Journal of Alloys and Compounds 502 (2010) 59–62

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jallcom

Dynamic strain aging of a commercial Al–Mg–Si–Cu alloy during equal channel angular extrusion process

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ARTICLE INFO

Article history:
Received 11 August 2008
Received in revised form 12 April 2010
Accepted 23 April 2010
Available online 16 May 2010

Keywords:
Al–Mg–Si–Cu alloy
Dynamic strain aging
Mechanical properties
Equal channel angular extrusion

ABSTRACT

In this research, the occurrence of dynamic strain aging (DSA) phenomenon during equal channel angular extrusion (ECAE) of a commercial air cooled Al–Mg–Si–Cu alloy is investigated. In order to detect the negative strain rate sensitivity behaviour associated with DSA, samples were ECAE-ed at various temperatures and strain rates. Subsequently, the extruded billets were subjected to mechanical and physical tests and it was found that at a certain ECAE temperature range there is a critical strain rate above which DSA occurs. The implication of the results to strengthening of dilute solid solutions is discussed.

1. Introduction

DSA phenomenon is one of the most feasible strengthening methods applicable to most solid solutions. The theoretical models of the phenomenon have presented an explanation in terms of the interaction between moving dislocations and diffusing solute atoms. Cottrell theory [1] is the first detailed theoretical model proposed for DSA. In this model, DSA starts when the diffusion rate of solute atoms becomes identical to the velocity of the mobile dislocations. Later, McCormick [2] showed that for substitutional alloys the predictions of Cottrell theory are significantly different from the experiment. Therefore, developing the idea of Sleeswyk [3], he proposed a more suitable hypothesis that during deformation, a mobile dislocation spends most of its time to surpass obstacles and its interaction with diffusing solute atoms, in the same way that Cottrell proposed, mainly occurs during the time that the dislocation is waiting in front of the obstacles [4]. Finally, Van den Beukel [5] improved the McCormick model by changing the onset of DSA criteria from Portevin–Le Chatelier effect to negative strain rate sensitivity.

In the case of a substitutional solid solution, which is the case in present research, the vacancies necessary for diffusion are assumed to be produced by the plastic deformation [4]. Equal channel angular extrusion (ECAE) is one of the most promising deformation processes introduced recently [6]. Using ECAE a relatively high deformation can be imposed on samples. Recently, a few researches have been carried out on both the post effects of ECAE on aging phenomena [7] and the dynamic precipitation of super saturated solid solutions during ECAE [8,9]. In 2005, Cabibbo et al. [10], concisely, discussed the DSA phenomena in ECAE at room temperature but yet, at least to the knowledge of the authors of this paper, no detailed work exists on the DSA phenomena during ECAE and above ambient temperature. Thus, the motive for this research was to inspect the occurrence of DSA in hot ECAE.

2. Materials and experimental procedures

Raw round samples having a diameter of 14 mm and chemical composition shown in Table 1 were cooled in air after solution treating at 560 °C for 2.5 h, allowing a minimal solid solution level in the matrix. Hot ECAE tests were conducted isothermally at different strain rates and temperatures using an Amsler pressing machine, ECAE die with $\varphi = 90^\circ$ and $\psi = 20^\circ$, and a customized electrical furnace capable of controlling the ECAE die temperature with precision of ±5 °C. Vickers harness tests and electrical resistivity experiments were carried out on sample cross sections using an Instron Wolpert-PIA Tester 722 and Sigmatest D 2.068 machines respectively. Furthermore, using conventional formulas [11] and a 3 mm diameter shear punch apparatus, the yield strength of the extruded alloy were evaluated. Thin foils for TEM observations were prepared by grinding the slices to a thickness of about 100 μm and twin-jet electropolishing was done using a solution of 30% nitric acid–70% methanol. TEM observations were carried out using a JEOL JEM-2100, operated at 200 kV.

3. Results and discussion

The average hardness of the billets ECAE-ed at different processing conditions is plotted in Fig. 1. For the samples extruded at room temperature, the hardness increases constantly as the extru-

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doi:10.1016/j.jallcom.2010.04.125

Table 1
Chemical composition of the alloy used in this research.

