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Microstructure and Hardness of Laser Shocked Ultra-fine-grained Aluminum

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Ultra-fine-grained commercial purity aluminum was produced by severe cold rolling, annealing and then straining at ultra-high rate by a single pass laser shock. Resulted microstructure was investigated by transmission electron microscopy. Microhardness of annealed 0.6 μm ultra-fine grained aluminum increased by 67% from 24 to 40 HV. Many 0.3 μm sub-grains appeared at the shock wave center after a single pass laser shock, while high density dislocation networks were observed in some grains at the shock wave edges. Accordingly, microhardness at the impact center increased by 37.5% from 40 to 55 HV. From the impact center to the edge, microhardness decreased by 22% from 55 to 45 HV.

KEY WORDS: Ultra-fine grains; Commercial purity aluminum; Laser processing; Microstructure; Hardness

1. Introduction

Laser shock processing (LSP) is a new technique for improving metal mechanical properties[1–3]. High intensity short pulsed laser beam (I>1 GW/cm^2, τ<100 ns) is used to impact and vaporize opaque sacrificial coating on the metal workpiece. Vapor and plasma absorb the incident laser energy and violently explode against the metal surface. A portion of the energy propagates as a shock wave into the metal. When the shock wave pressure exceeds metal dynamic yield strength, metal deforms plastically and a layer with residual compressive stress forms on its surface[4]. Consequently, metal near-surface microstructure and mechanical properties are modified by LSP[5].

LSP has been utilized for improving surface mechanical properties of structural materials with coarse grains (grain size up to a micrometer), such as aluminum[6], nickel-based super-alloys[7], titanium[8], magnesium alloys[9], stainless[10] and carbon steels[11]. However, only a few reports describe LSP of ultra-fine-grained (UFG) materials (sub-micron grain size). The aim of this work is to investigate microstructure evolution and resulted microhardness of commercial purity UFG aluminum caused by ultra-high strain rates during a single pass LSP.

2. Experimental

2.1 Materials

Commercial purity AA1060 aluminum sample was cut into 30 mm×100 mm×10 mm rectangular shape. The nominal composition (wt%) of Al plates was 0.25 Si, 0.40 Fe, 0.05 Cu, 0.05 Mn, Al 99.50. All specimens with 40 μm original grain size were annealed at 400°C for 1 h. Annealed samples were rolled to 0.95 reduction in 10 passes. Then cold rolled sheets were annealed at 150°C for 1 h. Sample surface was

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polished with different grade SiC paper (500 to 2400 grit), followed by cleaning in deionized water. Ultrasound in ethanol was used to degrease the sample surface, and LSP was conducted shortly after sample preparation.

2.2 Experimental procedure

Solid state Nd glass phosphate laser was used for shock processing operating at 1.064 μm wavelength with 23 ns full width at half maximum (FWHM) pulse. The spot diameter was 7 mm and the laser energy was 20 J. Samples were submerged into a water bath when they were processed by LSP. Water with a thickness of about 3 mm was used as the transparent confining layer to prevent the laser generated plasma from expanding rapidly away from the surface, and 100 μm thick Al tape was used as an absorber to protect the sample surface from thermal effects. The processing parameters used in LSP are shown in Table 1 in detail.

Table 1 Processing parameters used in LSP

Type	Value
Beam division of output/mrad	≤ 0.5
Spot diameter/mm	7
Pulse energy/J	20
Repetition rate/Hz	1
Laser wavelength/nm	1064
Export stability	$\leq \pm 0.5\%$
System ASE energy/mJ	~ 15

After a single pass LSP, the samples used for metallographic investigation were subjected to several successive grinding and polishing steps. Microstructure evolution of different places in the treated samples was characterized by JEM-2010 transmission electron microscopy (TEM) operating at 200 kV. Mechanically polished 40 μm thin foil was utilized for TEM sample preparation using double jet electrolytic thinning technique (30 V, 50 mA) in 75 vol.% methanol/25 vol.% HNO_3 mixture. Liquid nitrogen was used for cooling during the thinning process and the temperature was no higher than –30°C. Microhardness measurements were performed using MH-3 Vickers microhardness tester with 100 g load and 10 s holding time on as-polished and laser processed regions. For each point, an average microhardness value was determined on the basis of the measured data from 5 indentations.

