

## Effect of Equal Channel Angular Pressing and Annealing Treatment on the Evolution of Microstructure in AlMg0.7Si Aluminum Alloy

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**Abstract:** Samples of AlMg0.7Si aluminum alloy were deformed up to three passes using equal channel angular pressing (ECAP). Formation of a sub-micron structure after three passes of ECAP was demonstrated. Microstructural stability of the samples was investigated at temperatures 300-500 °C. At 300 °C, fine recrystallized structure was formed after 10 min which was remained stable when the annealing was proceeded up to 18 h. However, at 350 °C and higher, the microstructure was quite unstable. Even by 10 s annealing, the samples exhibited recrystallized structure which turned to abnormal grain growth when temperature enhanced to 500 °C and time up to 300 sec.

**Keywords:** Equal Channel Angular Pressing, AlMg0.7Si, Microstructural Stability.

### 1. INTRODUCTION

Equal channel angular pressing (ECAP) is a unique technique to severely deform metals and alloys towards obtaining an ultrafine grain (UFG) structure [1]. During the ECAP processing of an alloy, significant grain refinement occurs together with, significant strain hardening which results in remarkable enhancement of strength. Most of the existing reports on the strengthening of metals and alloys with severe plastic deformation (SPD) are focused on room temperature properties of these products [2–4]. However, it is important to have the knowledge on the stability of microstructure when these products are supposed to service at temperatures higher than room temperature. In fact, due to the extremely high level of deformation, the UFG products are involved with a high stored energy which can cause significant microstructural instability, as it has been reported in many metals and alloys [5–9]. Therefore, knowledge on microstructural stability of UFG materials leading to determination of their service temperature is quite important. There are a few reports in the open literature which discuss the stability of microstructure in UFG materials [5,10] and in specific for aluminum alloys [11–13]. However, the required information on

AlMg0.7Si aluminum alloy is limited. This may be due to the limited application of these alloys at high temperature or the wide industrial application of this alloy which causes any related information as classified.

In addition to the above-mentioned applications for studying the microstructural stability in UFG materials, it should be noted that alloys with fine and recrystallized grain structures may be produced using a combination of severe plastic deformation and annealing. Such microstructures in metals and alloys may be interesting due to two important applications, i.e., improvement of ductility of UFG materials and superplastic applications. In fact, during SPD processing, elongation as an essential property of alloys in most structural applications is tremendously reduced [2] which may limit the use of UFG products in some critical applications. However, limited ductility of UFG materials does not necessarily mean that these materials with their unique mechanical properties are not interesting. Indeed, a unique application of SPD processing may be production of stress relieved fine grain materials which can provide high elongation while keeping relatively high strength.

Stress relieved and equiaxed grain structures are as well convenient for superplastic applications if

they provide reasonable stability of microstructure [14–17]. Metals with fine and stable grain structure can show considerable superplastic deformation [14–17]. ECAP has been extensively used for preparation of metals with fine grain structure for superplastic deformation [15, 18–22]. Therefore, it is important to understand the effect of severe plastic deformation and consequent annealing on the grain structure of an alloy and indicate how the stability of the produced microstructure may change during high temperature deformation, e.g., superplastic deformation. To the knowledge of the authors, there is a lack of such information in the open literature. According to what was explained above, the aim of this study is to understand the annealing behavior of a AlMg0.7Si aluminum alloy after SPD processing.

## 2. EXPERIMENTAL PROCEDURE

AlMg0.7Si aluminium alloy rods were received in the form of billets having a diameter of 100 mm. Chemical composition of the billet material is shown in Table 1. The bars were machined to make cylindrical samples with a length of 120 mm and a diameter of 20 mm. The samples were annealed at 550 °C for 30 min to remove the effects of the stresses induced during the previous cycles of production.

ECAP was performed using a die consisting of two round channels of 20 mm diameter and equal in cross section intersecting at an angle of 90° and an outer curved corner of 22.5°. By using this set up, an accumulative strain of one would be imposed to the alloy in each pass of deformation. The samples were ECAPed at room temperature and a ram speed of 10 mm/s for up to three passes using rout A of deformation. 20 mm from each side of the ECAPed samples were cut in order to avoid the end effects on the microstructures and

the rests was cut in even pieces of 10 mm in length. Microstructural investigation of all the samples was performed in longitudinal cross section.