Element	Mg	Si	Cu	Fe	Mn	Al
wt%	0.9	1.4	0.9	0.6	0.1	95.8

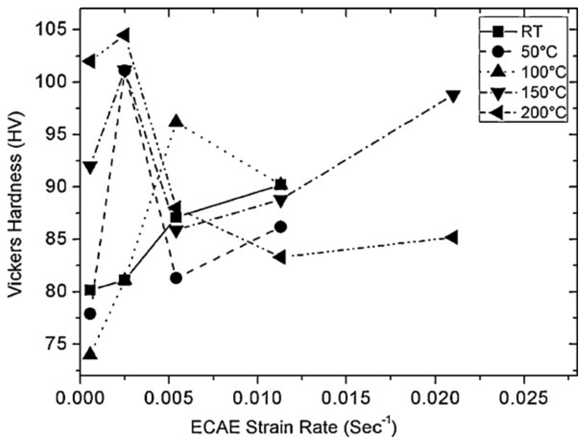

Fig. 1. Changes in samples mean hardness due to the change in ECAE strain rate for various ECAE temperatures.

sion strain rate increases. Such behaviour is realized as the classic behaviour of metals and indicates that no microstructure related phenomenon has taken place in extrusion at room temperature. Conversely, a peak hardness condition is observed at temperatures above the room temperature. As discussed extensively in the literature [5,12,13], such negative strain rate sensitivity behaviour can be attributed to DSA. Since one may argue that the upsurge in hardness might be due to static strain aging before and after deformation, especially at low strain rates which involve longer processing times, static strain aging tests were carried out on raw samples at temperatures and times corresponding to actual ECAE conditions. Fig. 2 shows the variation of hardness versus aging time at different aging temperatures. From Fig. 2 it can be seen that virtually no change in hardness value is observed meaning that no precipitation is occurred.

The presence of a peak hardness condition is due to the occurrence of two competing phenomena, DSA and recovery. Therefore, the mean hardness value of extrusion products is strongly dependent on the interaction between these two factors. As a result, if we assume that the extend of occurrence of recovery is infinitesimal at RT [14,15], from Fig. 1 as the mean hardness values of samples extruded at RT and 50 °C in the lowest strain rate (highest processing time) are identical two conclusions can be drawn: (1) The recovery mechanism contribution to the change in hardness is negligible at 50 °C; (2) DSA has not taken place. However, the presence of a sharp peak hardness value at 50 °C might be due to occurrence of DSA in the absence of recovery. The reason for the drop in sample mean hardness value at strain rates higher than $25 \times 10^{-4}\,s^{-1}$, according to the classic dislocation theory, is the direct proportionality of dislocation speed and applied strain rate. Moreover, the speed with which solute atoms responsible for DSA diffuse is proportional to the processing temperature and therefore constant at an arbitrary temperature. Hence, as the processing strain rate increases at a constant temperature, the probability that solute atoms diffuse and limit the movement of dislocation is decreased and therefore dislocation locking becomes less effective.

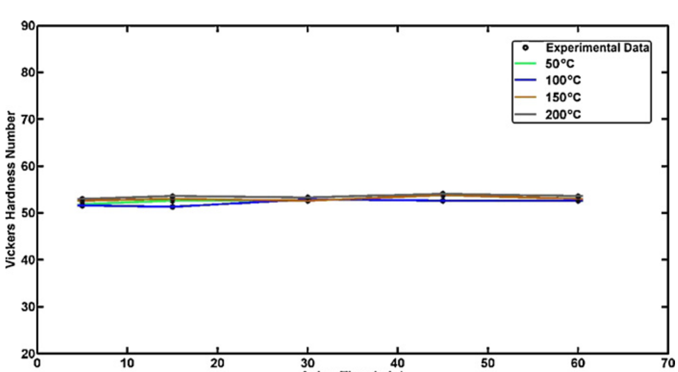

Fig. 2. Variation of hardness against aging time.

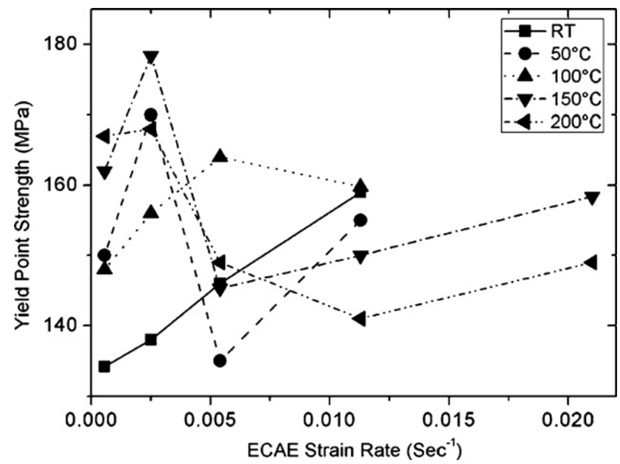

Fig. 3. Changes in samples yield point strength due to the change in ECAE strain rate for various ECAE temperatures.

