illustrated in detail in Fig. 1.

2.3. Surface Roughness Measurement and Microstructural Characterization

Surface roughness data of all three kinds of samples (S1, S2, and S3) were determined using a true color confocal microscope (Axio CSM 700), and the average value was adopted. The detailed measurement regions were schematically described as A, B1, and B2, as presented in Fig. 1. Typical microstructures of all three kinds of samples (S1, S2, and S3) were observed via transmission electron microscopy (TEM, JEM-2100). For the LSPed samples (S2 and S3), the TEM foils taken from the top surface and a depth of 30 μm below the top surface were prepared. Hence, four TEM foils, namely, B1, B2, C1, and C2, can be schematically presented, as shown in Fig. 1. The electron backscattered diffraction (EBSD) analysis was performed to investigate the misorientation and texture. Before EBSD test, all samples were ground with SiC papers (from 150# to 1500#), mechanically polished with diamond paste with W1.0 and W0.5 and then were finely electrochemical polished for 1 h. Next, EBSD was performed on a FEI Nano SEM Nova 430 equipped with an Oxford Instruments Nordlys 2S detector. The step size is 0.5 μm. The HKL Channel 5 software was used to analyze and display the data. In the present work, the EBSD analyses were conducted in Regions A, B1, and B2.

3. Results and Discussions

3.1. Surface Topography and Roughness

Fig. 2 shows the microstructural topographies of the as-received sample and LSPed samples, including 2D and 3D microstructural morphologies. Fig. 2a and b present the three-dimensional profile of the LSPed samples (S2 and S3) within a measurement region of 579 μm × 400 μm of the top surface, respectively. Abundant shallow and narrow micro-grooves were observed in the top surface of the sample S2, which may inherit fractional surface feature of the as-received sample [24]. After massive LSP treatment with a pulse of 6 J, apart from micro-grooves, several conspicuous scattered micro-bugles can be found. Fig. 2c to e are the two-dimensional surface profiles of three kinds of samples (S1, S2, and S3), respectively. The surface profiles of S2 and S3 with a length of 200 μm are taken from two lines (L1 and L2) perpendicular to the scratch direction, respectively. The surface profile of S1 fluctuates between −3.5 and 3.7 μm, whereas that of S2 fluctuates between −4 and 5 μm. As the pulse energy increases to 6 J, the surface profile varies between −5 and 3.5 μm.

Table 2 presents the average value (AVG) of surface roughness for the as-received sample and LSPed samples within a measurement length (l_r) of 0.80 mm. As seen from Table 2, the surface roughness of the as-received (S1) is 0.536 μm, which is obviously lower than 1.226 (S2) and 0.947 μm (S3) of both LSPed samples. These data are in a good agreement with the measurement values in surface profiles, indicating that massive LSP treatment increases the surface roughness of the as-received sample. The phenomenon is due to the increase in surface waviness [24]. Notably, the surface roughness of S3 is lower than that of S2 (0.947 μm versus 1.226 μm), indicating that higher pulse energy can cause a more uniform surface roughness.

3.2. Micro-structural Evolution After LSP with Different Pulse Energies

Our previous work [19] showed that there was a serve plastic deformation (SPD) layer with a depth of 20 μm and the following minor plastic deformation (MPD) layer along the depth direction of AISI 304SS samples subjected to two LSP impacts. Therefore, in the present work, the TEM images were taken from the top surface to a depth of 30 μm below the top surface of both LSPed samples.

Table 1
The chemical composition of AISI 304 stainless steels (wt%).

Elements	C	Mn	Cr	Mo	Ni	Cu	Si	Nb	Fe
Percent (wt%)	0.06	1.54	18.47	0.3	8.3	0.37	0.48	0.027	Balance

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