

INFLUENCE OF THE PROCESSING ROUTE ON THE MICROSTRUCTURE FORMATION AT SEMI-SOLID TEMPERATURES FOR ALUMINUM ALLOY

A380

Ivana Dumanić¹, Jure Krolo¹, Sonja Jozić¹, Branimir Lela¹, Dražen Bajić¹, Ljumović Petar¹

¹University of Split, Faculty of Electrical Engineering, Mechanical Engineering and Naval Architecture, Ruđera Boškovića 32, 21000 Split, Hrvatska

Original scientific paper / Izvorni znanstveni rad

Abstract

In this study, the influence of the material processing route for semi-solid thixoforming process was investigated. The main aim of this research was to achieve globular microstructure during samples heating at semi-solid temperatures. In order to achieve globular microstructure equal channel angular pressing (ECAP) was used to deform originally casted dendritic microstructure. During reheating of the cold worked billet in the semi-solid temperature range, partial remelting should result with extremely fine, uniform, and nondendritic spherical microstructure. Therefore, ECAPed specimens were subsequently heated at the appropriate temperatures above solidus to get a globular structure of A380 (AlSi9Cu3(Fe)) aluminum alloy. The microstructural evolutions during semi-solid heating at different holding times were carried out utilizing optical microscope. Conclusion indicated that the microstructure was significantly influenced by holding time. Also, it was demonstrated that slurry composed of spheroidal solid primary particles within a liquid matrix could be achieved by reheating (in semi-solid range) of the heavily deformed specimens. Finally, it was confirmed that ECAP is a promising route for processing of the semi-solid raw materials for thixoforming.

137

Keywords: *Semi-solid, aluminum, ECAP, globular microstructure, thixoforming.*

1. INTRODUCTION

Semi-solid metal (SSM) processing technologies involve the formation of metal alloys between solidus and liquidus temperatures providing the final component with enhanced properties. These technologies have some advantages such as lower energy consumption and cost [1], prolonged die life due to less thermal shock [2], limitation the risk of gas entrapment due to laminar flow [3]. SSM technologies are generally categorized into two groups: rheo-routes and thixo-routes. The rheo-route refers to the preparation of an SSM slurry directly from a liquid phase and its direct injection into a die or a mould for component shaping. The thixo-route refers to the preparation of a feedstock material, reheating of a feedstock material between solidus and liquidus temperatures and its shaping [4], [5]. It is necessary to get globular microstructure when the material is cooled (rheo-routes) or heated (thixo-routes) into semi-solid state. Such structure is required in order to achieve thixotropic properties of slurries. The fluid which surrounds solid globules acts as a lubricant and hence a better flow and lower viscosity and deformation forces are achieved [6]. Physical basics of thixotropy are: when the slurry is at rest, gravity will bring the particles into contact and the semi-solid will support its own weight and can be handled like a solid; during a deformation, shear breaks the bonds and viscous flow of material occurs [6].

In order to prepare suitable globular microstructure, numerous routes have been tested, e.g. rheo-route techniques: mechanical stirring, the cooling slope process, new rheocasting process; thixo-route techniques: thermomechanical treatments, spray casting, thixomolding [5]. One of the thermomechanical treatments routes, which has been proved to be capable to produce thixo feedstock, is severe plastic deformation technique (SPD) [5]. In severe plastic deformation (SPD) techniques high plastic strain is applied to a solid sample. As a result, the dendritic microstructure is deformed and bonds are broken. This structure, after heating at a semi-solid temperature, could be changed to the globular structure. Lately, one of the SPD techniques that has been gaining attention is equal channel angular pressing (ECAP) [5], [7], [8]. In this process, plastic deformations are achieved by pressing feedstock through channels of the same cross-sectional area. Channels are bent at an angle typically as 90° or 120° .

Several researchers investigated the microstructure of aluminum alloy produced by ECAP process. Nedjadj et al. [9], Meidani et al. [10] and Moradi et al. [11] concluded that ECAP followed by isothermal heating A356 alloy in the semi-solid range was suitable to produce semi-solid feedstock with the spheroidal solid phase. Ashouri et al. [12] and Moradi et al. [13] investigated the effects of the route and the number of passes in ECAP process for A356 alloy. It was observed that, if considering an industrial application, one-pass treated material was a good choice for semi-solid forming process. Campo et al. [14] presented that samples prepared by ECAP route have the most suitable characteristics for thixofforming A356 alloy in comparison with five various routes (commercial product, direct casting with and without electromagnetic stirring and grain refining, direct casting with mechanical vibration, direct casting followed by one ECAP pass). Aghaie-Khafri and Azimi-Yancheshme [15] concluded that for Al-Fe-Si alloy, ECAP and semi-solid heating resulted in an enhanced microstructure and mechanical properties. Also, they have found that mechanical properties of Al-Si-Fe samples after ECAP and semi-solid process were improved among the as received and only ECAPed samples [16]. Meshkabadi et al. [17] found that by processing ECAP route BA up to five passes and isothermally heating at 630°C for 15 min, the most suitable microstructure of 7075 aluminum alloy will be achieved. Torres et al. [18], [19] compared the two processing

routes for production of raw material for thixoforming. They concluded that the more suitable microstructure of Al-4.0wt%Si-2.5wt%Cu and Al-7.0wt%Si-2.5wt%Cu alloy was achieved by ECAP process than electromagnetic stirring process.

