

## Nanostructure control of age-hardenable Al 2024 alloy by high-pressure torsion

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**Abstract.** A concurrent strengthening process by high-pressure torsion (HPT) and fine precipitation hardening of an Al 2024 alloy has been studied. The HPT was conducted on disks of the alloys under an applied pressure of 6 GPa for 0.75 and 5 turns with a rotation speed of 1 rpm at room temperature. The HPT processing leads to microstructural refinement with an average grain size of ~240 nm and to an increase in hardness up to a saturation after 5 turns. Aging treatment is performed for sample after 5 turns at temperatures of 423 K for a maximum period up to 256 hours. The hardness increased above the hardness level after HPT processing through the subsequent aging. This study thus suggests that simultaneous hardening due to grain refinement and fine precipitation occurred by a combination of HPT processing and subsequent aging at 423 K.

### 1. Introduction

The commercially available Al 2024 alloy is known as a typical age-hardenable alloy with high specific strength, good fracture toughness and excellent fatigue properties. Ringer *et al.* [1] showed that the most probable precipitation sequence of 2xxx alloys in the  $\alpha + S$  phase ( $Al_2CuMg$ ) field is: supersaturated solid solution (SSSS)  $\rightarrow$  solute clusters  $\rightarrow$  Guinier-Preston-Bagaryatsky (GPB) zones + S  $\rightarrow$  S phase. Strength of the 2xxx alloys is improved during artificial aging as rapidly as 60 s by the formation of solute clusters [2]. The strength of metallic materials is also improved by grain refinement through the Hall-Petch relation. It is well established that severe plastic deformation (SPD) process achieves grain refinement regardless of sample states including supersaturation [3]. Horita *et al.* [4,5] studied whether it



is feasible to achieve a combined effect of grain refinement through the SPD process and fine precipitations through the post-SPD aging in an Al-Ag alloy. They showed that the combined effect is realized with a good strength and reasonable ductility. In this study, one of the SPD processes, high-pressure torsion (HPT) is adopted for grain refinement because a finer grain size is attained by intense shear under high applied pressures. The objective of this research is thus to investigate if simultaneous strengthening both by grain refinement and fine precipitation is achieved in the Al 2024 alloy.

## 2. Experimental materials and procedures

The material used in this study was a commercial Al 2024 alloy (4.6% Cu, 1.5% Mg, 0.6% Mn, 0.24% Fe, 0.09% Si, 0.07% Zn, 0.03% Cr, 0.01% Ti and 0.01% Ti+Zr with balance of Al in wt%). The as-received rod with 10 mm diameter was cut into disks of 1 mm thickness. The disks were subjected to solution treatment at 520 °C for 12 hr and then immediately quenched in ice water. HPT processing was conducted at room temperature (RT) under an applied pressure of 6 GPa for two different turns ( $N$ ) as 0.75 and 5. The rotation speed was set to 1 rpm. The thicknesses after 0.75 and 5 turns were reduced to 0.85 mm and 0.8 mm respectively by the HPT-processing. These thicknesses were used later for the calculation of the equivalent strain as attempted in earlier report [6]. The HPT-processed disks were aged at 423 K for periods up to 10 days. The disks were polished to a mirror-like surface and the Vickers microhardness was measured by application of 100 g for 15 s using a Mitutoyo HM-102 tester. Tensile tests were carried out with tensile specimens having dimensions of 1.5 mm gauge length, 0.7 mm width and 0.5 mm thickness. The samples were thinned for transmission electron microscopy (TEM) using a twin-jet electro-polisher in a solution of 25% HNO<sub>3</sub> and 75% C<sub>2</sub>H<sub>5</sub>OH at a temperature of 263 K under an applied voltage of 15 V. Microstructures were observed using a Hitachi H-8100 TEM at an accelerating voltage of 200 kV.

## 3. Results and Discussion

The Vickers microhardness increases with an increasing distance from the disk centre and this increase is more prominent after 5 turns than 0.75 turns as shown in Fig. 1(a). When the hardness is replotted against the equivalent strain as in Fig. 1(b), the hardness increase is well represented by a unique function of the equivalent strain. The hardness increases rapidly at the beginning of straining and reaches a steady state level where the hardness remains unchanged at a constant level of  $H_v \approx 250$  HV with further straining.