3. Results and Discussion

3.1 Microstructure evolution

TEM micrographs of UFG commercial purity aluminum before and after LSP are shown in Fig. 1. Figure 1(a) shows UFG commercial purity aluminum microstructure before LSP. It can be noted that severe cold rolling followed by annealing leads to dislocations losses and ultra-fine equi-axed sub-grains formation. In some grains, elongated substructures can be found, but dislocation density decreases dramatically as seen in Fig. 1(a). Due to the recovery process, ultra-fine equi-axed grain boundaries are sharp and smooth as seen in Fig. 1(a). Moreover, no dislocations pile-up and serrated grain boundaries are observed in Fig. 1(a). Figure 1(b) and (c) are TEM micrographs of grains at the center of the laser shock wave after LSP. It can be noted that the pile-up of high density dislocations induced by the shock wave contributes to dislocation walls in Fig. 1(c), and dislocation walls contribute to the formation of the subgrain boundaries in Fig. 1(b). Subgrain size is about 0.3 μm in Fig. 1(c). This is consistent with the results reported by Lu *et al.*[12], who investigated LY2 aluminum alloy deformed by multiple step LSP. During LSP, high density dislocation lines develop in the original grains, caused by the shock wave at high strain rate. Dislocation lines pile-up contributes to the formation of dislocation tangles and dense dislocation walls, their transformation into sub-grain boundaries, and the evolution of continuous dynamic recrystallization in the sub-grain boundaries to refined grain boundaries. In this paper, continuous dynamic recrystallization in subgrain boundaries may not take place due to a single pass LSP, so the typical recrystallized grain boundaries are not fully formed, but the microstructural morphologies of subgrain boundaries, dislocation lines and dislocation walls can be clearly observed. Figure 1(d) shows a TEM micrograph of grains at the laser shock wave edge. It can be noted that the microstructure at the wave edge is significantly different from that at the impact center. This is mainly due to the different laser energy distributions during LSP. In addition, it can be clearly seen that the high density dislocation lines are present inside the grains and the dislocation density inside the grains is slightly lower than that at the grain boundaries. The shorter the distance from the grain boundary is, the more obvious the dislocation tangles are, as seen in Fig. 1(d).

3.2 Microhardness

Figure 2 shows the microhardness of the commercial purity aluminum processed under different conditions. Both grain refinement and LSP technique cause microhardness increase. UFG commercial purity aluminum hardness is 40 HV, which is a 67% increase compared with 24 HV of the annealed sample. After LSP, the hardness increases further by 37.5% from 40 to 55 HV at the impact center. Even at the impact edge, the hardness value is 45 HV which is greater by 12.5% than that in the non-shocked region. Meanwhile, from the impact center to the edge, the hardness decreases by 22% from 55 to 45 HV, as shown in Fig. 3. The stress induced by the shock wave has a Gaussian distribution due to the intrinsic character