It is obvious from Fig. 1 that the peak hardness condition tends to higher strain rates as the processing temperature increases to 100 °C. This is due to the fact that by increasing the processing temperature, the diffusion coefficient of the atoms responsible for DSA (i.e. magnesium and silicon) increases and therefore these atoms can lock the dislocations moving with higher travelling speeds. Consequently, the peak strength is appeared at a higher strain rate. Moreover, comparison of sample mean hardness values at the lowest strain rate at RT and 100 °C indicates that the recovery phenomenon is more actively involved at 100 °C. Hence, unlike the sharp variation of hardness observed at 50 °C, the simultaneous occurrence of recovery and DSA at 100 °C results in a more gradual change in hardness.

At processing temperatures above 100 °C, the strain rate corresponding to the maximum hardness condition shifts back to lower values. At these temperatures the simultaneous occurrence of recovery and DSA is dominant (notice the high hardness values at lowest strain rate), but there exists a critical strain rate at which the softening effect of recovery overcomes the hardening effect of DSA. The most logical hypothesis that can be offered is that, above these critical strain rates the dislocations kinetic energy is high enough to cut the precipitates in smaller unstable sizes that will most probably dissolve in the matrix. Therefore, according to McCormick's DSA theory [2] the limiting barriers that can enhance the occurrence of DSA in substitutional alloys no longer exist and as a result the DSA strengthening effects are diminished.

Fig. 3 depicts the yield stress obtained from shear punch tests carried out on extruded samples. The solid line shows the change in yield stress with ECAE strain rate at room temperature. As reported by previous investigators [6], the yield stress increases constantly as the speed of ECAE (or alternatively ECAE strain rate) increases. But for samples extruded at higher temperatures, there exist a peak yield stress at a specific strain rate due to commencement of the DSA in the ECAE die. As extrusion temperature increases from 50 °C

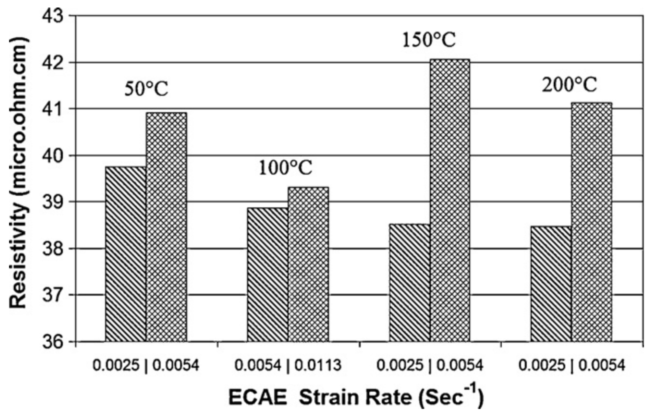

Fig. 4. Electrical resistivity values for peak and low strength samples at different ECAE temperatures.

to 100 °C the ECAE strain rate corresponding to the peak strength increases from 0.0025 s^{-1} to 0.0054 s^{-1}, confirming the hardness results shown in Fig. 1. The smaller yield strength of the extruded samples at 100 °C compared to that of 50 °C extruded samples, indicate the occurrence of recovery. In ECAE tests carried out at 150 °C and 200 °C, the strength drops dramatically at medium to high strain rates. Such large strength drop can be attributed to the occurrence of two phenomena; dislocation annihilation (or recovery) and dissolution of fine precipitates that enhance the commencement of DSA [16]. Higher extrusion temperature of 200 °C enhances both the recovery and dissolution of fine precipitates processes even more and results in a lower peak strength value comparing to the peak strength value at 150 °C. Considering both the hardness (Fig. 1) and yield strength (Fig. 3) values for different processing conditions, one can conclude that the best strengthening cycle for the material used in this research is hot ECAE at 150 °C using strain rate of 0.0025 s^{-1}.

The average electrical resistivities of selected samples are plotted in Fig. 4. At each processing temperature the left-hand side bar represents the electrical resistivity of the sample exhibiting the peak strength. From this figure, it is obvious that at all processing temperatures the peak strength sample electrical resistivity has minimal value. In fact, depletion of matrix from in-solution alloying elements, due to commencement of DSA phenomenon, has resulted in a lower electrical resistivity. For samples extruded at 50 °C and 100 °C, by increasing the ECAE strain rate a relatively small increase in the resistivity is observed. This is because increasing the strain rate has increased the dislocation speed while the diffusion rate of alloying elements remains the same and therefore DSA has not taken place optimally. The incomplete occurrence of DSA is identical to lower depletion of matrix from in-solution alloying elements. Note that such elements have contributions to electrical resistivity. From Fig. 4, it is evident that at 150 °C and 200 °C, increasing the strain rate from peak strength condition dramatically increases the average resistivity of the samples. This experimental result can prove the hypothesis offered in this research that above a certain processing temperature increasing the ECAE strain rate will result in dissolution of fine precipitates and therefore restrict the occurrence of DSA according to McCormick's aging theory [2].

