Strengthening of AA7075 alloy by processing with high-pressure sliding (HPS) and subsequent aging

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Abstract

High-pressure sliding (HPS) is a new severe plastic deformation process for sheet samples under high-pressure. In this study, the HPS process is applied to an age-hardenable 7075 aluminum alloy for grain refinement. HPS was conducted under a pressure of 1.0 GPa for sliding distances of 7.0 mm at room temperature and for 10 mm at 373 K and 423 K, and thus the average grain size was reduced to ~280 nm, ~ 240 nm and ~ 250 nm, respectively. Ultimate tensile strength of ~600 MPa was achieved by this grain refinement. Aging at 373 K led to further hardening to 745 MPa of ultimate tensile strength with 4% of elongation to fracture through a fine dispersion of precipitates. It is shown that simultaneous strengthening due to grain refinement and fine precipitation is achieved in the AA7075 by application of HPS and subsequent aging.

Keywords: *high-pressure sliding; severe plastic deformation; grain refinement; age-hardening; dispersed precipitates*

1. Introduction

Ultrafine-grained (UFG) materials produced by severe plastic deformation (SPD) often exhibit superior mechanical properties because of the small grains [1-3] with a large density of grain boundaries that hinder the motion of dislocations. Then the yield strength increases with decreasing in the grain size as fitted by Hall-Petch law [4, 5]. Bulk UFG materials are defined as materials having homogeneous and equiaxed microstructures with an average grains size less than 1.0 μm and with a majority of boundaries having high angles of misorientations [6]. During the last 3 decades, several SPD methods were developed together with their performances, which mainly include high-pressure torsion (HPT) [7-9], equal-channel angular pressing (ECAP) [10], accumulative roll-bonding (ARB) [11], multi-directional forging (MDF) [12], cyclic extrusion and compression (CEC) [13] and repetitive corrugation and strengthening (RCS) [14]. Also researches have been active in developing new SPD methods or modifying existing SPD methods to enhance the performance, size or shape, for example ECAP with parallel channels (ECAP-PC) [15], ECAP-conform [16] and continuous-HPT [17, 18], high-pressure sliding (HPS) [19] and some others [20-22]. For the HPS processing, it is possible to apply for ribbon-shape or sheet-shape specimens under a high pressure as illustrated schematically in Fig. 1. Until now, several materials such as pure Al [19], an Al-Mg alloy [23] and an

AZ61 [23] alloy have been processed successfully using the HPS to obtain bulk samples of UFG structures.

Fig. 1 Schematic illustrations of high-pressure sliding (HPS). (a) Assembly of two anvils with guiding pins, one plunger and two thin sheet samples. (b) Cross sectional view of assembly.

There are some attempts that combine two different strengthening methods, especially grain refinement and precipitation hardening to produce alloys with superior mechanical properties using ECAP[2, 24, 25], cryo-milling [26, 27] and HPT [28, 29]. However, it is difficult to expect the combined effects of both strengthening mechanisms using traditional thermo-mechanical treatments because of the difficulty in achieving fine precipitation in UFG structures by aging. In addition, it is important to disperse fine precipitates within the fine grains by subsequent aging while keeping the small grain size. This paper

investigates whether it is possible to achieve the simultaneous strengthening of age-hardenable high strength AA7075 by grain refinement and dispersion of small precipitates using the HPS process and subsequent aging treatment.

2. Experimental procedures

The present investigation was carried out on an AA7075 plate with the chemical composition shown in **Table 1**.

Table 1 Chemical composition of AA7075 aluminum alloy (mass%).

Zn	Mg	Cu	Fe	Cr	Si	Mn	Ti	Al
5.63	2.56	1.68	0.21	0.19	0.14	0.05	0.02	Bal.

Samples for HPS processing were cut to sheets with dimensions of $3.0 \times 100.0 \times 0.8 \text{ mm}^3$ using electro-discharge machine (EDM) from the plate. Then, the sheet samples were subjected to solution treatment at a temperature of 773 K for 5 h. They were quenched quickly into ice water. Cold rolling was conducted after the solution treatment at room temperature using the plate with the initial thickness of 10 mm with a thickness reduction of 44% for comparison with the HPS-processed samples. HPS was conducted at room temperature, 373 K and 423 K under an applied pressure of 1.0 GPa. The plunger was pressed with sliding distances of 7 mm and 10 mm at a sliding speed of 2.5 mm/s. For processing at 373 K and 423 K, a heating unit controlled by thermo-couples was set on the

anvil. After the processing, the anvils and samples were cooled to room temperature with use of a fan for 3 min. Equivalent strain during HPS processing was estimated by the following equation (1) [19].

$$\varepsilon_{eq} = x/\sqrt{3} h \quad (1)$$

where, ε_{eq} is the equivalent strain, x is the sliding distance of plunger with respect to anvil and h is the height (thickness) of sample.

