Development of the continuous process method for ECAP using a tri-axis rotary die and microstructural evolution of semi-solid aluminium alloy

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Abstract

The continuous ECAP process which can provide three-dimensional strain was developed in this study. A die with tri-axis crossed channels having channel angle φ of 90° and curvature ψ of 0° was created. As a result, continuous ECAP process via route Bc for AC4CH aluminium semi-solid alloy could be performed at 573K at pressing speed of 0.5mm/sec using the developed die. The continuous method cut the processing time in half compared to the non-continuously conventional method. The grains became fine from 100µm to approximately 2µm. Workpiece processed 8 passes exhibited four times higher toughness than as-cast alloy.

Keywords: equal-channel angular pressing, severe plastic deformation, ultrafine grained, semi-solid cast, continuous process, Tri-RD-ECAP, Al-Si-Mg alloy.

1 Introduction

The process of grain refinement in metal materials is generally known as the way to improve their strength and ductility without losing toughness. In recent years, much attention has been paid to methods which can give the grain refinement by severe deformation. The methods can improve the microstructure of alloy more energy efficient compared to rapid solidification, stirring during solidification, and heat treatment. Equal-Channel Angular Pressing (ECAP) is especially different from other kinds of the method such as using rolling and drawing, and is not restricted in bulk shape. This implies there is no limit for the amount of



accumulated strain which can be provided during the process. Many researches regarding ECAP process with various materials has been reported (e.g. [1]) after Segal [2] had first introduced the method in 1981.

The application of ECAP method induces shear strain under hydrostatic pressure, and leads to expression of the expected effect to the materials by multiple processing. In other words, it can't reach severe plastic deformation without any iteration. However, it is apparent that establishing continuous manufacture is most important for practical use. At present, the conventional process is difficult to apply ECAP method as industrial technology because there is a need to take apart a die every time and then thermal history of a workpiece must be controlled. In order to solve the problem, RD-ECAP method developed by Nishida in 2000 is capable of processing continuously, but has an issue which the processing route is restricted [3, 4].

The purpose of this study is to develop the method of the continuous ECAP process which fabricates a sample via arbitrary processing route. Comparing the non-continuously conventional method, it is checked the validity of the method using the developed die. By establishing to process without the work of taking a workpiece from die, concepts of this method are as follows: (1) to improve production efficiency, (2) to reduce thermal variation applied to a workpiece, and (3) to permit to perform ECAP process via Bc route. The microstructural evolution of semi-solid aluminum alloy with ECAP process and the effect for improving material properties are discussed.

2 Processing of equal-channel angular pressing

ECAP method is one of the severe plastic deformations which create ultrafine grain and provides improvement of mechanical properties of metal materials. Figure 1 depicts the schematic of a die using the conventional ECAP process. Features of the die are the intersection angle of the two channels, φ , and the curvature of the outer corner, ψ . When a billet is pushed into a channel which is



Figure 1: Schematic of a typical die for ECAP process.



bent through an abrupt angle, simple shearing is applied to it at the corner of channel. Repeated process can be performed because the die channel of entry and exit side has same cross-section shape. The equivalent strain, ε , generated in the workpiece depends on the configuration of die corner and number of ECAP passes, N, and is theoretically given by the following relation [5].

$$\varepsilon = \frac{N}{\sqrt{3}} \left[2 \cot\left(\frac{\varphi}{2} + \frac{\psi}{2}\right) + \psi \csc\left(\frac{\varphi}{2} + \frac{\psi}{2}\right) \right]$$
(1)

If a workpiece is rotated 90° in the same sense about extrusion direction axis between consecutive passes, called route Bc, it is known that better fine microstructure can be obtained due to a generated three-dimensional strain [6]. It is reported that grains become fine resulting from dynamic recrystallization which is accompanied by severe deformation without work hardening [7, 8].

3 Experimental

3.1 Test material

Workpieces having rectangular shape $(10\text{mm} \times 10\text{mm} \times 60\text{mm})$ for the ECAP process is prepared from the AC4CH aluminium semi-solid alloy ingot without heat treatment. The chemical composition of the applied ingot is as follows (in mass %); 7.14Si, 0.41Mg, 0.01Cu, 0.01Zn, 0.11Fe, 0.01Mn, 0.01Ni, 0.04Ti, and the balance Al.