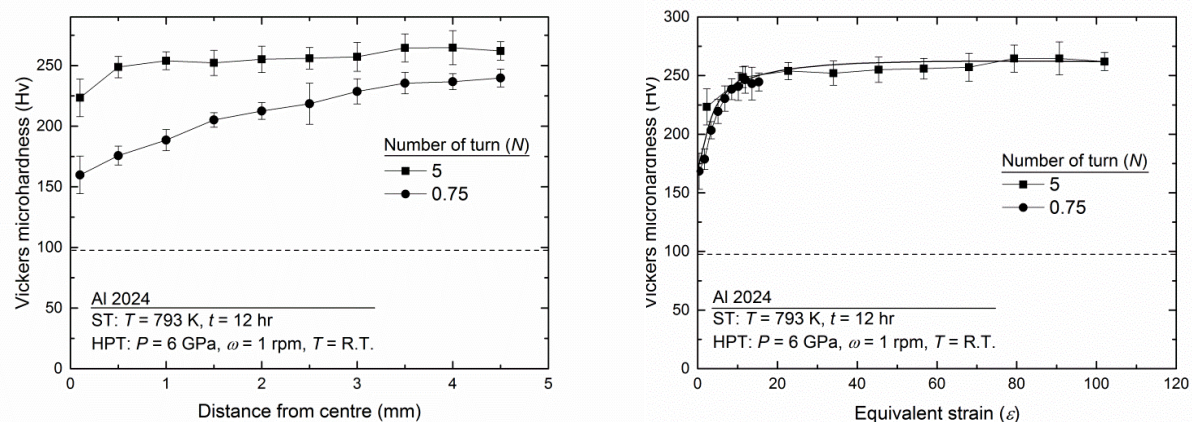


Fig. 1 Vickers microhardness vs (a) distance from disk centre and (b) equivalent strain.

When aging was undertaken at 423 K, an appreciable increase in hardness is realized for the HPT-processed sample at each of the different states of equivalent strain as shown in Fig. 2. For the all states of equivalent strain, the hardness increase exhibits almost the same trend with respect to the aging time.

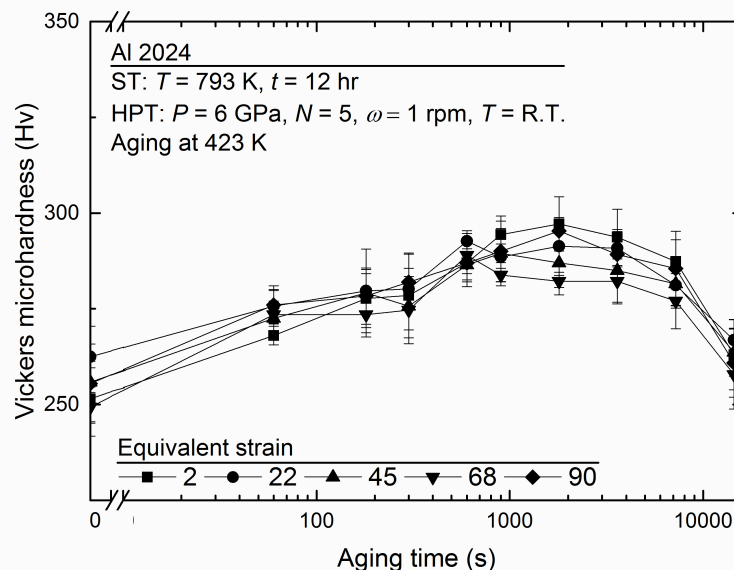


Fig. 2 Aging behavior of Al 2024 after HPT processing.

