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Laser shock peening induced surface nanocrystallization and martensite transformation in austenitic stainless steel

Liucheng Zhou, Weifeng He, Sihai Luo, Changbai Long, Cheng Wang, Xiangfan Nie, Guangyu He, XiaoJun Shen, Yinghong Li *

Science and Technology on Plasma Dynamics Lab, Air Force Engineering University, Xi'an 710038, PR China

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Surface nanocrystallization and deformation-induced martensite in AISI 304 stainless steel subjected to multiple laser shock peening (LSP) impacts were investigated by means of EBSD and TEM observations. A layer of isometric nanocrystalline with a size of 50–300 nm has been formed on the surface after three LSP impacts. And a grain refinement mechanism induced by ultra-high strain rate plastic (>10^7 s^{-1}) deformation during multiple LSP impacts in AISI 304 stainless steel (AISI 304ss) is proposed based on the microstructural observations. Both multidirectional mechanical twins and multidirectional martensites bands led to grain subdivision at the top surface during multiple LSP impacts.

1. Introduction

Laser shock peening (LSP) can significantly improve the fatigue performance of metallic components by forming a considerable residual compressive stress and even grain refinement occurs on the surface of metal through the action of laser shock wave [1–3]. AISI 304 stainless steels (AISI 304ss) are widely used when both high strength and good corrosion resistance are required. The high pressure plasma shock wave was induced by laser irradiation on the materials and caused severe plastic deformation in the material [4]. In this work, a surface nanostructured layer was formed on the AISI 304ss after multiple laser shock peening treatments, and the reason of grain refinement was discussed.

There are a number of reports for the grain refinement mechanism and martensite transformation of AISI 304ss by severe plastic deformation [5,6]. For example, according to Lu et al. [7], the ultra-high strain rate plastic deformation leads to the generation of dislocation lines, dislocation tangles, dislocation walls and mechanical twins (MTs) in the original coarse grains when subjected to multiple LSP. Then the dislocation structures are transformed into subgrain boundaries, which finally evolve into nanoscale grain boundaries through the intersections of mechanical twins and dynamic recrystallization. Chen at el [8]. discussed the deformation mechanism of AISI 304ss subjected to surface impacts over a wide range of strain rates (10~10^5 s^{-1}) and they found that the strain rate between 10 and 10^3 s^{-1} only activated dislocation motions and martensite transformations, resulting in nanocrystallines and ultra-fine grains. However, higher strain rates (10^4~10^5 s^{-1}) produced a high density of twin bundles with nanoscale thickness in the bulk material. Ye et al. [9] investigated the microstructure evolution of AISI 304ss by LSP at room and cryogenic temperature (liquid nitrogen temperature) respectively. They found that a nanostructured surface layer was synthesized after LSP at both room and cryogenic temperature and indicated that the deformation-induced martensite (DIM) was generated by LSP at room temperature only when the laser-generated plasma pressure is sufficiently high (>5.56 GPa). Luo et al. [10] indicated that LSP couldn't cause deformation-induced martensite. These phenomena may be due to the fact that there is an absorbing layer which avoids the thermal effect from heating the surface by the laser beam during LSP. Gerland and Hallouin [11] investigated the microstructure evolution in AISI 304ss by LSP with a very short laser pulse (0.6 ns) and extremely high laser intensity (250–1620 GW/cm^2), and they found that the martensite embryos were formed at the intersections of deformation twins within the pressure range of 15–25 GPa. However, these studies do not agree on the conditions for DIM, neither on the role of DIM for grain refinement. The aim of this paper was to

* Corresponding author.
E-mail address: yinghong_li@126.com (Y. Li).

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reveal the formation mechanism of surface nanocrystallines of AISI 304ss induced by multiple LSP treatment by means of Transmission Electron Microscopy observations (TEM), X-ray Diffraction (XRD) and Electron Back Scattered Diffraction (EBSD), and to study the effect of DIM for grain refinement.

2. Experimental procedure

2.1. Material

Samples were cut by a water jet cutter from a plate (thickness 9.0 mm) of AISI 304ss and the sample was annealed in vacuum condition at 1080 °C for 1 h. Prior to the LSP treatment, the surface of samples should be polished with SiC paper with the roughness ranging from 500 to 2400 to make the surface roughness at Ra 0.3 μm. Chemical composition of 304ss are shown in Table 1.

2.2. LSP experiments

LSP experiments were performed using a Q-switched Nd:YAG laser operating at 1 Hz repetition rate with a wavelength of 1064 nm and the full width at half maximum of the pulses was about 20 ns. The laser beam size was 4 mm and the laser intensities used was 4.3 GW/cm^2. Samples were submerged in a water bath, then processed by LSP. A water layer with a thickness of about 1 mm was used as the transparent confining layer and professional aluminum tape with a thickness of 100 μm was used as the absorbing layer. The application of the protective layer is to protect the surface of the metal from direct ablation and to promote a better coupling with the laser energy [12]. The water confinement is to confine the diffusion of the high temperature plasma produced by laser irradiation and to increase the pressure of shock wave [13].