It is evident from the literature study, that no studies were found on the use of ECAP in semi-solid processing aluminum alloy A380. Therefore, the goal of this work is to produce feed material with globular structure by ECAP process followed by heating in semi-solid range for A380 alloy.

2. EXPERIMENTAL PROCEDURE

Selected alloy for the research was aluminum alloy A380 with the chemical composition given in Tab. 1.

Tab. 1: Chemical composition of aluminum alloy A380 (Wt. %)

Elements	Fe	Si	Mn	Ni	Cr	Ti	Cu	Pb	Mg	Zn	Sn
Min. - Max. (%)	1.3	8 - 11	0.55	0.55	0.15	0.25	2 - 4	0.35	0.05 - 0.55	1.2	0.15

A first experimental step was to cast selected aluminum alloy A380 (EN AC - AlSi₉Cu₃(Fe)) in the form of the round bar. Aluminum was melted at 700 °C and poured into the steel die to form round bars with 15 mm in diameter and 100 mm in length, Fig. 1. After casting samples were annealed to increase ductility at 350 °C temperature in duration of 5 hours. Samples were cooled in a closed furnace. Aluminum melting and annealing were performed in the 3 kW furnace "Demiterm Easy" with maximum working temperature of 1150 °C.



Fig. 1: Aluminum alloy A380 after casting

After samples annealing, they were processed with equal channel angular pressing (ECAP). This processed is well known as severe plastic deformation (SPD) process. Main characteristics of the SPD processes are significant crystal grain refinement and unique mechanical and physical properties improvement. In this research, ECAP process was mainly used for introduction of the significant amount of plastic deformation and cold work inside casted samples. ECAP tool used in this research has two intersecting channels with 15.1 mm in diameter. At the channel intersection, it is of the main importance to define inner and outer die angle. Die angle determines which amount of the plastic deformation will be introduced into processed material. Inner die angle for the ECAP tool used in this work is 90 °, and outer die angle is 12 ° which is defined with 3 mm radius. According to the Iwahashi et al. [20] analytical approach, this tool geometry gives total plastic shear strain around 1. Graphite grease was used for the process lubrication. ECAP tool used for this research is presented in Fig. 2.



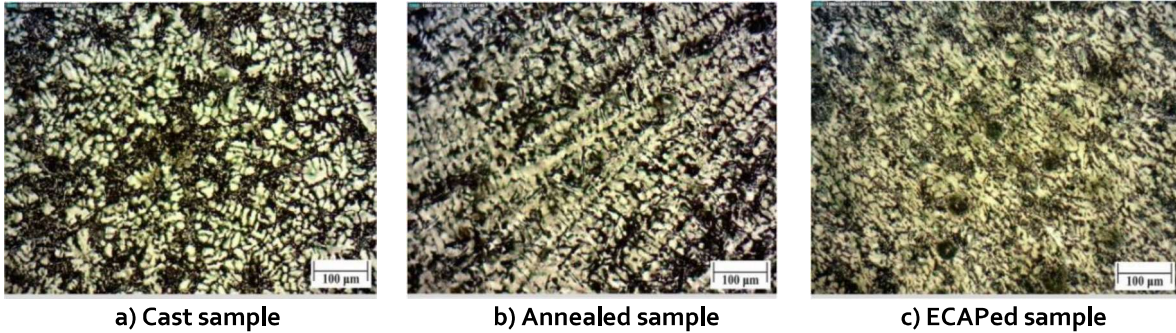
Fig. 2: ECAP tool utilized for severe plastic deformation