Fig. 3 shows a series of bright-field images (left) and dark-field images (centre) and the corresponding SAED patterns (right) after HPT processing of the samples at two different states with equivalent strains of (a)  $\epsilon \approx 0.3$  and (b)  $\epsilon \approx 70$ . The arrows in the SAED patterns indicate diffracted beams used for taking the dark-field images. At the initial straining stage, a

high density of dislocations is present within the grain as seen in the dark field image in Fig. 3(a). As the hardness increases to the saturated level, the grain size becomes smaller and the SAED pattern exhibits a ring-like form as in Fig. 3(b). The evolution of the SAED pattern from a web-like in Fig. 3(a) to a ring-like in Fig. 3(b) indicates an increase in the misorientation angles between neighbouring grains with straining. The TEM observation in Fig. 3 thus confirms that grain refinement is achieved to a grain size of  $\sim 240 \pm 80$  nm. When the sample after  $N = 5$  was aged at 423 K for 4 h, superlattice spots are visible in the SAED pattern as marked by s in Fig. 3(c). This indicates that precipitates of S-phase ( $\text{Al}_2\text{CuMg}$ ) contributed to the hardening of the alloy.

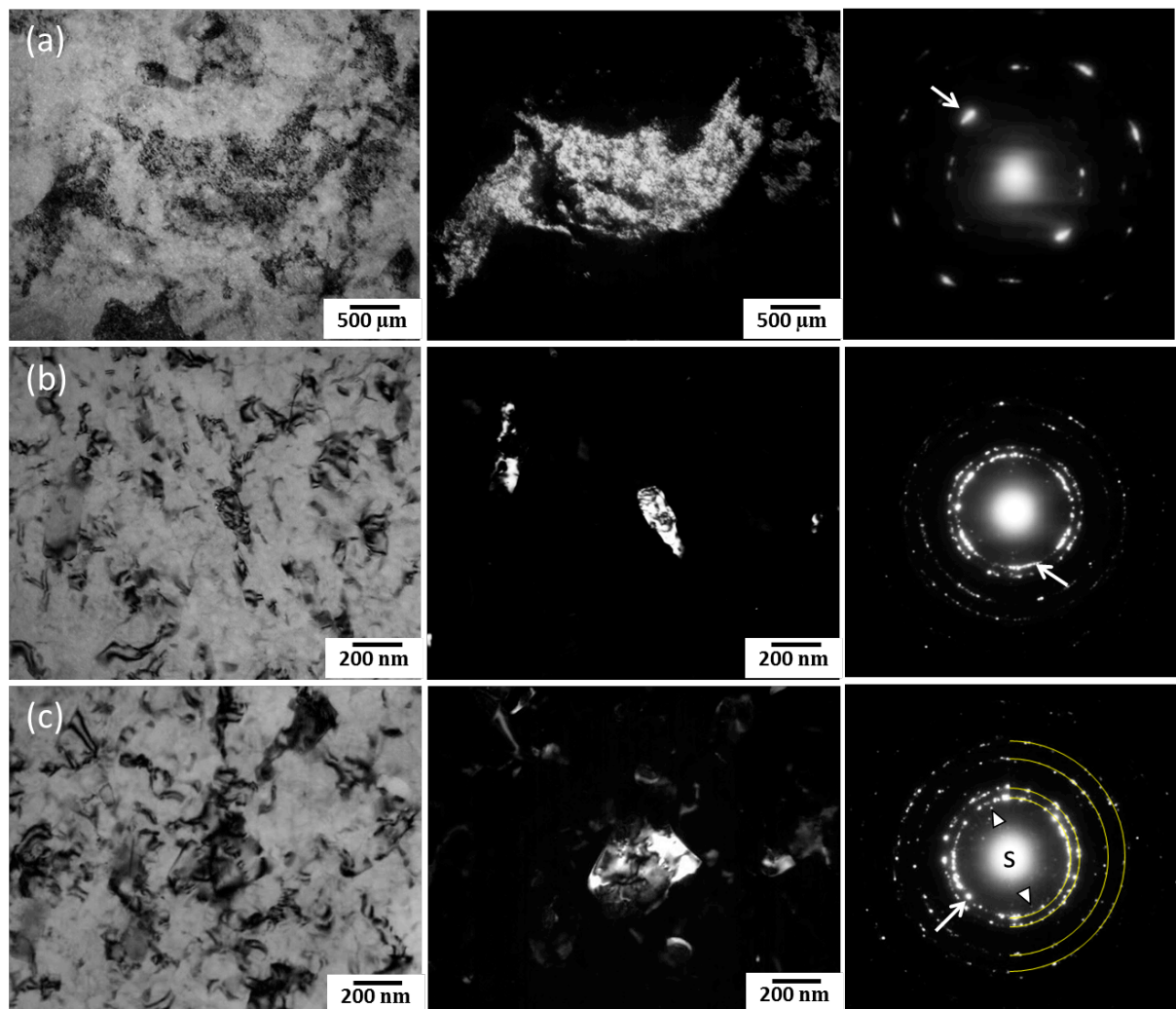


Fig. 3 TEM micrographs and SAED patterns of Al2024 after HPT at (a) initial stage ( $N = 0.75$ ), (b) saturated stage ( $N = 5$ ) and (c) saturated stage ( $N = 5$ ) and aged at 423 K for 4 h. Arrows in (a)-(c) indicate diffracted beams for dark-field images and s in (c) for S-phase.

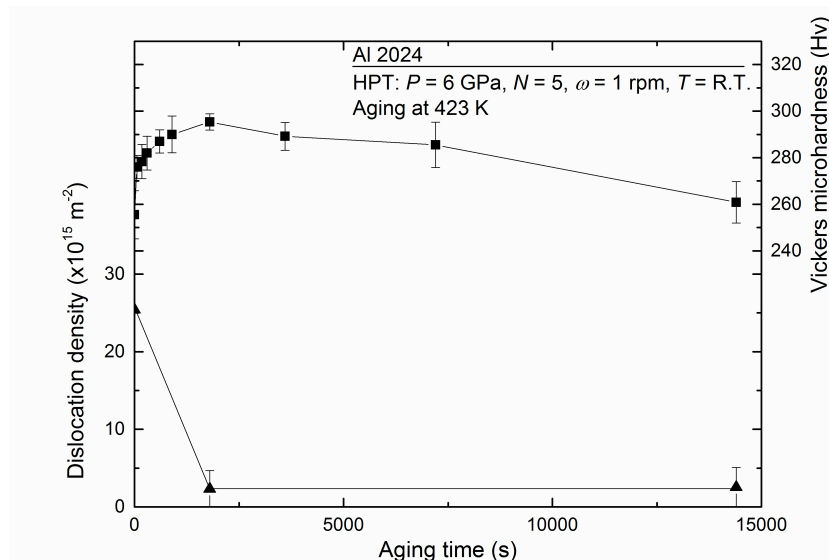


Fig. 4 Evolution of dislocation density with aging.

