



# Age hardening and thermal stability of Al–Cu alloy processed by high-pressure torsion

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## ARTICLE INFO

### Article history:

Received 15 October 2014

Received in revised form

26 December 2014

Accepted 29 December 2014

Available online 6 January 2015

### Keywords:

Severe plastic deformation (SPD)

High-pressure torsion (HPT)

Ultrafine-grained materials (UFG)

Precipitation hardening

Aluminum alloys

## ABSTRACT

An age-hardenable Al–4 wt% Cu alloy is severely deformed using high-pressure torsion (HPT) to refine the microstructure to an average grain size of  $\sim 210$  nm. High saturation hardness of 205 Hv and high tensile strength of 820 MPa are achieved after the HPT processing. It is shown that the strength of the HPT-processed alloy is further improved by natural aging at room temperature or by artificial aging at 353 K. A peak hardness followed by softening appears within a few days after natural aging and within a few minutes after aging at 353 K, suggesting the low thermal stability of the alloy. Quantitative evaluation of different strengthening mechanisms shows that the grain boundary hardening through the Hall–Petch relationship and the precipitation hardening through the Orowan relationship are dominant strengthening mechanisms.

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## 1. Introduction

High-pressure torsion (HPT) is a well-known severe plastic deformation (SPD) process and it is capable of refining the grain size to the submicrometer or nanometer range and consequently of enhancing the strength and hardness in most metallic materials [1–6]. In HPT process, a thin disc sample is compressed between two anvils under a high pressure and shear strain is concurrently introduced by rotating the anvils with respect to each other [1]. Since the earliest work by Bridgman in 1935 [1], it has been shown that the hardness and strength of materials evolve into saturation levels with straining [1–17], where hardness, strength, grain size and dislocation density remain unchanged with further increase in the shear strain. It is of particular scientific and industrial interest to find some strategies to improve the hardness and strength of HPT-processed materials above the saturation levels.

For ultrafine-grained (UFG) materials processed by HPT, besides the strengthening by grain refinement through the Hall–Petch relation [18,19], the strength can be improved by some other factors such as dislocation accumulation through the Bailey–Hirsch relationship [21], solid solution hardening through the

Fleischer and Labusch relationships and precipitation hardening through the Orowan relationship [21,22]. It was shown that although the contribution of solid-solution hardening to the total strength of HPT-processed materials is less than 15%, the solute atoms can significantly improve the strength because of their contribution to extra grain refinement [12]. Moreover, solute atoms can usually improve the thermal stability of the UFG materials [5]. Earlier studies showed that solute atoms can produce extra strengthening in UFG materials by formation of precipitates [23–28], although precipitation may occur heterogeneously because of formation of precipitates on dislocations and grain boundaries during aging [29–31].

The purpose of this research is to study the evolution of microstructure, mechanical properties and thermal stability in a precipitation-hardenable Al–Cu alloy during HPT processing and after post-HPT aging. The contribution of different hardening mechanisms such as hardening by grain refinement, hardening by dislocation accumulation, solid solution hardening and precipitation hardening is investigated.

## 2. Experimental materials and procedures

The material used in this study was an Al–4 wt% Cu (1.7 atom% Cu) Alloy. The material was received in a form of an ingot with dimensions of  $1.5 \times 10 \times 25$  cm<sup>3</sup>. The ingot was homogenized at 793 K for 24 h in an air atmosphere and slowly cooled down in the

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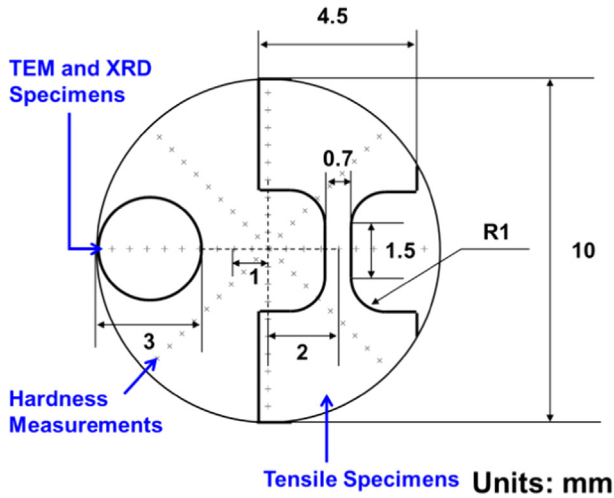


Fig. 1. Schematic illustration of HPT disc and locations for hardness measurements, tensile specimen and TEM and XRD discs.

furnace. Discs of 1 mm thickness and 10 mm diameter were cut from the homogenized ingot using a wire-cutting electric discharge machine (EDM). The discs were solution-treated at 823 K for 5 h in an argon atmosphere and immediately quenched into ice water.

Each disc was processed by HPT at room temperature (R.T.) under an applied pressure of  $P=6$  GPa for  $N=0.5, 1$  and 5 turns at a rotation speed of  $\omega=1$  rpm. The thicknesses of discs were reduced from 1 mm to  $\sim 0.8$  mm during the HPT processing. The HPT-processed discs were stored at room temperature for up to 18 days to investigate their natural aging and thermal stability. Several discs were aged at 353 K in the air atmosphere for certain periods of time up to 5 days.

