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Effects of laser shock processing on surface microstructure and mechanical properties of ultrafine-grained high carbon steel

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ABSTRACT

Ultrafine micro-duplex structure (ferrite (α)+cementite (θ)) formed in a pearlitic Fe–0.8C steel after four passes of equal channel angular pressing (ECAP) at 923 K via route Bc. Subsequently the surface of the ultra-micro-duplex structure was treated by multiple laser shock processing (LSP) impacts with different laser pulse energies. The microstructure was obviously refined due to the ultra-high plastic strain induced by LSP. The lattice parameter of α -Fe increased with the laser pulse energy, indicating that the cementite dissolution was enhanced. In addition, residual stress and microhardness increased with the LSP pulse energy.

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1. Introduction

Laser shock processing (LSP) is a new and promising surface treatment technique used to improve fatigue durability [1], corrosion [2] and wear resistance [3], along with mechanical properties of metals and alloys [4]. The generated shock wave can introduce deep compressive residual stresses of several hundreds of MPa by exposing metallic samples to high power density and short pulse laser beam [5-7]. Recently, LSP has been used to harden the surface and improve the mechanical properties of some structural metal components made from aluminum alloys [8-11], nickel-based super-alloys [12], titanium [13] and magnesium alloys [14], stainless steels [15,16] and carbon steels [17,18]. Traditional materials with coarse micrometer-level grains have been studied extensively. However, few studies have focused on the LSP effects on ultrafine-grained materials. Therefore, the aim of this paper is to investigate the effects of LSP pulse energy on microstructure and mechanical properties of the ultrafine-grained materials. The underlying grain refinement mechanism due to plastic deformation after LSP of ultrafine-grained high carbon steel was revealed. The results discussed in this paper provide

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some useful insights for researchers in the field of ultrafinegrained materials surface modification.

2. Materials and experimental procedure

The material used in this study was commercial high carbon steel (Fe-0.8 wt% C). To obtain different pearlite lamellae prior to equal channel angular pressing (ECAP) [19-21], all specimens were annealed at 1273 K for 30 min in vacuum, and then placed into a salt bath furnace at 873 K for 30 min. This was followed by water cooling outside the furnace. The samples were subsequently machined into cylindrical bars, 49 mm in length and 8.3 mm in diameter. Four passes of ECAP were conducted on the samples at 923 K using the Bc route method, in which the specimen was rotated 90° along the longitudinal axis between the passes. An ECAP die was prepared with $\Phi = 120^{\circ}$ and $\Psi = 30^{\circ}$, so that severe deformation could occur during each pass. It is known that the Bc route method is effective in yielding a homogeneous microstructure [22]. Prior to the LSP treatment, sample surface was polished with different grades SiC papers (500-2400), followed by cleaning in deionized water. Ultrasound in ethanol was used to degrease the sample surface, and LSP was conducted shortly after sample preparation.

The LSP experiments were performed using a solid state Nd:glass phosphate laser operating at 1 Hz with a wavelength of 1064 nm, and the full width at half maximum (FWHM) of the pulses was about 10 ns. The spot diameter was 3 mm. Samples

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were submerged in a water bath during LSP processing. A water layer, about 1 mm thick, was used as the transparent confining layer and commercial 100 μ m thick Al tape was used as an absorbing layer to protect the sample surface from thermal effects. The samples were treated by four LSP impacts with different laser pulse energies of 2 J and 6 J. The processing parameters used in LSP are shown in Table 1 in detail. During multiple LSP impacts, the laser beam was perpendicular to the sample surface, which kept the multi-laser effect at the same location of the sample, and the Al tape was replaced after each of multiple LSP impacts.

After different LSP pulse energy treatment, the samples used for metallographic investigation were subjected to several successive grinding and polishing steps. Field-emission scanning electron microscopy (FESEM, QUANTA FEG650) was used for surface morphology analysis. To examine the laser shock processed deformed surfaces, the specimens were etched using 4% Nital solution after LSP. Transmission electron microscopy (TEM, [EM-2010] was used to examine the microstructure of the processed specimens. Thin foil mechanically polished down to 40 µm was utilized for TEM sample preparation by using a double jet electrolytic thinning technique (30 V, 50 mA) in a 93 vol% acetic acid/7 vol% perchloric acid mixture. Liquid nitrogen was used in cooling during the thinning process, with the temperature raising no higher than 243 K. The XRD experiment was carried out on a D8 ADVANCE X-ray diffractometer. The tube voltage and current were 35 kV and 40 mA, respectively. The tube anode was CuK α 1 (λ =0.15406 nm), and the width of the receiving silt was 2 mm. The precise position of each peak was determined with the method "width of the curve at half" [23]. The final lattice parameter of ferrite was obtained through some data treatments such as the extrapolation function and least square method. The surface residual stresses of the samples with and without LSP were determined by using XRD with $\sin^2 \psi$ method. The X-ray beam diameter was about 2 mm. The X-ray source was CrKa ray

Table 1

Processing parameters used in LSP.