2.3. Measurement equipment & methods

The microstructure change of the different layers in the treated samples subjected to LSP impacts was characterized by transmission electron microscopy (TEM). The cross-sectional TEM samples were made as follows: both sides of the samples were grinded to make its thickness less than 20 μm. By means of lowering the Ion Milling (Gatan691) from 4.8 kv to 3.2 kv and decreasing the angle from 15° to 4° to prepare the thin zone. This step takes 30 mins. The TEM foils at the surface were prepared by a combination of single and twin-jet electro polishing. The observation equipment is TEM-3010.

The XRD qualitative analysis of phases of 304 alloy before and after LSP is conducted. The XRD analysis was obtained via MFS-7000 X-ray diffraction equipment using Cu-Kα radiation, a take-off angle of 6°. The generator settings were 40 kV and 35 mA. The diffraction data were collected over a 2θ range of 30–80°, with a step width of 0.02° and a counting time of 5 s per step.

Residual stress measurements were performed by a standard X-ray diffraction technique according to the sin2Ψ method in the equipment X-350A, using Cu-Kα radiation, the diffraction plane was {220} and the speed of the ladder scanning was 0.10 s^{-1}. The X-ray beam voltage and electricity were 26.0 kV, 6.0 mA, respectively.

The micro-hardness change of the samples before and after laser shock processing was measured by a MVS-1000JMT2 micro-hardness test machine with a 200 g load and 10s holding time. An average of five measurements was used for each reported data point.

Table 1
Chemical composition of 304ss.

Composition	C	Si	Mn	Cr	Ni	Fe
Percentage (wt.%)	0.08	1.0	2.0	18.0–20.0	8.0–10.5	balance

3. Results and discussion

The changes of microstructure and mechanical performance after different LSP impacts as are shown in Fig. 1.

Fig. 1(a) and (b) demonstrate the residual stress within a surface depth of 1600 μm and the Vickers microhardness within a surface depth of 1000 μm of the 1 and 3 LSP impacted AISI 304ss samples. The dotted lines in these two figures indicate the stress and the hardness of the non-impacted sample, namely, matrix stress and matrix hardness. It is clear that the LSP impact has a powerful effect on the mechanical properties of the AISI 304ss stainless steel, and a significant increase in residual stress and hardness is observed. Compared to 1 LSP impact, the effect of the 3 LSP impacts is stronger. In addition, the material surface has the maximal residual compressive stress and the maximal microhardness. For example, the 3 LSP impacted sample has a maximum compressive stress of 435 MPa and a maximum hardness of 279 HV0.5 in the surface, much larger than those of the non-impacted sample (about 15 MPa and 216 HV0.5, respectively). However, an increased depth gives rise to a sharp reduce in the stress and hardness, and they tend to be stable at a certain depth, 1400 and 800 μm, respectively. The enhanced mechanical properties of the impacted samples can be attributed to the deformation-induced martensite and surface nanocrystallization, as discussed later.

Fig.1(c) shows the TEM image of the surface of AISI 304ss after 3 LSP treatments. It is observed that the laser shock has formed the 30–500 nm nanocrystallites on the surface of the sample. The corresponding diffraction pattern confirms the presence of the nanocrystallites with random orientation, which is dominated by circles, as is shown in Fig.1(d).

Many literature consider that the twining is a prevalent deformation mechanism of AISI 304ss by severe plastic deformation for the grain refinement [4,6]. However, besides mechanical twining, we found that the DIM is an important deformation mechanism for grain refinement in the top surface at ultra-high strain rates in AISI 304ss subjected to multiple LSP impacts. The XRD qualitative analysis of phases of AISI 304ss treated by different impacts is conducted, as is shown in Fig. 2.

Without regard to the influence of instrumental broadening, the Bragg diffraction peak of AISI 304ss have broadened, which indicates that the grain refinement, lattice deformation and the increasing of microstress have occurred in the surface layer of the alloy. Meanwhile, non-processed AISI 304ss only consisted of an austenite phase, but the DIMs took place on the surface layer subjected to different LSP impacts treatment. After LSP with laser intensity of 4.3 GW/cm^2 and single impact, the two peaks (A200 and A220) corresponding to austenite disappeared, while the three peaks (M110, M200 and M211) corresponding to martensite shown up, indicating that the laser shock induced the formation of martensite. In the research of Ye [6], it also found that the DIM of AISI 304ss subjected to LSP, with the laser power density is higher than 5.6 GW/cm^2 was needed. However, in our work, the DIM takes place after single impact with laser intensity of 4.3 GW/cm^2. The number of impacts will also greatly affect DIM. After three impacts, three peaks (M110, M200 and M211) were intensified. On the other hand, the peaks (A111) corresponding to the austenite phase decrease in intensity with an increase in laser impacts, indicating that more and more austenite phase has transformed to martensite at more impacts. The purpose of multiple laser shock impacts is to provide longer time and more energy to plastic deformation and thus induces more martensite, and the volume fraction of martensite also increased.