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#### Original Research

## Precipitation behavior and grain refinement of burnishing Al-Zn-Mg alloy

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#### ABSTRACT

Burnishing is a unique strengthening approach to improve the strength of surface layer and remains the ductility of the interior of metallic materials. In this work, burnishing treatment was employed to improve the surface microstructure of naturally aged Al-Zn-Mg alloys after solid solution. Transmission electron microscopy, high-resolution transmission electron microscopy, X-ray diffraction and nano-indentation were used to characterize the effects of the burnishing on the microstructures of surface layer and Guinier-Preston (GP) zones. It was indicated that GP zones uniformly distributed and dispersed in the matrix before burnishing, and the amount of GP zones decreased dramatically after burnishing processing. Additionally, the grains in the surficial layer were refined into nano-crystals with an average grain size of 78 nm. Burnishing treatment not only led to formation of large number of dislocation substructures in the sub-surface and near-matrix surface, but also promoted the precipitation of metastable  $\eta'$  phase at grain boundaries. The synergistic effects of the grain refinement, dislocation multiplication and the precipitation of  $\eta'$  phase strengthen the burnished layer of Al-Zn-Mg alloy.

#### 1. Introduction

As important structural materials, aluminium alloys especially high strength aluminium alloys have been found wide applications in aerospace and automobile industries due to their light weight, good corrosion resistance and excellent ductility and etc. Recently, the improvement of microstructure and the optimization of mechanical property of the surface layer of aluminium alloys have drawn increasing attention because of their importance during application in industries [1-3]. Severe plastic deformation and mechanical attrition are often employed to produce nanostructure on the surface of metallic materials including aluminium alloys [4-6]. As an important method for severe plastic deformation imposed on the surface of metals and alloys, burnishing treatment is a unique strengthening approach to remarkably improve the strength of surface layer and remain the ductility of the interior of metallic materials [7-10]. As a result, burnishing treatment has drawn increasing attention in researches about the development of metallic structural materials for specific application [10-12].

A lot of studies have been concentrated on developing burnishing technology for improving the mechanical properties of metallic materials. Meanwhile, the relative mechanism for the mechanical behavior response to the burnishing processing has also draw increasing interest of researchers, especially the dependence of mechanical properties on the evolution of microstructures of the materials. In general, severe plastic deformation of alloy surface could give rise to the formation of

gradient structures. Burnishing is such a kind of surficial treatment by which coarse grains in the surface layer of materials could change into nanostructured ones, leading to bimodal [8], multimodal [9] or gradient [10-13] structures.

It has been reported that severe plastic deformation has influence on the precipitation or re-dissolution of the Guinier-Preston (GP) zones in aluminium alloys. For example, some investigations suggested that the GP zones vanish under large deformation at room temperature [14], and some investigations indicated the presence of GP zones and atomic clusters under the same conditions [15,16]. Therefore, it is necessary to probe the burnishing induced microstructural evolution of precipitation in nano-crystal regime. In this study, naturally aged Al-Zn-Mg alloy after solid solution treatment is employed for burnishing treatment. A systematic study is made about the effect of burnishing on the microstructural evolution of precipitation and GP zones in the gradient layers from the surface to the matrix of the alloy. In order for clear illumination, unburnished coarse crystal state of the same alloy was used for a comparative study.

#### 2. Experimental preparation

The composition of Al-Zn-Mg alloy for burnishing treatment is shown in Table 1. Alloy rods with diameter of 80 mm and length of 100 mm were homogenized at 490  $^{\circ}\text{C}$  for 10 h and then immediately quenched into water at room temperature. One rod was subjected to

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Table 1
Chemical composition of Al-Zn-Mg alloy (wt%).

Zn	Mg	Cu	Fe	Si	Al
5.83	2.43	1.55	0.28	0.087	Bal.

natural aging for 6 h, designated as sample A. In order to generate  $\eta'$  phase for comparison, the other rod was aged at 185 °C for 1 h, designated as sample B.