Туре	Value
Beam divergence of output (mmmrad) Spot diameter (mm) Pulse energy (J)	≤2 3 2, 6
Pulse width (ns)	< 15
Repetition rate (Hz)	5
Laser wavelength (nm)	1064
Beam profile	Top hat
Pulse to pulse energy stability (% rms)	< 1.5

and the diffraction plane was α phase (211). The feed angle of the ladder scanning was $0.1^{\circ} \, \text{s}^{-1}$. The scanning starting angle and terminating angle were 152° and 160° , respectively. For the measurement of the residual stress along the depth direction, the electropolishing material removal method was used. Microhardness was measured using an MH-3 Vickers microhardness tester with 200 g normal load and 10 s holding time on the aspolished and laser processed regions. An average microhardness value was determined based on five indentation measurements.

3. Results and discussion

3.1. Microstructures evolution

Fig. 1 shows the TEM micrographs of the initial microstructure of the Fe–0.8 wt% C steel before and after ECAP. It can be seen that the initial microstructure before ECAP is fully pearlitic. The thickness of cementite lamellae is about 30 nm, and the average lamellae spacing is about 150 nm, as seen in Fig. 1a. After four ECAP passes, an ultrafine microduplex structure (α + θ), with a grain size at the sub-micrometer level, can be observed in Fig. 1b. The original cementite lamellae disappeared and are completely spheroidized, with about 150 nm average diameter of the cementite particles. Meanwhile, the ferrite grains are equiaxed, with very sharp boundaries and an average grain size of 400 nm. The microstructure of ECAPed ultrafine-grained high carbon steel was investigated and the corresponding grain refinement mechanism initiated by ECAP was explored in reference [24].

Fig. 2a and b shows typical SEM images of the ultrafinegrained steel after LSP with the laser pulse energy of 2 J and 6 J, respectively. After LSP with a low energy of 2 J, the cementite is further refined to smaller grains, as seen in Fig. 2a. The amount of the cementite decreases with the laser pulse energy. From Fig. 2a-d, it can be inferred that the cementite dissolution occurs when the coarse grains are refined under the effect of laser shock wave. This phenomenon is mainly due to dislocations and surface energy effects. The combination force of dislocations in the ferrite and the carbon atoms in the cementite is larger than that of the carbon atom in the cementite. Thus, carbon atoms are pulled out from the cementite into the ferrite by dislocations, which consequently results in the formation of a Cottrell atmosphere. This process can reduce the energy and lead to a partial dissolution of the cementite. After LSP with a high energy, more dislocations are formed, leading to further observed dissolution of the cementite. Thus it can be seen in Fig. 2b that the amount of the cementite is reducing significantly. Fig. 2c and d shows TEM images of the ultrafine-grained steel after LSP with the laser pulse energy of 2 J



Fig. 1. TEM morphology before and after ECAP: (a) pearlite and (b) ultrafine-grained structure after four ECAP passes.



Fig. 2. SEM micrographs of the ultrafine-grained structure with different laser pulse energies: (a) 2 J and (b) 6 J; TEM micrographs with the laser pulse energy of (c) 2 J and (d) 6 J.

and 6 J, respectively. After LSP with the laser pulse energy of 2 J, high density dislocation lines develop in the original grains. Dislocation lines pile-up contributes to the formation of dislocation tangles and dense dislocation cells, as seen in Fig. 2c. With the increasing laser pulse energy, dislocation cells become sharper, forming sub-grains; then continuous dynamic recrystallization occurs in the sub-grain boundaries, resulting in a progressive accumulation of boundary misorientations and finally leading to a gradual transition in boundary character with the formation of high angle grain boundaries [8,15]. In Fig. 2d, the ferrite is refined with the grain size of 150 nm.