The samples were annealed in a salt bath at 300, 350, 420 and 500 °C. Since the aim of this study was to investigate the stability of the deformed microstructures, annealing was performed on samples with the maximum deformation, i.e., three passes. The grain structure was studied by a polarized light optical microscope. The samples were examined using a JEOL 6500 FEG-SEM.

## 3. RESULTS AND DISCUSSIONS

### 3. 1. Initial Microstructure

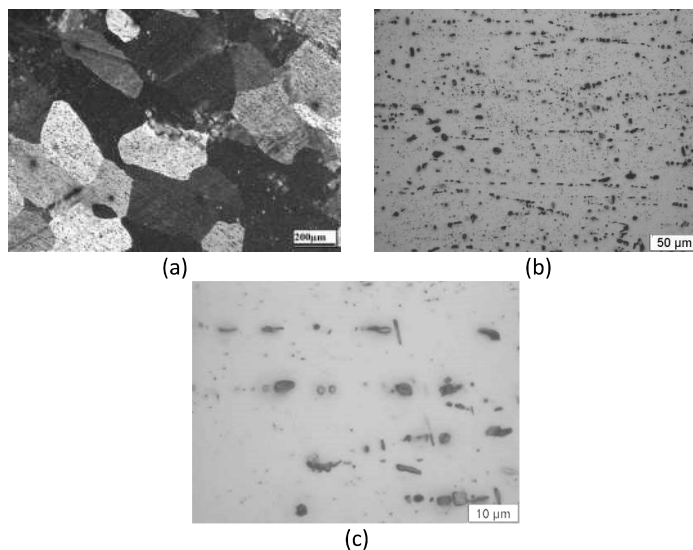
Initial microstructure of the AlMg0.7Si sample prior to ECAP is shown in Fig. 1. A fully recrystallized, coarse and equi-axed grain structure can be seen in this figure. Acquiring such microstructure is an outcome of applying long annealing at high temperature, i.e., 30 min at 550 °C, which has resulted in full recrystallization of the sample. The distribution of second phase particles has been shown in Fig. 1 (b) and (c). In Fig. 1 (b), a relatively high fraction of second phase particles which are homogeneously distributed is observed which is routine for 6xxx series aluminum alloys. In Fig. 1 (c), one can see that the particles are relatively coarse, within a size range around 1 to 5 µm.

### 3. 2. As-Deformed Microstructures

Microstructures of the samples after 1, 2 and 3 passes of ECAP are shown in Fig. 2. After one pass ECAP (Fig. 2(b)), the grains are elongated and a few shear bands are observed. By increasing the number of ECAP passes to 2, yet the initial grains are detectable. Formation of a

**Table 1.** Chemical composition of the alloy used in this study.

Element	Al	Si	Fe	Cu	Mn	Mg	Zn	Cr
Wt.%	Base	0.3	0.3	0.1	0.1	0.6	0.1	0.1

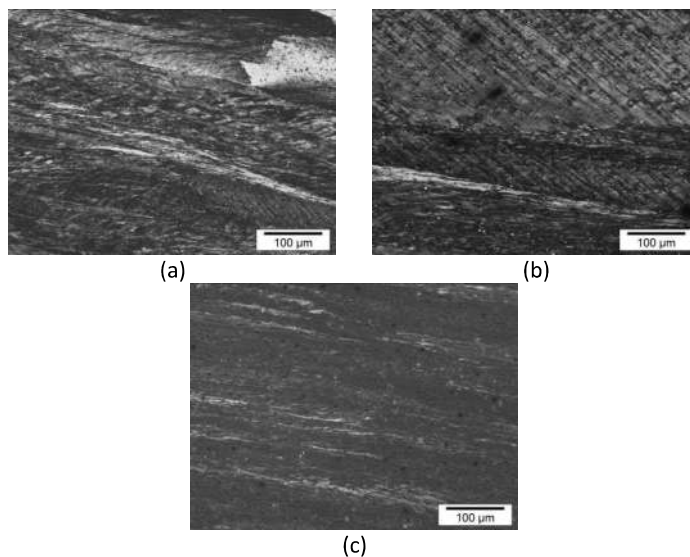


**Fig.1.** Initial microstructure of the alloy used in this investigation, (a) grain structure, (b) and (c) distribution of second phase particles at low and high magnification.

