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The effect of initial aging treatment on the microstructure and mechanical properties of cryorolled 6016 Al alloy

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ABSTRACT

The effect of pre-aging and peak-aging treatments prior to cryorolling on the microstructure and mechanical properties of 6016 Al alloys were investigated by hardness measurements, tensile tests, differential scanning calorimetry (DSC) and transmission electron microscopy (TEM). The results showed that high density of dislocation was induced by cryorolling with 80% thickness reduction. The pre-aging treatment enabled the alloy to have stronger work hardening effect during cryorolling and higher precipitation potential during subsequent aging than the peak-aging treatment. This was due to that the preaging enhanced the dislocations accumulating in the cryorolling of pre-aged specimen, and promoted the secondary precipitation during subsequent aging after cryorolling. Meanwhile, the yield strength and ultimate tensile strength of the pre-aged specimen subjected to cryorolling and subsequent aging significantly increased by 72% and 50%, respectively, while the ductility remained similar, as compared to the conventional peak-aged specimen.

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1. Introduction

Precipitation-hardenable 6xxx series Al alloys are extensively used in automotive industries due to their high specific strength. good formability, weldability and corrosion resistance [1–3]. However, further improvement in their strength is desired to expand the applications of the alloys as lightweight structural materials for substituting steels. The mechanical properties of 6xxx series Al alloys can be enhanced by utilizing appropriate thermo-mechanical treatments. In the last decade, rolling at cryogenic temperature was widely used to produce ultrafine-grained microstructures in pure metals and alloys [4-7]. These ultrafine-grained materials with grain sizes less than 1 µm exhibit high strength as expected from the Hall-Petch relationship [8]. It has been recently reported that good combinations of high strength and ductility of precipitationhardenable Al alloys can be achieved by cryorolling and subsequent aging treatments [7,9]. High density of dislocations accumulated from cryorolling due to suppression of dynamic recovery was beneficial to form the ultrafine-grain structures during aging, which enhanced mechanical properties of alloys [9]. In addition, formation of fine precipitates during aging further increased both strength and ductility by pinning and trapping dislocations [10].

Careful control of aging condition is essential for optimizing microstructure of cryorolled Al-Mg-Si alloys due to the complicated

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precipitation sequence of these alloys during aging. The precipitation sequence of Al-Mg-Si alloys is generally considered as $SSSS \rightarrow$ solute clusters \rightarrow GP-zones $\rightarrow \beta'' \rightarrow \beta$ (Mg₂Si) [11,12]. SSSS represents a supersaturated solid solution. Both solute clusters and GP-zones are described as early precipitates. Solute clusters are usually found in naturally aged and low-temperature aged alloys. The spherical GP-zones are thermally more stable than solute clusters and fully coherent with matrix. The needle-like β'' precipitates that form at peak-aged treatment are monoclinic, coherent with matrix along the needle-axis and grow along $< 100 >_{Al}$ [13]. The rod-shaped metastable phase β' present in overaged alloys is hexagonal [14]. The platelet-like equilibrium phase β forms finally in the precipitation sequence and has a cubic anti-fluorite structure [15]. In many earlier studies [16-18], aging was only carried out after cryorolling to modify the microstructure. Moreover, aging temperature has a strong influence on the competition between precipitates hardening effect and softening effect resulting from recovery and recrystallization during aging [19]. It has been proved that only when cryorolled alloys are aged at a relatively low temperature, hardening effect is able to overcome softening effect [19]. The post-cryorolling low-temperature aging treatment has significantly improved the strength and ductility of 7xxx and 2xxx series Al alloys [7,10]. However, it provides limited increment in strength for 6xxx series Al alloys [20]. This might due to the fact that the effect of precipitation hardening in 6xxx series Al alloys is weak at low-temperature aging. Accordingly, control of precipitation during thermo-mechanical treatment is critical for improving the mechanical properties. Buha and Lumley [21,22] reported that when heat-treatable Al alloys were partially aged at a typical T6 temperature, and then held at a reduced temperature, secondary precipitation was expected to occur, leading to increase in tensile properties. From this point of view, if 6xxx series Al alloys are under aged prior to cryorolling, precipitation hardening will be enhanced during post-cryorolling low-temperature aging by secondary precipitation.