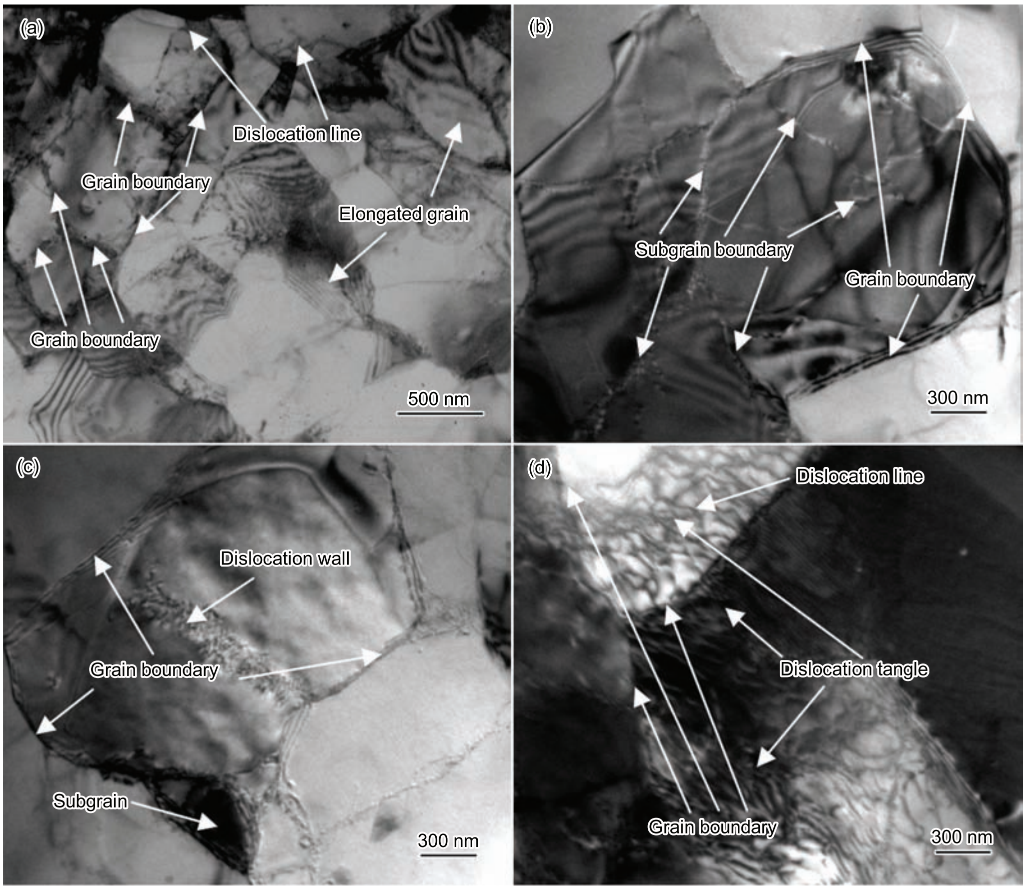

Fig. 1 TEM micrographs of UFG commercial purity aluminum: (a) before LSP, (b) and (c) at the center of the laser shock wave after LSP, (d) at the edge of the laser shock wave after LSP

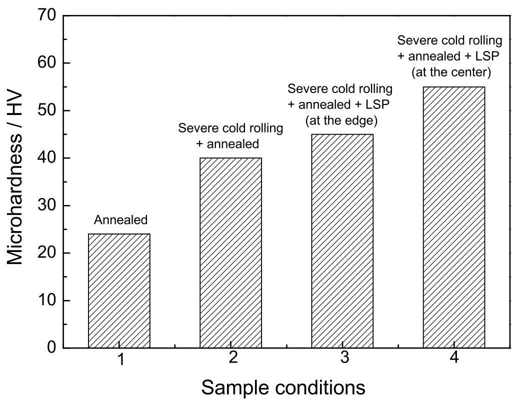

Fig. 2 Microhardness of commercial purity aluminum after different processing conditions

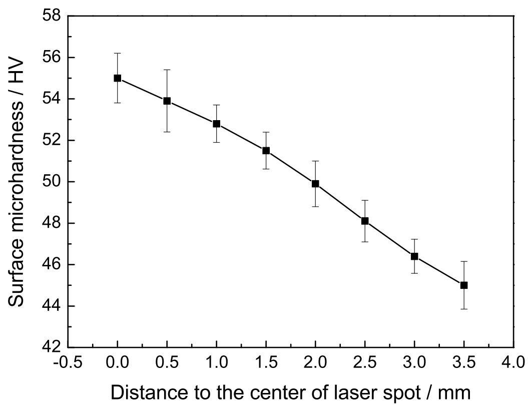

Fig. 3 Microhardness profile on the impact surface

of the laser energy. After LSP, these are severe plastic deformations in the microstructure of UFG commercial purity aluminum, which result in the slip and pile-up of high density dislocations, thus leading to the formation of dislocation walls and pinning of dislocation. Meanwhile, the concentrations of local stress cause the work hardening of the materials and thus increase the microhardness. During ultra-high strain rate loading, the dislocations at the center of the laser shock wave tangle up to form the subgrain boundaries, which finally refine the UFG commercial purity aluminum, as shown in Fig. 1(b) and (c).

Therefore, LSP can improve the microhardness of the UFG commercial purity aluminum, but hardness value slightly increases by 37.5% at the impact center. This is because UFG commercial purity aluminum structure in this study actually recovers rather than recrystallizes after severe cold rolling and annealing, and many sub-structures still remain inside the grains.

As can be seen from Fig. 1(a), in some grains, there are still elongated substructures and some dislocation lines can also be found obviously.

4. Conclusions

(1) The UFG commercial purity aluminum was produced by severe cold rolling and annealing with 0.6 μm ultra-fine grains. Many of sub-grains with 0.3 μm size formed at the center of the laser shock wave after a single pass LSP, while high density dislocation networks were observed in some grains at the edge of the laser shock wave.

(2) Ultra-fine grains obviously contribute to the metal microhardness increase. Compared with annealing, the microhardness of commercial purity aluminum after grain refinement increases by 67%; while after LSP, microhardness at the impact center increases by 37.5% compared with the ultra-fine-grained sample. From the impact center to the edge, the hardness decreases by 22% from 55 to 45 HV.

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