large number of shear bands at an angle of  $45^\circ$  with the elongation direction of grains is observed. Since the initial grains are yet detectable, formation of a submicron grain structure by grain fragmentation mechanism is not likely to be occurred at this stage. After 3 passes of ECAP, the initial grains are hardly

detectable. This indicates that the initial grains are extra-thinned which may indicate the occurrence of dynamic recrystallization (DRX) and formation of UFG structure by grain fragmentation.

The effect of up to 3 passes of ECAP deformation on the evolution of second phase

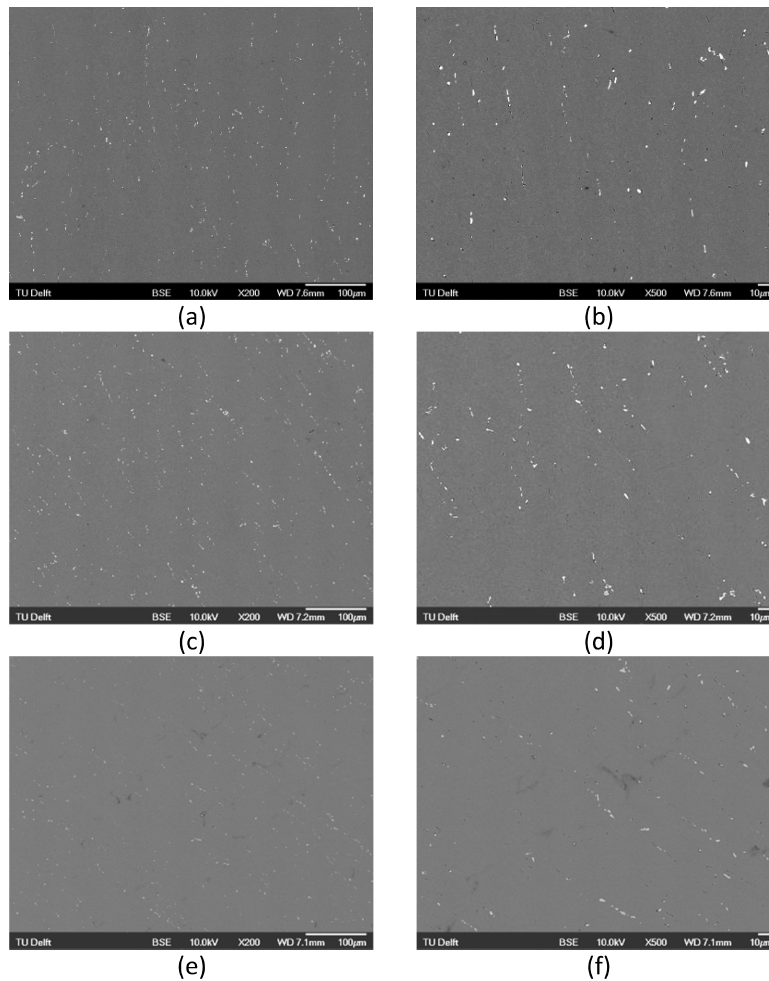


**Fig. 2.** Microstructure of the alloy after (a) one, (b) two and (c) three passes ECAP.

particles has been shown in Fig. 3. It is hardly possible to indicate any effect of ECAP deformation on the volume fraction, sizes or distribution of second phase particles. The size and distribution of these particles are extremely important since they can affect the recrystallization response and thereby, the microstructural stability of the alloy. In fact, if the size of the particles is larger than 1  $\mu\text{m}$ , they can act as recrystallization nucleation sites by particle stimulated nucleation (PSN) mechanism [23]. However, if the particles are finer than 100 nm, they may act as recrystallization inhibitors by Zener pinning effect [23].

