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Improved high cycle fatigue property of ultrafine grained pure aluminum

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1. Introduction

Ultrafine-grained (UFG) materials with the average grain size less than 1 μ m, have received considerable attention over the past decades [1]. The severe plastic deformation (SPD) methods provide various approaches to prepare bulk UFG materials with specific microstructure and significantly enhanced strength controlled by their intrinsic deformation mechanisms [2]. However, SPD UFG materials are prone to recovery and recrystallization during highcycle fatigue (HCF) deformation due to their high density of defects and non-equilibrium high-energy state grain boundaries (GBs), which led to the cycle softening and decreased fatigue ratio compared to their coarse-grained (CG) counterparts [3]. Therefore, achieving a stable microstructure in the UFG materials is the top priority in order to improve the cyclic deformation properties.

Friction stir processing (FSP) is a new thermo-mechanical processing technology, which has been proven to be an effective method of preparing bulk UFG materials with stable microstructures [4,5]. The UFG materials prepared by FSP usually contain high fraction of high-angle grain boundaries (HAGBs), low dislocation density, weak texture and excellent GB stability, which is an ideal model material for the investigation of the cyclic deformation behaviors of the UFG materials [6,7].

ABSTRACT

Ultrafine-grained (UFG) pure Al with uniform and stable microstructure produced by friction stir processing (FSP) significantly increased the high-cycle fatigue (HCF) strength compared with coarse-grained and other UFG materials prepared by severe plastic deformation (SPD). There was no obvious surface damage for FSP-UFG pure Al and the improved fatigue damage resistance can be attributed to the uniform microstructure and high microstructural stability. The ring-island stress distribution of FSP-UFG impeded the formation of large-scale shear bands, and enhanced the coordinated deformation during cyclic deformation. In addition to the enhanced tensile strength, increasing the fatigue strength exponent, which is decided by the microstructural stability, is also an effective method of improving the fatigue strength. © 2020 Elsevier B.V. All rights reserved.

> In this study, UFG pure Al was prepared by FSP and its fatigue behavior was investigated to examine if its HCF fatigue strength can be improved compared to those processed by other SPD techniques and to understand the intrinsic fatigue damage mechanism of the UFG pure Al with a stable microstructure.

2. Material and methods

Commercially pure Al (1060) was used as base material (BM). The plates were FSPed along the rolling direction at a relatively low rotation rate of 100 rpm with a processing speed of 20 mm/ min. The processing tool with a shoulder 10 mm in diameter and a conical threaded pin 3 mm in root diameter and 2 mm in length was used. For comparison, the BM was annealed at 500 °C for 8 h to acquire the CG sample.

Microstructural characterization and analysis were carried out by scanning electron microscopy (SEM), electron backscattered diffraction technique (EBSD) and transmission electron microscopy (TEM). TEM foils were prepared by double-jet electrolytic polishing using a solution of 30 ml HNO₃ and 70 ml CH₃OH at 248 K under a potential of 12 V.

Tensile and fatigue specimens, with a gauge section of 10 mm \times 2.5 mm \times 2 mm, were machined parallel to the FSP direction. Uniaxial tensile tests were carried out at room temperature with an initial strain rate of 1 \times 10⁻³ s⁻¹. HCF tests were performed on an Instron E3000 machine, and a sinusoidal load-time function with a frequency of 60 Hz and a stress ration R = -1 was used.







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3. Results and discussion

Uniform microstructure with equiaxed grains was successfully achieved in the FSP-UFG sample, as shown in Fig. 1a, and the average grain size was refined to about 0.7 µm. The dislocation density was very low in the FSP-UFG Al due to the dynamic recovery and grain refining mechanism during FSP [4]. This is different from other SPD UFG materials, where high density of individual dislocations or dislocation tangles frequently appeared in the grain interior [10]. Moreover, the fraction of HAGBs in the FSP-UFG Al was as high as 88%, and the distribution of misorientation angles was similar to that of the random distribution for a cubic polycrystalline material (Fig. 1b). Different form the LAGB, HAGB was hard to transfer through for dislocations and slip bands [11], which contributed to the microstructure stability under the cyclic deformation.

The tensile and fatigue properties of CG and UFG samples are shown in Fig. 1c and 1d, respectively. CG sample shows a typical continuous work hardening behavior with a low ultimate tensile strength (UTS) of 60 MPa and a low fatigue strength of 22 MPa (Table 1). However, the FSP-UFG sample shows a distinct yielding peak followed by fast strain softening with a high tensile strength of 175 MPa and a high fatigue strength of 60 MPa, nearly three times of that of CG sample. The relationship between stress amplitude $\Delta \sigma/2$ and fatigue life $2N_f$ can be expressed by the following Basquin equation:

$$\Delta\sigma/2 = \sigma_f'(2N_f)^{\nu} \tag{1}$$

where *b* is the fatigue strength exponent, σ'_f is the fatigue strength coefficient. Though the fatigue strength coefficient was closely related to the tensile strength, it has been proven that the fatigue

strength exponent (*b*), decided by the microstructure stability, also played an important role [6,12]. From Table 1, FSP-UFG Al exhibited a higher *b* value, so improved fatigue strength was achieved in FSP-UFG Al, which was higher than other SPD UFG materials [8,9].