3.2 Continuous ECAP process using a tri-axis rotary die

A developed die is shown in fig. 2. The channel configuration is through the three axes which are met perpendicularly in the centre of this die with square section $(10 \text{mm} \times 10 \text{mm})$.



Figure 2: Schematic of a Tri-RD-ECAP facility. The die has a channel which is orthogonal three axes in shape, i.e. $\varphi 90^\circ$, $\psi 0^\circ$.



Procedures of ECAP process using the developed die are as follows: Firstly, L-shaped channel, i.e. $\varphi=90^{\circ}$, $\psi=0^{\circ}$, is formed by filling four ways (e.g. O-C, O-D, O-E and O-F in fig. 2) with plunger. Exits of blocked way are restrained by support parts. Secondly, the first pass of ECAP is completed when a workpiece is inserted through one free face side of the die and is pushed out toward another side with plunger, i.e. the workpiece is taken a path of A-O-B and is finally located in O-B. Thirdly, the workpiece is processed via route Bc if taken a second path of B-O-C by rotating the die until B-side faces upward and C-side is set up on free face: The ECAP processing route can be arranged depending on procedure of the rotation of the die.

By repeated this operation, it is possible to perform ECAP process continuously via arbitrary processing route. It is expected ϵ =1.15 is applied to a workpiece theoretically during 1 pass. We termed this method "Tri-RD-ECAP". In contrast, the non-continuous ECAP is called "the conventional method" in this paper.

3.3 Fabrication of samples by Tri-RD-ECAP

Samples are fabricated using the die for Tri-RD-ECAP described above. For validation of this developed method, a conventional split die with one-way channel (φ =90°, ψ =0°) is also used for comparative discussion. In addition how the process influences microstructure and mechanical properties of materials is investigated by evaluation tests described later in this chapter.

A workpiece is extruded by pressing a plunger at constant speed using a universal testing machine (Shimadzu Corp., AG-500kNI). Tool temperature is controlled using cartridge heaters which are set up in a substructure and walls. A graphite lubricant is used between a sample and the die. Experiment condition is listed in table 1. Press load required for extrusion is monitored during the process.

	Tri-RD-ECAP (continuous process)	Conventional ECAP (non-continuous process)	
Die			
Processing temperature [K]	573	473	573
Extrusion speed [mm/s]	0.5		
ECAP Processing route	Bc		
Number of ECAP passes	0 (as-cast), 4, 8		

Table 1: Conditions of fabricating samples.





Evaluation tests of a sample are as follows:

Tensile test. Quasi-static deformation characteristic at the average strain rate of 1.6×103 /sec is obtained at room temperature using a universal testing machine (INSTRON Corp., 5500R). Dumbbell specimen with 12mm in initial gauge length and 4mm in width is prepared by cutting along extrusion direction using wire-electric discharge machine.

Impact test. Toughness characteristic is obtained using Charpy impact testing machine (Shimadzu Corp., 5kgf·m). Specimens are prepared based on JIS NO.3 test piece (JIS Z2202). Fracture surface of a sample after test is observed using Scanning Electron Microscope (SEM / JEOL Ltd., JSM7001FD).

Microstructure observation. Samples are mechanically polished and are observed using an optical microscope. Electron Back Scattering Diffraction analysis (EBSD) is also performed using SEM for measurement of grain size.

4 Results and discussion

4.1 Comparison of the method of Tri-RD-ECAP and a conventional ECAP

In this experimental, the processing time after insert of a workpiece and before completion of pressed 4 passes was 31minutes by the continuous method. Comparing that with 59minutes by the non-continuous conventional method, the development of the continuous processing method improves production efficiency.

The relationship between the number of processing passes and the maximum value of press load for each pass is shown in fig. 3. Tri-RD-ECAP put more strain on the die than the conventional method. The fact suggests that problems specific to a split die occurs dominantly because the gap between die parts is enlarged due to complexity of die composition. And the developed die increase the press load with numbering up the processing pass while the conventional die show the opposite as shown in Figure. Since a workpiece is expanded to all over



Figure 3: Change in the maximum value of press load required for ECAP with the number of processing passes.

the path after second passes, this is attributed to the fact that a friction is largely influenced between path walls and a workpiece. Especially, it is shown a high increasing rate of press load after seventh passes because a workpiece runs though the path which is taken before.