Obviously optical microscopy does not provide enough resolution to detect the

substructure with average sizes around or less than 1  $\mu\text{m}$ . In order to demonstrate the formation of the substructure around or smaller than 1  $\mu\text{m}$ , backscattered FEG-SEM images of the sample deformed for three passes are shown in Fig. 4. It is clear that a sub-micron cellular structure is formed. This substructure is probably formed by geometrical dynamic recrystallization (GDRX) mechanism [23]. GDRX occurs as a result of extra-thinning of parent grains to the order of subgrain sizes leading to formation of new grains [23]. In fact, if a metal or alloy is subjected to a large deformation, steep reduction in the thickness of the grains occurs. Due to continuity of the material and the presence of particles, the grain boundaries become serrated. By extra-



**Fig. 3.** Low and high magnification SEM images showing the distribution of second phase particles after (a) and (b) one, (c) and (d) two, (e) and (f) three passes ECAP.

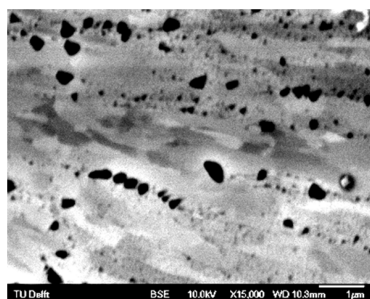


Fig. 4. Backscatter FEG-SEM image of the AlMg0.7Si sample after three passes ECAP.

thinning of grains with serrated boundaries, interpenetration of grain boundaries occurs, resulting in a microstructure composed of small equiaxed grains.

### 3. 3. Effect of Annealing Treatment on Microstructure

Microstructural evolution of the samples annealed at 300, 350, 420 and 500 °C for 10 s are shown in Fig. 5. As explained in the section Materials and Methods, only the samples deformed for three passes which result in

submicron structure are annealed and discussed from this point further. It is clear in the figure that by increasing temperature, the structure is promoted towards recrystallization. At 300 °C, the sample hardly indicates recrystallized grains. At 350 °C, partial recrystallization has occurred at 350 °C evidenced by a few equiaxed fine grains inside the elongated grain structure. At higher temperatures, i.e., 420 and 500 °C, both samples are fully recrystallized. However, it can be seen that the microstructure shown in Fig. 5 (d), attributed to annealing at 500 °C, is coarser than that shown in Fig. 5 (c) which is annealed at 420 °C.

It is obvious that such short time annealing, i.e., 10 s, would not provide enough useful information on the stability of the structure. Therefore, annealing is performed for longer periods of time at different temperatures. Microstructural evolution of the samples annealed at 300 °C is presented in Fig. 6. It is clear from Fig. 6 (a) that recrystallization may hardly occur after 30 s. However, after 120 s, few equiaxed and fine grains are formed which turn the structure to a partially recrystallized one. With further annealing to 300 s, the fraction of