After the HPS processing, samples were subjected to aging treatment at the temperature of 373 K. Vickers microhardness measurement and tensile test were conducted to examine their mechanical properties. For the microhardness measurement, the samples were polished mechanically to a mirror-like surface using abrasive sandpapers and then alumina powders. In each hardness measurement, a load of 200 g was applied for a duration time of 15 s. An average was taken from 15 measurements at positions of $L = 20, 35, 50, 65$ and 80 mm from the front edge of the sheet sample. For tensile test, the HPS-processed samples were polished mechanically to a thickness of 0.6 mm, and tensile specimens were cut using EDM with the dimensions of 2.1 mm gauge length and 0.7 mm width. Tensile tests were conducted with an initial strain rate of $1.4 \times 10^{-3} \text{ s}^{-1}$ at room temperature. Transmission electron microscopy (TEM) was conducted to observe microstructures and to analyze crystal structure. TEM

samples were taken from the vertical direction of $L = 45 \sim 55$ mm area. Disks with 3 mm in diameter for TEM observation were punched out from the sheet shape sample. Disks were ground to a thickness of 0.15 mm and thinned using a twin-jet electro-polishing facility in a solution of 70 vol.% CH₃OH and 30 vol.% HNO₃ at 263 K.

3. Results and Discussion

3.1 Importance of HPS process

Figure 2 shows the appearance of samples before and after processing by HPS including those for cold rolling. Color lines are marked on both surfaces of the sample in Fig. 2(a) before the HPS processing. The shifts of the markers indicate introduction of shear strain by the HPS processing. Figures 2(b), (d) and (f) show the surfaces on the anvil side and Fig. 2(c), (e) and (g) the surfaces on the plunger side after the HPS processing. It is clear that the markers are shifted by the distances corresponding to the sliding distances, indicating that no slippage occurred between the sample and plunger and the sample and anvil. It also suggests that the shear strain is introduced homogeneously throughout the samples during the HPS processing. It is important to note that samples were deformed without cracking even at room temperature by the HPS processing. However, cracks are visible on the sample after cold rolling with 46% thickness reduction as shown in Fig 2.(h) despite the smaller strain introduced by the cold

rolling. Estimation of the equivalent strain through eq.(1) results in ~ 5.8 and ~ 8.2 for the sliding distances of $x = 7.0$ mm and 10 mm, respectively, in the HPS processing, whereas ~ 0.7 for the 46% thickness reduction in the cold rolling. It is important to emphasize that shear deformation under high pressure suppress the crack initiation.

Fig. 2 Appearance of samples (a) before HPS, (b)-(d) after HPS process under the applied pressure of 1.0 GPa with $v = 2.5$ mm/s for (b) $x = 7.0$ mm at R.T., (c) $x = 10$ mm at 373 K, (d) $x = 10$ mm at 423 K and (e) before cold rolling and after 46% cold rolling.

3.2 Improvement in tensile properties

The Vickers microhardness is plotted in [Fig. 3](#) against the distance from the edge as defined in inset. The hardness is increased more than twice when compared to the hardness level after solution treatment. This hardness increase should be due to work hardening and, as shown later, due to grain refinement arising from intense shear strain introduced by the HPS processing. The hardness level is invariably higher for processing at 373 K than at R.T. and this can be because the sliding distance is larger as 10 mm for the processing at 373 K. However, the hardness level is slightly lower for the processing at 423 K than after processing at R.T. This can be because a certain extent of recovery occurs during and/or after the HPS processing. The hardness values at edge parts are slightly lower than those at the center parts. It is considered that the shear strain was lower due to less introduced due to less constriction at the edge parts.

Fig. 3 Vickers microhardness of HPS-processed AA7075 under $P = 1.0$ GPa with $v = 2.5$ mm/s for $x = 7.0$ mm at R.T. and $x = 10$ mm at 373 K and 423 K.

Hardness measurements were also carried out on the cross sectional planes as shown in Fig 4. Inhomogeneous hardness variation exists at both samples processed at R.T. and 373K, and this suggests that microstructural development may be inhomogeneous across the cross sections. However, the results regarding the hardness level are consistent with microhardness measurements (Fig. 3): the hardness level is invariably higher at 373 K than at R.T.

Fig. 4 (a) Schematic illustration of HPS-processed sheet with locations for hardness measurement. (b) and (c) are variations of Vickers microhardness on cross sections perpendicular to sliding direction after HPS processing under $P = 1.0$ GPa with $v = 2.5$ mm/s for $x = 7.0$ mm at R.T. and $x = 10$ mm at 373 K, respectively.

Tensile tests were conducted at 5 different positions ($L = 20, 35, 50, 65, 80$ mm) and the results are summarized in [Table 2](#) including those after aging of the HPS-processed samples.