Next, we report on the mechanical properties of ECAP processed sample. Static tensile strength and fracture strain are shown in figs. 4(a) and 4(b) respectively. A sample pressed at 573K by Tri-RD-ECAP exhibited the intermediate properties between the materials pressed at 473K and at 573K by the conventional method. This implies that the effect of thermal variation reduction by processing continuously is operating. The microstructure developed by warm or hot ECAP should always be affected by interactions of deformation,



Figure 4: Influence of ECAP process on (a) static tensile strength and (b) fracture strain. Material strength is slightly decreased and its ductility is increased with further processing. The mechanical properties of the continuous ECAPed material correspond to the case of material which is processed by conventional ECAP at lower temperature.



i.e. cause of dynamic recrystallization, and annealing, i.e. static recrystallization [9]; the static annealing may be occurred when the material remains inside straight segments of the heated channel, except for the corner part where simple shear is applied at, and is removed from the die between the processing passes. As the result, it has been reported that an elevation in the processing temperature provides to decreasing the volume fraction of new grains and increasing the average grain size due to a reduction in the accumulation efficiency of strain [10]. Although there is no distinct difference in these microstructure observations, the result leads to our presumption that the reduction of thermal variation may decrease the effect of the static recrystallization.

Figure 5 depicts the result of Charpy impact value. While strength was slightly decreased and ductility was increased with further processing, material toughness indicated by the impact value was greatly improved. We have plotted additionally the evaluation result of a sample processed at 473K using the other conventional die (φ =90°, ψ =15°) reported in ref. [11] in fig. 5. It is extremely interesting data that the same improvement in material toughness is provided without depending on processing method, i.e. thermal history, processing temperature, and configuration of die corner. Particularly, the size of ψ is the parameter regarding the processing strain as is obvious from equation (1), and many researches have been investigated the effect of it on ECAP processing workpiece. There are past findings that deformation behavior is less related it if ψ is lower than 20° [12, 13]; this can be explained by investigations that the dependence of strain distribution on ψ is decreased with a higher work hardening material [14] and that an apparent angle of ψ becomes 22° on a quasi-perfect



Figure 5: Influence of ECAP process on impact properties. Material toughness is improved by ECAP without depending on processing method, processing temperature, and configuration of die corner.

plastic solid due to the occurrence of dead-zone [15]. The results of this study correspond to this case. Moreover, pictures of fracture cross-section of samples after impact test are shown in fig. 6. It is observed that the surface become dimple type specific to high ductility materials by ECAP.



Figure 6: Appearance of fracture of (a) as-cast AC4CH semi-solid alloy, and materials pressed (b) 4 passes and (c) 8passes by Tri-RD-ECAP from Charpy impact test.

In conclusion, although having a drawback of requiring high press load, Tri-RD-ECAP method reduces production time and thermal variation applied to a workpiece. We have verified the validity of Tri-RD-ECAP as the toughness of AC4CH semi-solid alloy is improved. It can be expected that this approach contributed to enhancement of the possibility for a way to improve mechanical properties by ECAP processing.

4.2 Evolution of semi-solid microstructure by ECAP process

Figure 7 depicts microstructure photographs of a sample before and after Tri-RD-ECAP. With further processing, the globular primary α -Al and the eutectic phase in the construction of a semi-solid AC4CH alloy is mixed and is dispersed finely.

Furthermore, the observation of crystal orientation mapping is shown in fig. 8. Grains with 100 μ m in diameter at first became fine to approximately 2 μ m by ECAP. Note that one Al grain defined as a same crystal orientation region includes some globular primary α and eutectic α (see fig. 8(a)). Although the process of grain refinement makes generally a material strengthen, the effect is complicated in the case of eutectic materials.