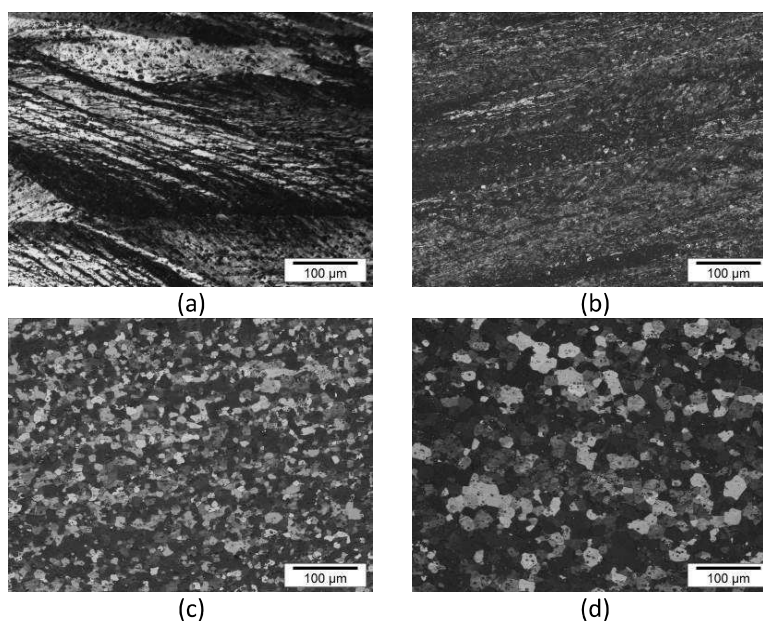
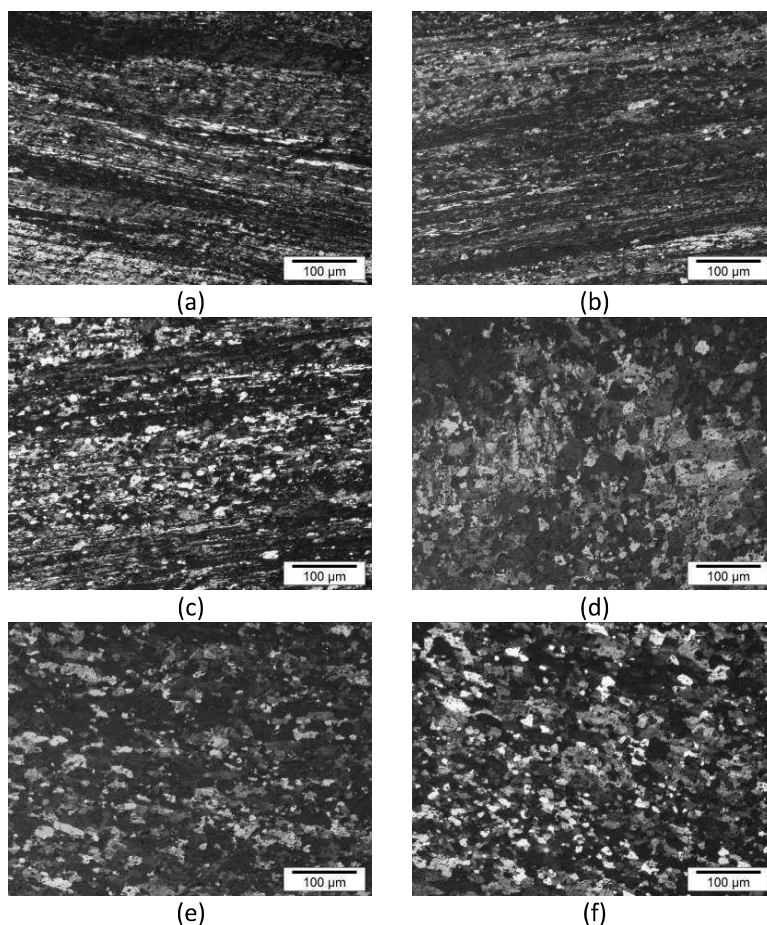


Fig. 5. Microstructure of the 3 pass deformed samples after annealing for 10 s at (a) 300 (b) 350, (c) 420 and (d) 500 °C.



**Fig. 6.** Microstructural evolution of the 3 pass deformed samples during annealing at 300 °C for (a) 30 (b) 120, (c) 300, (e) 600 s, (f) 1 and (d) 18 h.

recrystallized grains is increased. When the annealing time is increased to 10 min, a fully recrystallized microstructure is observed. The most interesting point is that the microstructure is quite stable at this temperature evidenced by the fact that no recognizable grain growth has occurred after 1 and up to 18 h annealing.

Microstructures of the samples annealed at 350 and 420 °C for 30 s and 18 h are shown in Fig. 7. It can be seen that both samples are fully recrystallized after 30 s and do not undergo further change with increasing annealing time to 18 h. This indicates that after the samples are fully recrystallized, they do not undergo further microstructural evolution, i.e., normal or abnormal grain growth, with increasing annealing time. This is in general in line with

what was found for annealing at 300 °C. However, one can indicate the effect of increasing annealing temperature as reducing the time to achieve fully recrystallized grain structures. In addition, the microstructure is relatively coarser when the sample is annealed at higher temperature.