Figure 5 shows representative results of tensile testing for the samples processed at R.T., 373 K and 423 K including the sample after solution treatment. Tensile strength increases with appreciable ductility by the HPS processing: 0.2% yield strength (YS), ultimate tensile stress (UTS) and elongation (EL) to failure are 550 MPa, 650 MPa and 7.5% at R.T., 600 MPa, 620 MPa, 7.0% at 373 K, and 550 MPa, 580 MPa, 10.0% at 423 K. The HPS process is effective to increase YS and UTS while it maintains a reasonable ductility. These data are almost comparable to those of conventional AA7075 after T6 treatment, 505 MPa, 570 MPa, 11.0%, respectively [30]. The cold-rolled material processed in this study shows 420 MPa, 510 MPa, 7.0% after 44% thickness reduction, respectively. These values are acceptable because some published papers reported tensile properties of cold-rolled 7075 with various cold rolling conditions, values are reported as 270 ~ 500 MPa, 320 ~ 570 MPa and 5 ~ 9%, respectively. [31-33].

Fig. 5 Stress-strain curves of solution-treated sample, cold-rolled sample and HPS-processed samples under $P = 1.0$ GPa with $v = 2.5$ mm/s for $x = 7.0$ mm at $T = \text{R.T.}$, $x = 10$ mm at 373 K and $x = 10$ mm at 423 K.

Table 2 Tensile properties after HPS processing at different temperatures and subsequent aging.

	Aging time, h	σ_{YS} , MPa	σ_{UTS} , MPa	EL, %
HPS at R.T.	0	525 ± 28	592 ± 23	7.5 ± 2.2
	5	560 ± 48	640 ± 72	9 ± 2.1
	10	604 ± 31	722 ± 20	5.6 ± 2.7
	50	578 ± 13	656 ± 34	2.7 ± 1.9
HPS at 373 K	0	560 ± 29	612 ± 25	6.8 ± 1.0
	1	585 ± 17	668 ± 13	5.3 ± 1.6
	5	576 ± 39	672 ± 30	8.9 ± 2.1
	20	526 ± 20	637 ± 7.5	8 ± 0.8
HPS at 423 K	0	532 ± 20	566 ± 11	8.4 ± 2.0

3.3 Aging behavior after HPS processing

3.3.1 Aging temperature

Normally, diffusivity can be explained with both temperature and activation energy by Arrhenius Equation. In the case of ultra-fine grained materials, it has been estimated and reported for its fast diffusion because of their lower activation energy by generated grain boundaries and large amount of defects after SPD processing [34-36]. Especially, the diffusion coefficients in an Al alloy processed by severe plastic deformation are 10^{12} - 10^{22} times higher than the lattice diffusion coefficients and are comparable to the surface diffusion coefficients or grain boundary diffusion coefficients [36]. Also, some reports are available about very fast precipitation with coarse particles on grain boundaries in ultrafine-grained Al alloys when the aging temperature is high [28, 29]. It is necessary to set a lower aging temperature than the conventional T6 temperature in 7075 alloy to control precipitation and to avoid grain growth.

3.3.2 Hardness variation with aging

Figure 6 shows hardness variations with aging at a temperature of 373 K for samples processed by HPS at R.T. and 373 K including a sample after solution treatment. Figure 6(b) is an enlargement of vertical axis for the HPS-processed

samples in [Fig. 6\(a\)](#). It is apparent that the HPS-processed samples are further strengthened by the aging treatment. Whereas it takes more than 50 h to reach a peak level for the solution-treated sample, the aging is well accelerated so that the peak level is reached faster: 5 and 10 h for the HPS-processed samples at R.T. and 373 K, respectively. It should be noted that the amount of hardness increase by the aging is less for the HPS-processed samples than for the solution-treated sample: 26 H_v and 18 H_v , respectively, for the former while more than 80 H_v for the latter. It is considered that a high density of lattice defects such as dislocations and grain boundaries generated by the HPS processing accelerated atomic diffusion and thus enhanced precipitation kinetics [\[2\]](#). It should be noted that heating and cooling time (which take for ~5 min) before and after HPS processing is negligible on the precipitation because the time is too short to influence when compared to the time for peak aging.

Fig. 6 (a) Aging behavior at 373 K of solution-treated and HPS-processed AA7075 under $P = 1.0$ GPa with $\nu = 2.5$ mm/s for $x = 7.0$ mm at R.T. and $x = 10$ mm at 373 K and (b) enlargement of vertical (H_v) scale for HPS-processed samples.