Fig. 1 shows the schematic illustration of the burnishing process [17]. The surface of the natural aged sample A was subjected to burnishing at room temperature with cutting fluid as cooling media. An artificial poly-crystalline diamond was used as burnishing tool head. The burnishing tool head was pressed a certain depth (ap) into the sample surface when burnishing and moved along the sample with a certain feed (f), while the sample rotated with a certain speed (N) on a lathe. The specific burnishing parameters were as follows: the pressing depth ap = 100  $\mu$ m, the feed f = 5  $\mu$ m/round, and the rotation speed of the sample N = 500 r/min.

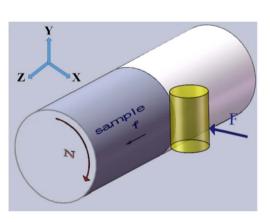
Thin lateral sections of the burnished sample A were truncated at approximately 200  $\mu m$  in thickness. For transmission electron microscopy (TEM) and electron back scattered diffraction (EBSD) observation, the samples were further mechanically thinned using waterproof abrasive paper. To observe the microstructure in burnished surface, the grinding started from the other side. To observe the microstructure in sub-surface and near-matrix-surface of sample A, the burnished surface was thinned by 25  $\mu m$  and 60  $\mu m$  respectively. All the specimens were subsequently thinned only from the other side to the observed side using a Gatan Dual Ion Milling System with an Ar + under cooling by liquid nitrogen. For comparison, unburnished specimens of sample A and sample B were mechanically thinned using the same method to 40–50  $\mu m$ , followed by electropolish in 30% nitric acid solution [18], with temperature range of  $-35\,^{\circ}\text{C}$  to  $-40\,^{\circ}\text{C}$ .

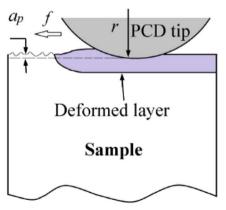
The microstructures of Al-Zn-Mg alloy and the precipitates were characterized by TEM, high-resolution transmission electron microscopy (HRTEM) (JEM-2100F and JEM-2010) and electron backscatter diffraction microscopy (EBSD, Zeiss Ultra 55). The X-ray diffraction (XRD) was carried out using Cu K $\alpha$  radiation at the voltage of 40 kV and current of 200 mA. The nano-indentation tests of the surface, sub-surface, near-matrix and matrix were performed on MTS Nano indenter XP system with a maximum depth of 2  $\mu m$  and an applied strain rate of 0.05 s $^{-1}$ .

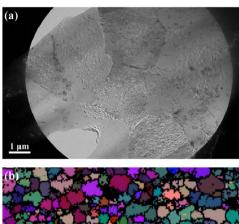
#### 3. Results and discussion

#### 3.1. Observation of microstructures before and after burnishing

Fig. 2(a) and (b) show the TEM and EBSD images of the microstructure of the naturally aged Al-Zn-Mg alloy before burnishing. It







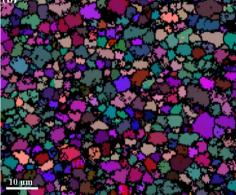


Fig. 2. TEM (a) and EBSD (b) images of Al-Zn-Mg alloy before burnishing.

reveals that the grains of the specimen are coarse, uniform in size, and equiaxial. The grains are generally greater than 3  $\mu$ m in size and possess straight grain boundaries.

Fig. 3(a) shows the TEM image of burnished Al-Zn-Mg alloy on surface layer. It is found that burnishing refines the coarse grains of the alloy into nanocrystals. The nano-crystallized grains exhibit relatively narrow size distribution, with an average grain size of 78 nm and the size range of 40–80 nm accounting for 58%, as shown in Fig. 3(b). Because of the nano-crystalline grains, the selected area electronic diffraction patterns exhibit continuously dotted rings. In this case, dislocation walls, dislocation cells as well as sub-structures are scarcely observed, possibly due to large strain in the surface of the specimen by burnishing. The interactions and tangling of numerous dislocations could lead to the formation of small-angle grain boundaries under large strain. Meanwhile, large strain could transform the small-angle grain boundaries produced by dislocation cells into large-angle grain boundaries [19]. Hence, the grains are refined into nano-crystals, and the dislocation substructures vanish.

The XRD patterns of burnished surface layer and matrix are shown in Fig. 3(c). It can be seen that the XRD peaks of surface are broader than that of matrix. This peak broadening could be attributed to the

Fig. 1. Schematic illustration of burnishing treatment

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