Effect of annealing at 500 °C on the evolution of microstructure is shown in Fig. 8. At 500 °C, no significant difference between the samples annealed for 10 s, shown in Fig. 5 (d), is observed with that annealed for 30 sec, shown in Fig. 8 (a). In fact, both microstructures are fully recrystallized with relatively fine microstructures. In addition, no sign of abnormal grain growth is observed when the annealing time is short, i.e., up to 120 s. However, when

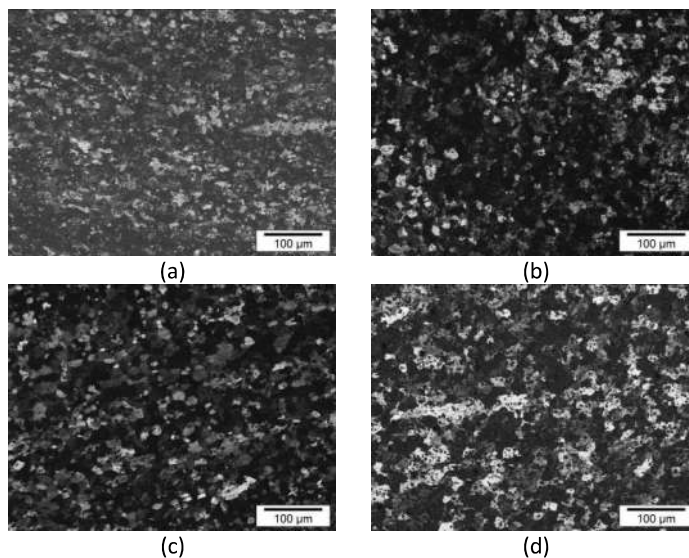


Fig. 7. Microstructure of the 3 pass deformed samples after annealing, (a) 30 s and (b) 18 h at 350 °C, (c) 30 sec and (d) 18h at 420 °C.

annealing is continued for longer than 120 s, the microstructure becomes highly unstable and abnormal grain growth may occur. In order to detect the onset of abnormal grain growth, the whole 10 by 20 mm<sup>2</sup> area of the samples is investigated using optical microscopy. It was guaranteed that no sign of abnormal grain growth is observed up till 120 s annealing at 500 °C.

However, only one single grain, as shown in Fig. 8 (c), becomes unstable and starts to grow abnormally after 5 min. It is of interest to note that it is the only one single grain in the whole area indicating the onset of abnormal grain growth. At longer time, Fig. 8 (d), abnormal grain growth is more clearly seen.

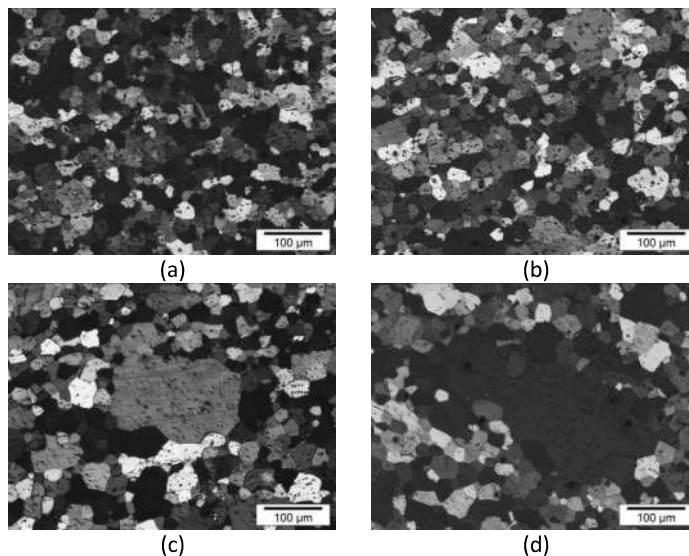
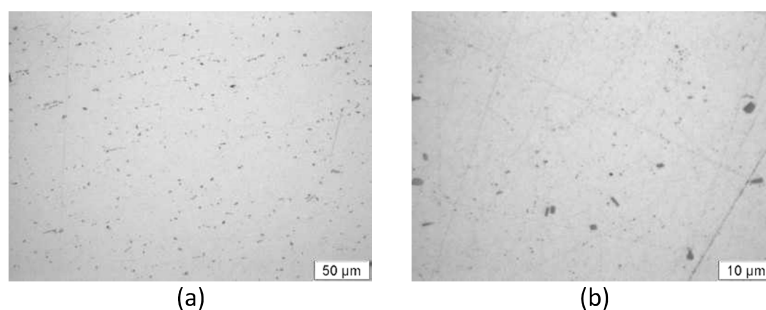


Fig. 8. Microstructural evolution of the 3 pass deformed samples at 500 °C for (a) 30 (b) 120, (c) 300 and (d) 600 sec.