Fig. 2 shows the surface damage morphology and the dislocation substructures of CG sample under a stress of 30 MPa and FSP-UFG sample under a stress of 120 MPa. In the CG sample, cracks with length of 100 ~ 200 µm formed along the slip bands (Fig. 2a). By comparison, no obvious cracks, slip bands and other damages were observed in the FSP-UFG sample, as shown in Fig. 2b, indicating the higher microstructure stability than that of other SPD UFG materials where large-scale shear bands formed [13]. After HCF, the CG sample exhibited the typical fatigue dislocation cells (Fig. 2c). For the FSP-UFG sample, no traditional dislocation structures were observed, and only a few dislocation tangles existed at some GBs, as shown in Fig. 2d. Obviously, it is different from traditional grain coarsening mechanism in SPD UFG materials [14,15], and there is no grain growth phenomenon occurred in FSP-UFG sample during fatigue deformation (Fig. 2d).

Fig. 3a describes the relationship between the HCF strength and tensile strength of pure Al samples [8,9,16–18]. It can be seen that the fatigue strength gradually improved with the increase of tensile strength, but declined at high tensile strength (more than 160 MPa) region. The relationship between tensile strength (σ_b) and fatigue strength (σ_W) can be expressed by the following equation [19]:

$$\sigma_W = (C - P \cdot \sigma_b) \times \sigma_b \tag{2}$$

where P represents the sensitive factor, C is a constant. The curves by this equation are shown in Fig. 3a, and all the data followed well with the curves, except for the FSP-UFG sample. The decrease in the fatigue strength at high-strength level can be attributed to various



Fig. 1. (a) TEM microstructure, and (b) misorientation angle distribution of FSP-UFG Al, (c) tensile and (d) fatigue properties of CG and UFG samples [8,9]. CD-UFG: cold drawing UFG material. ECAP-UFG: UFG material fabricated by equal channel angle pressing.

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| atigue properties, tensile properties and grain sizes of pure Al with different processing methods. | |

| Sample | Grain size (µm) | UTS (MPa) | Fatigue strength (MPa) | Fatigue strength coefficient (MPa) | Fatigue strength exponent b |
|-------------|-----------------|-----------|------------------------|------------------------------------|-----------------------------|
| CG | 500 | 60 | 22 | 52 | -0.049 |
| FSP-UFG | 0.7 | 173 | 60 | 244 | -0.085 |
| ECAP-UFG[8] | 1.0 | 162 | 52 | 320 | -0.108 |
| CD-UFG [9] | 0.4 | 212 | 40 | 452 | -0.145 |



Fig. 2. Surface damage morphology and TEM microstructures after fatigue: (a) and (c) CG sample, (b) and (d) FSP-UFG sample.



Fig. 3. (a) Relationship between fatigue strength and tensile strength in pure Al samples [8,9,16–19], (b) KAM analysis of FSP-UFG Al.

structure defects, such as the high density of dislocations and nonequilibrium GBs in UFG materials processed by SPD [14]. However, the FSP-UFG sample contained low density of dislocations and nearequilibrium GBs, which indicate the higher microstructure stability (*b* value), so improved fatigue strength was obtained in the FSP-UFG Al.

Based on EBSD mapping, kernel average misorientation (KAM) analysis has been widely used to qualitatively characterize the change of geometrically necessary dislocations, which can reveal the stress distribution state after cyclic deformation. The KAM distribution of FSP-UFG sample is shown in Fig. 3d, and the higher KAM value represents larger dislocation density. The black lines represent the HAGBs, and the white lines represent the low angle boundaries (LAGBs, $2^{\circ} \leq$ misorientation angle less than 15°), respectively. It can be observed that the grains with high KAM value and low KAM value were alternately distributed showing a ring-shaped distribution characteristic, and the stress distribution also formed a ring-shaped island, which would prevent the defor-

mation being transmitted in the shear direction. This kind of stress distribution was difficult to form large-scale deformation zone, and would lead to an enhanced coordinated deformation effect, suppressing the phenomenon of extrusion of the entire deformed grains. Dislocation activities near the GBs dominated the deformation, even though there was no fatigue-induced grain coarsening which manifested the high GB stability of FSP-UFG material.

4. Conclusions

The HCF properties and the fatigue damage mechanism of FSP-UFG pure Al were investigated. Compared to CG and other SPD samples, the fatigue strength of FSP-UFG sample was essentially increased due to the uniform microstructure and high microstructural stability. The higher fatigue strength exponent (*b* value) was achieved for FSP-UFG pure Al, and manifested the preferable structural stability and HCF damage resistance compared with other SPD UFG materials. The ring-island stress distribution impeded the formation of large-scale shear deformation zone, and led to an enhanced coordinated deformation effect.

CRediT authorship contribution statement

B.B. Wang: Investigation, Data curation, Writing - original draft. **L.H. Wu:** Methodology, Formal analysis. **P. Xue:** Formal analysis, Supervision, Funding acquisition. **D.R. Ni:** Methodology, Formal analysis. **B.L. Xiao:** Methodology, Formal analysis. **Y.D. Liu:** Supervision. **Z.Y. Ma:** Supervision, Methodology, Formal analysis.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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