For each disc sample, Vickers microhardness was measured at 8 different radial directions, as illustrated in Fig. 1, by application of a load of 100 g for a dwell time of 15 s.

For phase analyses, 3 mm discs were prepared from the edge of disc samples, as shown in Fig. 1. X-ray diffraction (XRD) analyses were performed on 3 mm discs using the Cu  $K\alpha$  radiation at an accelerating voltage of 40 kV and a current of 36 mA in a scanning step of  $0.02^\circ$  and a scanning speed of  $0.5^\circ/\text{min}$ .

Tensile specimens having 1.5 mm gauge length, 0.7 mm width and 0.5 mm thickness were cut from the disc samples using EDM, as illustrated in Fig. 1. Tensile tests were carried out at room temperature with an initial strain rate of  $2 \times 10^{-3} \text{ s}^{-1}$ .

For microstructural observations, the disc samples were punched to small discs with 3 mm diameters, as shown in Fig. 1, and further ground to a thickness of 0.12 mm. The 3 mm discs were thinned using a twin-jet electrochemical polishing system using a solution of 20%  $\text{HNO}_3$  and 80%  $\text{CH}_3\text{OH}$  at a temperature of 260 K with an applied voltage of 12 V. The microstructural examinations were performed using transmission electron microscopy (TEM) at an accelerating voltage of 200 kV. Selected-area electron diffraction (SAED) patterns were also taken from regions with  $6.3 \mu\text{m}$  diameter for each microstructural examination.

### 3. Results

#### 3.1. Mechanical properties after HPT

The variations of Vickers microhardness are plotted in Fig. 2 (a) against the distance from the center of discs after processing for  $N=0.5, 1$  and 5 turns. The lower dotted lines show the hardness levels for the homogenized and solution-treated samples.

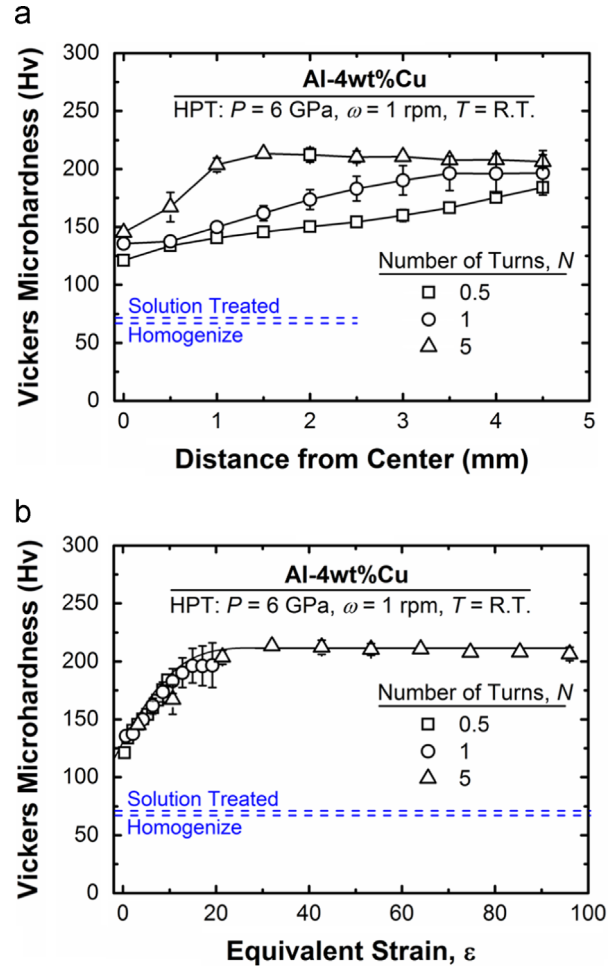


Fig. 2. Vickers microhardness plotted against (a) distance from disc center and (b) equivalent strain for samples processed by HPT after various turns.

Microhardness increases with increasing the distance from the disc center as well as with increasing the numbers of turns, but the hardness tends to saturate to a steady state level of 210 Hv at  $\sim 1$  mm from the disc center after 5 turns. All hardness levels after the HPT processing are drastically higher than those after the solution treatment.

All microhardness values in Fig. 2(a) are plotted against the von Mises equivalent strain in Fig. 2(b). The von Mises equivalent strain,  $\epsilon$ , was calculated by the following Eqs. (2) and (3):

$$\epsilon = \frac{2\pi r N}{\sqrt{3}t} \quad (1)$$

where  $r$  is the distance from the disc center,  $N$  is the number of turns and  $t$  is the thickness of disc. It is apparent that all the points laid essentially on a single curve regardless of the number of turns and distance from the disc center. It is noticeable that the saturation occurs at a well-defined equivalent strain beyond  $\sim 30$  where the hardness remains constant with further straining. The hardness-strain behavior and the occurrence of steady state for the Al-Cu alloy is consistent with those reported earlier in numerous metallic materials [2–12] including low-purity Al (99%) [14], but is different from the hardness behavior of high-purity Al (99.99%, 99.999%) which exhibits a hardness peak followed by softening at large strains [14–17].

Fig. 3 shows the stress-strain curves after tensile testing conducted at room temperature with an initial strain rate of  $2 \times 10^{-3} \text{ s}^{-1}$  for the samples processed by HPT for various turns including the sample after solution treatment. The tensile strength

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