BF and DF TEM images in Fig. 5 show a random size and distribution of precipitates and high density of dislocations. Such material which is reinforced by a good distribution of precipitates can significantly improve the mechanical properties. Precipitates, in fact, inhibit the movement of mobile dislocations and grain growth by pinning the dislocations. The increase in strength of the alloy resulting from dynamic strain aging is due to the refined precipitates and grain/subgrain site in addition to the high density of dislocations and point defects. Increasing the strain rate increased the fraction of defects and provided a high density of heterogeneous nucleation sites for precipitation during DSA. These metastable precipitates are nucleated and grown up quickly to form very fine and uniformly dispersed precipitates. Metastable phases are believed to be effectively resistant as obstacles to the movement of dislocations and thus new mobile dislocations should generate and results in higher stable dislocation density [17]. Moreover, from two main competitive phenomena, dynamic recovery and DSA, dynamic recovery is relatively suppressed by the solute and/or precipitate content in matrix and the aging responses in the alloy is accelerated by using ECAE contributing to a higher strengthening effect. Furthermore, the hydrostatic pressure developed during ECAE enhances the density of mobile dislocations [18]. In addition, in the case of ECAE there is a noteworthy plastic deformation arising from the shape of deformation zone that produces a high dislocation density. However, some precipitates are remarkably dissolved at higher forming temperatures. In these conditions there is a less effective resistance against the movement of dislocation.

At the temperatures higher than dynamic aging regime because diffusion-controlled mechanisms are activated during warm processing, dislocation density reduces, more solute atoms go out of the solid solution state, and some precipitates are formed due to the enhanced mobility of solute atoms. Also, coarsening of precipitates may occur due to dislocations acting as short cut of diffusion path for solute atoms during dynamic strain aging.

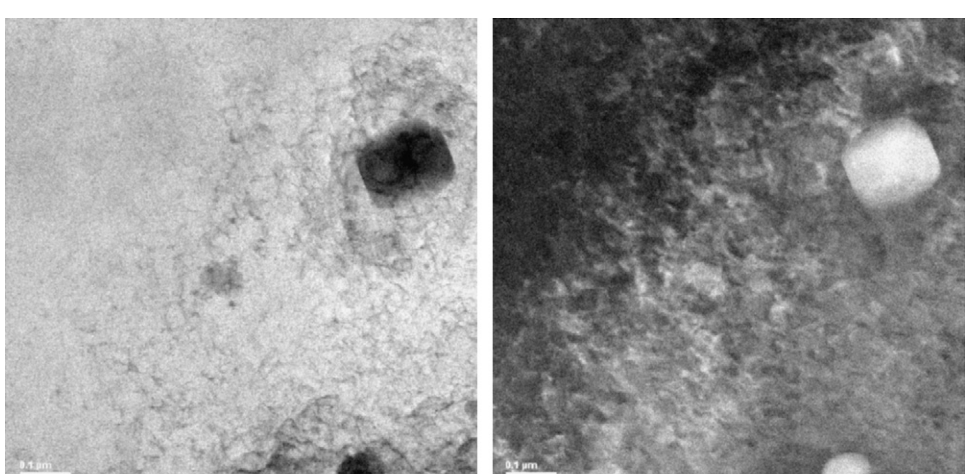

Fig. 5. Bright field and dark images of the alloy in transverse direction (TD) of the as-pressed billet after one pass at 150 °C and 0.0025 s^{-1}.

At the strain rates higher that dynamic aging regime since the deforming time decreases or strain rate increases, the chance of solute atoms to diffuse and lock the mobile dislocations decreases at a constant temperature. However, in very high strain rates, solute atoms cannot move fast enough to lock dislocations successfully and the hardness decreases consequently.

4. Conclusion

In this research, by coupling the effects of ECAE and DSA a novel strengthening technique is introduced. Despite most aging researches fulfilled in the literature, this research is carried out on an air cooled sample with minimal in-solution content. However, using the optimum processing conditions for this promising strengthening technique (ECAE at 150 °C and 0.0025 s^{-1}) a high strength product with yield strength of 178.4 MPa can be produced. The drop in hardness and yield strength along with a dramatic increase in electrical resistivity above a critical strain rate at processing temperatures of 150 °C and 200 °C can be attributed to the dissolution of fine precipitates that enhance the DSA process of the tested alloy according to McCormick's aging theory [2].

Acknowledgments

The authors would like to thank the Iran National Science Foundation and the Sharif University of Technology, Tehran, Iran for their financial supports and the research facilities used in this work. Also, the help of Mr. A.R. Eivani at Delft university of Technology for carrying out the electrical resistivity tests is highly appreciated.

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