**Fig. 9.** (a) low and (b) high magnification images showing the distribution of second phase particles in the alloy after annealing at 500 °C for 1 hr.

Distribution of second phase particles in the sample with three passes ECAP and annealing at 500 °C for 1 hr is shown in Fig. 9. In comparison to the distribution of particles in the initial state shown in Fig. 1, it is clear that application of annealing has resulted in dissolution of second phase particles. It is generally known that a very large grain always grows more slowly than the average grains relative to the grain assembly and will eventually re-join the normal size distribution [23]. In addition, a dispersion of second-phase particles, which definitely exist in the AlMg0.7Si alloy [24], will prevent growth above the limiting grain size. However, at high temperatures, e.g., 500 °C, these particles may dissolve as shown in Fig. 9 in comparison to Fig. 1 [25]. The dissolution of these particles does not occur at once and overall. Therefore, some regions of the materials may be locally evacuated of the particles which can provide the possibility of sudden growth of some grains indicated by abnormal grain growth.

#### 4. CONCLUSIONS

AlMg0.7Si aluminium alloy was deformed up to three passes using equal channel angular pressing (ECAP), aiming at producing ultrafine grained structure. Microstructural stability of the samples was investigated at temperatures between 300-500 °C. According to the results of the current investigation, the following conclusions were made;

1. Formation of ultra-fine grain structure in

AlMg0.7Si alloy after three passes of ECAP was demonstrated.

2. The structure was unstable during annealing at temperatures higher than 300 °C and indicated a great desire towards recrystallization.
3. When the annealing temperature was increased to 500 °C, the structure becomes highly unstable and abnormal grain growth occurred even if the treatment was performed for a relatively short period of time, e.g., 300 s.

#### REFERENCES

1. Segal, V. M., "Materials processing by simple shear", *Mater. Sci. Eng. A.*, 1995, 197,157–164.
2. Valiev, R. Z., Langdon, T. G., "Principles of equal-channel angular pressing as a processing tool for grain refinement", *Prog. Mater. Sci.*, 2006, 51, 881–981.
3. Kim, W. J., Chung, C. S., Ma, D. S., Hong, S. I., Kim, H. K., "Optimization of strength and ductility of 2024 Al by equal channel angular pressing (ECAP) and post-ECAP aging", *Scr. Mater.*, 2003, 49, 333–338.
4. Kim, W. J., Kim, J. K., Park, T. Y., Hong, S. I., Kim, D. I., Kim, Y. S., Lee, J. D., "Enhancement of strength and superplasticity in a 6061 Al alloy processed by equal-channel-angular-pressing", *Metall. Mater. Trans. A.*, 2002, 33, 3155–3164.
5. Lugo, N., Liorca, N., Sunol, J. J., Cabrera, J. M., "Thermal stability of ultrafine grains size of