In the present study, the 6016 Al alloy subjected to initial aging, cryorolling and further low-temperature aging treatments was investigated. Pre-aging and peak-aging treatments were performed prior to cryorolling with the purpose of comparing their influence on the microstructure and mechanical properties of the alloy during cryorolling and subsequent aging treatments. We also aimed to improve the mechanical properties of the alloy as compared to its conventionally peak-aged counterpart.

2. Experimental

The 6 mm thick hot rolled 6016 Al alloy plates with chemical composition of 1.58 Si, 0.46 Mg, 0.2 Cu, 0.18 Mn, 0.12 Fe, 0.011 Zn, 0.058 Ti, 0.006 Cr (wt%) were used in the present study. The plates were solution treated (ST) at 520 °C for 1 h and then quenched in water to room temperature. After that the specimens were immediately pre-aged (PA) at 180 °C for 10 min and peak-aged (T6) at 180 °C for 15 h, respectively. The PA and T6 specimens were cryorolled (CR) up to 80% thickness reduction. Cryorolling was conducted by dipping the specimens in liquid nitrogen for 30 min before the first rolling pass. After each rolling pass, the specimens were dipped in liquid nitrogen for 10 min before the next rolling pass. The diameter of the rolls was 170 mm and the rolling speed was 22 rpm. In order to improve the mechanical properties, the cryorolled specimens were subjected to further artificial ageing at 100 °C for 96 h.

Hardness was measured on a MH-5L microhardness tester using a load of 500 g with a dwell time of 10 s. Ten readings were recorded to obtain an average hardness value of each specimen. The tensile tests were conducted on a SHIMADZUAG-X50KN computer controlled test machine. The sample with 12.5 mm gauge length was stretched along the rolling direction with a tensile rate of 10 mm/min. For each condition, three parallel specimens were tested in order to verify the experimental results. The microstructural characteristics after different processes were studied by means of differential scanning calorimetry (DSC) and transmission electron microscopy (TEM). The DSC experiments were carried out under an argon atmosphere using a METTLER-1100LF system with a heating rate of 20 °C/min. TEM observation was carried out using Zeiss Libra 200FE TEM operated at 200 kV. Specimens were prepared by electropolishing using a Struers Te-nuPol-5 machine. The electrolyte was consisted of 30% HNO₃ in methanol and kept at a temperature in a range of -25 °C to -35 °C.

3. Results

3.1. Microstructural characterization

The typical microstructures of the PA and T6 allovs are shown in Fig. 1. The bright-field TEM images reveal strain contrast of spherical GP zones in the PA specimen and needle-like β'' precipitates in the T6 specimen, as were observed in the earlier studies [11,23]. GP zones and β'' precipitates are homogeneously distributed and coherent with matrix. The black dots in Fig. 1 (b) should be the β'' precipitates viewed end-on. After cryorolling with 80% thickness reduction of PA and T6 specimens, the severely deformed microstructures are shown in Fig. 2(a) and (b), respectively. The elongated substructures with ill-defined boundaries, which contain high density of dislocation, are observed in both specimens. In addition, the PA specimen after cryorolling shows relatively larger dislocation-tangling zone and higher dislocation density, which indicates that the pre-aging treatment helps to enhance the dislocations accumulating during deformation. Fig. 2 (c) shows the microstructure of the PA specimen subjected to cryorolling and subsequent aging. Compared with Fig. 2(a), numerous elongated substructures have recovered into well-defined subgrains after aging. Meanwhile, the amount of dislocation reduces drastically as a result of extensive recovery.

3.2. DSC analysis

Fig. 3 shows the DSC curves obtained from the ST specimen, as well as from the ST, PA and T6 specimens after cryorolling. The heating rate was kept constant at $20 \,^{\circ}$ C/min. The scan from the ST



Fig. 1. TEM bright-field images of 6016 Al alloys under (a) PA and (b) T6 condition.

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