Fig. 4 shows the variation of dislocation density with the aging time along with the variation of the hardness. The dislocation density is high after HPT processing but it decreases with the aging time. The decrease in the dislocation density should be attributed to annihilation due to dislocation recovery through enhanced diffusion. The decrease in dislocation density may contribute to reduction in strength. Nevertheless, the aging behaviour exhibits the opposite trend until the peak age, and thus this indicates that the contribution from the precipitation hardening well exceeds the softening due to dislocation recovery. It should be noted that measurement of the grain size at the peak aging ( $\sim 260 \pm 80$  nm) and overaging ( $260 \pm 90$  nm) shows no sign of grain growth through the aging. It is worthwhile addressing here that the increase in the hardness during the initial stage of aging was neither due to the grain growth nor the dislocation recovery but it is due to the precipitation hardening.

#### 4. Conclusions

- (1) The grain size was refined to  $\sim 240 \pm 80$  nm and Vickers microhardness was significantly increased through application of HPT processing.
- (2) The hardness values after 0.75 and 5 turns fell well on a single curve when they are plotted against the equivalent strain.
- (3) The hardness increases with straining by HPT and saturates to a constant level at 260 HV after the HPT processing for 5 turns.

- (4) Simultaneous strengthening due to grain refinement and fine precipitation is achieved when the HPT-processed samples through 5 turns are aged at 423 K.

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