- pure copper obtained by equal-channel angular pressing”, *J. Mater. Sci.*, 2010, 45, 2264–2273.
6. Cao, W. Q., Gu, C. F., Pereloma, E. V., Davies, C. H., “Stored energy, vacancies and thermal stability of ultra-fine grained copper”, *Mater. Sci. Eng. A.*, 2008, 492, 74–79.
  7. Zhang, Y., Wang, J. T., Cheng, C., Liu, J., “Stored energy and recrystallization temperature in high purity copper after equal channel angular pressing”, *J. Mater. Sci.*, 2008, 43, 7326.
  8. Gubicza, J., Chinh, N. Q., Lábár, J. L., Hegedus, Z., Langdon, T. G., “Principles of self-annealing in silver processed by equal-channel angular pressing: The significance of a very low stacking fault energy”, *Mater. Sci. Eng. A.*, 2010, 527, 752–760.
  9. Chen, Y. J., Li, Y. J., Walmsley, J. C., Dumoulin, S., Gireesh, S. S., Armada, S., Skaret, P. C., Roven, H. J., “Quantitative analysis of grain refinement in titanium during equal channel angular pressing”, *Scr. Mater.*, 2011, 64, 904–907.
  10. Zhilyaev, A. P., Nurislamova, G. V., Valiev, R. Z., Baro, M. D., Langdon, T. G., “Thermal stability and microstructural evolution in ultrafine-grained nickel after equal-channel angular pressing (ECAP)”, *Metall. Mater. Trans. A.*, 2002, 33, 1865–1868.
  11. Mao, J., Kang, S. B., Park, J. O., “Grain refinement, thermal stability and tensile properties of 2024 aluminum alloy after equal-channel angular pressing”, *J. Mater. Process. Technol.*, 2005, 159, 314–320.
  12. Iwahashi, Y., Horita, Z., Nemoto, M. and Langdon, T. G., “Factors influencing the equilibrium grain size in equal-channel angular pressing: Role of Mg additions to aluminum”, *Metall. Mater. Trans. A.*, 1998, 29, 2503–2510.
  13. Horita, Z., Fujinami, T., Nemoto, M. and Langdon, T. G., Equal-channel angular pressing of commercial aluminum alloys: grain refinement, thermal stability and tensile properties, *Metall. Mater. Trans. A.*, 2000, 31, 691–701.
  14. Ashby, M. F. and Verrall, R. A., “Diffusion-accommodated flow and superplasticity”, *Acta Metall.*, 1973, 21, 149–163.
  15. McFadden, S. X., Mishra, R. S., Valiev, R. Z., Zhilyaev, A. P., Mukherjee, A. K., “Low-temperature superplasticity in nanostructured nickel and metal alloys”, *Nature.*, 1999, 398, 684.
  16. Edington, J. W., Melton, K. N., Cutler, C. P., “Superplasticity”, *Prog. Mater. Sci.*, 1976, 21, 61–170.
  17. Sherby, O. D. and Wadsworth, J., “Superplasticity-Recent advances and future directions”, *Prog. Mater. Sci.*, 1989, 33, 169–221.
  18. Berbon, P. B., Komura, S., Utsunomiya, A., Horita, Z., Furukawa, M., Nemoto, M. and Langdon, T. G., “An evaluation of superplasticity in aluminum-scandium alloys processed by equal-channel angular pressing”, *Mater. Trans. JIM.*, 1999, 40, 772–778.
  19. Figueiredo, R. B. and Langdon, T. G., “Record Superplastic Ductility in a Magnesium Alloy Processed by Equal-Channel Angular Pressing”, *Adv. Eng. Mater.*, 2008, 10, 37–40.
  20. Watanabe, H., Mukai, T., Ishikawa, K. and Higashi, K., “Low temperature superplasticity of a fine-grained ZK60 magnesium alloy processed by equal-channel-angular extrusion”, *Scr. Mater.*, 2002, 46, 851–856.
  21. Komura, S., Furukawa, M., Horita, Z., Nemoto, M. and Langdon, T. G., “Optimizing the procedure of equal-channel angular pressing for maximum superplasticity”, *Mater. Sci. Eng. A.*, 2001, 297, 111–118.
  22. Kim, W. J., Kim, J. K., Park, T. Y., Hong, S. I., Kim, D. I., Kim, Y. S., Lee, J. D., “Enhancement of strength and superplasticity in a 6061 Al alloy processed by equal-channel-angular-pressing”, *Metall. Mater. Trans. A.*, 2002, 33, 3155–3164.
  23. Humphreys, F. J., Hatherly, M., “Recrystallization and related annealing phenomena, Elsevier, Kidlington, UK, 2012, 113-114 and 293-302 and 368-378 and 427-436.
  24. Samaras, S. N. and Haidemenopoulos, G. N., “Modelling of microsegregation and homogenization of 6061 extrudable Al-alloy, *J. Mater. Process. Technol.*, 2007, 194, 63–73.
  25. Birol, Y., “Precipitation during homogenization cooling in AlMgSi alloys”, *Trans. Nonferrous Met. Soc. China.*, 2013, 23, 1875–1881.