Fig. 3 shows the XRD patterns of the ultrafine-grained steel after LSP with different energies. Compared with the ultrafinegrained steel after four passes of ECAP, the α -Fe peaks of the samples after LSP shift to smaller diffraction angles. The higher the LSP pulse energy, the more significant the left shift of the α peaks. The lattice parameter of α -Fe is 0.285776 nm in the ultrafine-grained steel. After LSP with the energy of 2 J and 6 J. the lattice parameter of α -Fe is 0.286311 nm and 0.286590 nm, respectively. The lattice parameter of α -Fe is increased with increasing LSP pulse energies. The carbon content in the pearlite ferrite after different LSP pulse energy can be estimated according to the relationship between the lattice parameter and carbon constant of α -Fe given by Fasiska and Wagenblast [25]. Corresponding change of the carbon content in α -Fe is 0.13% and 0.21% after 2 J and 6 J LSP, respectively. This indicates that the LSP pulse energy affects cementite dissolution quantities. The higher the LSP pulse energy, the larger the dissolution of cementite.



Fig. 3. Changes of XRD peak position of α -Fe in ultrafine-grained structure before and after different LSP pulse energies.

3.2. Residual stress

Residual stress profiles of the samples with and without four LSP impacts with different pulse energies in depth direction are presented in Fig. 4. It can be seen that the untreated regions are



Fig. 4. Residual stress profiles of the samples before and after different LSP pulse energies in depth direction.



Fig. 5. Microhardness of the ultrafine-grained structure after different LSP pulse energies.

approximately in the zero stress state, indicating that the effect of initial stress on the shock waves may be ignored [26]. From Fig. 4, it should be noted that the residual stress increases with the increasing laser pulse energy. After LSP with the laser pulse energy of 2 J and 6 J, the maximum residual stress is -183 MPa and -239 MPa, respectively. High-level compressive residual stress is generated by the LSP impact in the near-surface layer and remains in the compressive residual stress state up to a depth of approximately 670 µm and 780 µm after LSP with the laser pulse energy of 2 J and 6 J, respectively. The maximum compressive residual stress gets located at the treated surface and the value of the compressive residual stress decreases gradually with the increase in the distance to the treated surface. Note that after LSP with the laser pulse energy of 2 J, the increasing rate of surface compressive residual to the treated surface ranging from $0 \,\mu m$ to 250 μm is obviously higher than that when the distance to the treated surface ranges from 250 µm to 670 µm. Similarly, after LSP with the laser pulse energy of 6 J, the increasing rate of surface compressive residual to the treated surface ranging from $0 \,\mu\text{m}$ to 400 μm is obviously higher than that when the distance to the treated surface ranges from 400 μ m to 780 μ m.

After LSP, the compressive residual stress exists in the near surface layer, and the maximum compressive residual stress is located at the treated surface. However, after shot peening the maximum compressive residual stress gets located at the subsurface [27,28]. This difference is mainly due to the thermal effect. An absorbing layer is used during LSP which avoids the thermal effect from heating of the surface by the laser beam, whereas during shot peening, small beads with high velocity hit the metal surface resulting in the thermal effect at the top surface.

3.3. Microhardness

Measured microhardness values of the ultrafine-grained steel near the surface after different LSP pulse energies are shown in Fig. 5. Microhardness increases with the LSP pulse energy. Meanwhile, microhardness in the impact center obviously improved in comparison with the corresponding values at the edge. After LSP with the laser pulse energy of 6 J, the microhardness increased by 30% from 291 HV (before LSP) to 377 HV at the impact center. This is because the stress induced by the shock wave has a Gaussian distribution due to the intrinsic character of the laser pulse energy. After LSP, there are severe plastic deformations in the microstructure of ultrafine-grained steel resulting in the slip and pile-up of high density dislocations, leading to the formation of dislocation cells and pinning of dislocation (Fig. 2c). With increasing LSP pulse energy, the grain is further refined (Fig. 2d). Therefore, after LSP, the surface microhardness increases mainly due to dislocation strengthening and grain refinement.

4. Conclusions

In conclusion, the effects of LSP with different laser pulse energies on the microstructure and mechanical properties of the ultrafine-grained high carbon steel were studied. After four LSP impacts with a laser pulse energy of 6 J, the ferrite can be obviously refined to 150 nm grain diameter. The lattice parameter of α -Fe increases with the laser pulse energy, indicating that the cementite dissolution increases. In addition, the values of surface residual stress and microhardness increase with the LSP pulse energy. After four LSP impacts with the laser pulse energy of 6 J, the maximum residual stress and microhardness were -239 MPa and 377 HV, respectively.

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