Figure 7: Microstructure of (a) as-cast AC4CH semi-solid alloy, and materials pressed (b) 4 passes and (c) 8passes by Tri-RD-ECAP. Whitish area; primary α -Al, Dark area; α +Si+Mg2Si.



Figure 8: Crystal orientation mapping normal direction by EBSD. (a) As-cast AC4CH semi-solid alloy, and materials pressed (b) 4 passes and (c) 8 passes by Tri-RD-ECAP. The ultrafine-grained aluminum can be obtained by processing ECAP.



It has been reported by Nakayama that the morphology of eutectic Si particles is greatly associated with micro-crack generation and propagation [16]. It is expected that a material indicates ductile property on local deformation due to high ductility of primary Al if having microstructure whose primary Al is dispersed finely and homogeneously and is connected like net as shown in fig. 7(b) or fig. 7(c); this presumption agrees with past findings [4]. Thus, the developed ECAP method can improve material ductility and toughness without much decreasing strength for AC4CH semi-solid alloy.

5 Conclusion

We have developed the method of the continuous ECAP process, Tri-RD-ECAP, which fabricates a sample via arbitrary processing route and have discussed the microstructural evolution of semi-solid aluminum alloy with ECAP process and the effect for improving material properties. The results we obtain are as follows:

- 1) Continuous ECAP process with 8 passes via route Bc for AC4CH aluminium semi-solid alloy can be performed at 573K at pressing speed of 0.5mm/sec using a tri-axis rotary die (φ =90°, ψ =0°).
- The extrusion load for the developed continuous ECAP process is elevated with each pass although the case of the conventional ECAP process is reduced.
- 3) When a workpiece is pressed 8 passes by ECAP, the grain size become fine from 100 μ m to approximately 2 μ m and the Charpy impact value increase four times compared to as-cast alloy as is the case in the non-continuously conventional method. The primary α -Al and the eutectic phase become complexly intertwined by processing ECAP.

References

- [1] R. Z. Valiev, T. G. Langdon, Progress in Materials Science, 51, 881-981, 2006.
- [2] V. M. Segal, Materials Science and Engineering, A197, 157-164, 1995.
- [3] Y. Nishida, H. Arima, J. C. Kim, T. Ando, Scripta Materialia, 45, 261-266, 2001.
- [4] A. Ma, K. Suzuki, Y. Nishida, N. Saito, I. Shigematsu, M. Takagi, H. Iwata, A. Watazu, T. Imura, Acta Materialia, 53, 211-220, 2005.
- [5] Y. Iwahashi, J. Wang, Z. Horita, M. Nemoto, T. G. Langdon, Scripta Materialia, 35(2), 143-146, 1996.
- [6] M. Furukawa, Z. Horita, T. G. Langdon, Materials Science and Engineering, A332, 97-109, 2002.
- [7] T. Sakai, H. Miura, Tetsu-to-Hagane, 94(12), 590-598, 2008.
- [8] T. Sakai, K. Yoshida, J. Koyama, E. Nakamachi, Journal of Japan Institute of Light Metals, **59(4)**,179-184, 2009.
- [9] O. Sitdikov, T. Sakai, E. Avtokratova, R. Kaibyshev, K. Tsuzaki, Y. Watanabe, Acta Materialia, **56**, 821-834, 2008.



- [10] I. Mazurina, T. Sakai, H. Miura, O. Sitdikov, R. Kaibyshev, Materials Science and Engineering, A486, 662-671, 2008.
- [11] F. Nomura, T. Matsuba, T. Tanaka, Y. Imaida, Advanced Materials Research, 123-125, 483-486, 2010.
- [12] C. Xu, T. G. Langdon, Scripta Materialia, 48, 1-4, 2003.
- [13] C. Xu, M. Furukawa, Z. Horita, T. G. Langdon, Materials Science and Engineering, A398, 66-76, 2005.
- [14] S. C. Yoon, P. Quang, S. I. Hong, H. S. Kim, Materials Processing Technology, 187-188, 46-50, 2007.
- [15] H. S. Kim, M. H. Seo, S. I. Hong, Materials Science and Engineering, A291(1/2), 86-90, 2000.
- [16] Y. Nakayama, T. Miyazaki, Materials Transactions, 52(11), 2045-2051, 2011.