3.3.3 Tensile properties after aging treatment

Stress-strain (S-S) curves of the samples subjected to under-aging, peak-aging and over-aging at 373 K are shown in Fig.7. The results are well consistent with

the hardness variations shown in Fig. 6. YS, UTS and EL further increase with aging until the peak levels: YS, UTS and EL at peak aging are 640 MPa, 745 MPa and 4% for the sample deformed by HPS at R.T. and 550 MPa, 690 MPa and 10% for the sample deformed by HPS at 373 K. It is well demonstrated that the combination of both HPS processing and subsequent aging is promising for extra strengthening of the AA7075 alloy.

Fig. 7 Stress-strain curves of AA7075 after aging at 373K for samples subjected to HPS processing under $P = 1.0$ GPa with $v = 2.5$ mm/s for $x = 7.0$ mm at R.T. and $x = 10$ mm at 373 K.

3.4 Microstructure analyses

3.4.1 After HPS process

Figure 8 shows Dark-field (DF) images and selected area electron diffraction (SAED) patterns after HPS processing **(a)** at R.T., **(b)** at 373 K and **(c)** at 423 K: DF images on the left and SAED patterns on the right. Inspection reveals that the SAED patterns exhibit ring-type forms, indicating that the microstructures consist of small grains with high-angle grain boundaries, where the grain size is well in the submicrometer levels as ~280 nm, ~ 240 nm and ~ 250 nm for the samples processed by HPS at R.T., 373 K and 423 K, respectively. Grain size distributions are shown in **Fig. 9**. Here, the grain size was measured on the DF images where the two orthogonal axes of bright areas were measured and averaged from a total of more than 100 grains. In the DF images shown in **Fig. 8 (b)** and **(c)**, many dots with white contrasts are visible. They are confirmed as intermediate- η' and equilibrium- η (MgZn_2) precipitates from the SAED patterns where MgZn_2 peak positions are depicted with rings in red. In **Fig. 8(c)**, precipitates are formed clearly with an Ashby-Brown (coffee bean) contrast in the areas marked with dotted lines and they are enlarged in **Fig. 8(d)** and **(e)**. In addition to these coherent particles, incoherent particles are also observed as indicated by yellow arrows in **Fig. 8(c)**. These TEM observations thus provide evidence that precipitation occurred during HPS processing at 373 K and 423 K.

Fig. 8 Dark-field images including SAED patterns of HPS-processed AA7075 under $P = 1.0$ GPa with $v = 2.5$ mm/s for (a) $x = 7.0$ mm at R.T., (b) $x = 10$ mm at 373 K and (c) $x = 10$ mm at 423 K. (d) and (e) for enlarged areas marked by dotted lines in (c).

Fig. 9 Grain size distributions of samples (a) after HPS processing (as-HPS) at R.T., 373 K and 423 K, (b) after aging at 373 K following HPS processing at R.T., and (c) after aging at 373 K following HPS processing at 423 K.

3.4.2 Microstructure evolution during aging treatment

Figure 10 shows TEM micrographs including SAED patterns of the samples aged at 373 K (a) for 5 h and (b) for 10 h after HPS processing at 373 K: BF images on the left and SAED patterns on the right. An ultrafine-grained microstructure with a grain size of ~250 nm is still retained after 5 and 10 h of the aging. In the Bright-field (BF) images, small dots with dark contrasts are visible in both images. Inspection of the SAED patterns reveals that these particles should be η' precipitates. It is considered that such small precipitates are responsible for extra strengthening of the AA7075 alloy above the HPS-processed state as shown in Fig.6 and Fig.7. These results are very similar to other ultra-fine grained 7xxx series alloys that processed by different SPD methods [3, 37].

Fig. 10 Bright-field images including SAED patterns of HPS-processed AA7075 under $P = 1.0$ GPa with $v = 2.5$ mm/s for $x = 10$ mm at 373 K followed by aging at 373 K for (a) $t = 5$ h and (b) $t = 10$ h.

4. Conclusions

An AA7075 alloy was subjected to aging after HPS processing. It was possible to achieve simultaneous strengthening due to grain refinement and fine dispersion of precipitates.

(1) Grain refinement was achieved to average grain sizes of ~280 nm, ~ 240 nm and ~ 250 nm in the samples processed by HPS at R.T., 373 K and 423 K, respectively.

(2) The values of 0.2% yield strength (YS), ultimate tensile stress (UTS) and elongation (EL) to failure were measured as 550 MPa, 650 MPa 7.5% and 600 MPa, 620 MPa, 7% after HPS processing at R.T. and 373 K, respectively. The values of YS and UTS were further increased by aging treatment to 640 MPa and 745 MPa for the sample processed by HPS at R.T. and to 550 MPa and 690 MPa for the sample processed by HPS at 373 K, while the values of EL were well retained as 4% and 10%, respectively.

(3) It was possible to achieve simultaneous strengthening due to grain refinement and fine dispersion of precipitates by HPS processing and by subsequent aging.

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