After ECAP process samples were heated at the appropriate semi-solid temperature. The selected temperature will define solid fraction inside samples, where 50 % of the solid fraction is a good selection for the thixoforming process [21]. According to the Birol et al. [21], the temperature to achieve 50% solid is estimated to be 567 °C for A380 alloy. Therefore, for this research selected temperature for semi-solid heating was 570 °C \pm 3 °C to fit into mentioned solid fraction range. Six casted, annealed and ECAPed samples were prepared for heating at the semi-solid temperature. Samples were held at semi-solid temperature for different times in order to determine an optimal time to achieve optimal semi-solid globular microstructure. Selected times were 1 min, 2 min, 2 min and 40 seconds, 5 min, 8 min and finally 12 min and 30 seconds, for samples A, B, C, D, E, F and G, respectively. In order to achieve good repeatability, surface temperature was measured on all sample with contact thermometer DT 02 and type K thermocouple probe. Average heating rate for all samples to reach the desired semi-solid temperature on the surface was 1,6 °C/s (average 6 minutes). After that samples were heated at the above-mentioned times. For metallography analysis, optical microscope "OPTON Axioskop" and computer software "BIOVIS MP2000" were utilized. In the first preparation step samples were manually grinded and polished. However, to obtain high-quality metallographic images, samples were electropolished with "Struers LectroPol-5" device with two component A2 electrolyte. Finally, samples were etched with 0.5% HF (hydrofluoric acid) reagent in a duration of 10 s at room temperature.

140

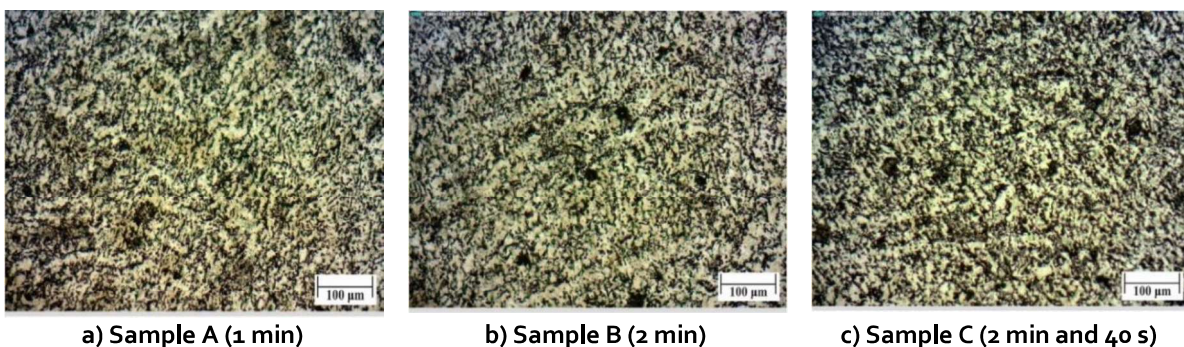
3. RESULTS ANALYSIS AND DISCUSSION

After casting, annealing, ECAP and semi-solid process, samples were cut on the metallographic cutter to avoid any heat influence. Fig. 3 a shows microstructure of the cast sample A. According to Fig. 3 a characteristic dendritic structure appeared in as cast sample and even after annealing dendritic microstructure was also preserved, Fig. 3 b. Eutectic was formed in the needle like structure. After the ECAP process, dendritic structure was highly deformed and a significant amount of the distortion energy was introduced into the samples, Fig. 3 c. These heavily distorted regions would create the location of the recrystallization nucleus. During samples reheating, recrystallization occurs in the solid state and new grains nucleate and grow with high angle grain boundaries. When the temperature rises to above solidus, the high-energy grain boundaries of these new grains are penetrated by liquid, leading to the fragmentation of original grains to small equiaxed grains. The presence of liquid causes grain growth and spheroidization of the newly formed grains [13].



a) Cast sample b) Annealed sample c) ECAPed sample
Fig. 3: Dendritic microstructure of as cast aluminum A380, cast and annealed sample and heavily deformed ECAPed sample

In order to determine optimal holding time at semi-solid temperature six samples were held at semi-solid temperature for a 1 min, 2 min, 2 min and 40 seconds, 5 min, 8 min and finally 12 min and 30 seconds which corresponds to the samples A, B, C, D, E, F and G, respectively, Fig. 4. Fig. 4 a shows sample A which was held only for one minute at semi-solid temperature and already characteristic ECAPed and heavily deformed microstructure disappeared. That indicated that the recrystallization process and partial remelting already take significant effect. However, despite the fact that dendrite structure had completely disappeared for sample A, still there is no clear sign of globular microstructure formation. The same observation can be given and for sample B and C with 2 min and 2 min and 40 seconds holding times, respectively, Fig. 4 b and c. However, with 5 min holding time (sample D), larger white areas started to form which should correspond to the solid α – aluminum matrix. Solid areas were surrounded with liquid of low melting phase, Fig. 4 d. Further holding time increment (8 min) results with the formation of the very small solid globular areas for sample E, Fig. 4 e. However, these formed globules are somewhat inhomogeneous and small which suggested that even longer holding times should be introduced for semi-solid homogeneous globular microstructure formation and larger globules. There is no sign of any dendritic microstructure. Desired globules size for thixoforming process are usually under 100 μm [22]. Therefore, finally holding time of 12 min and 40 s for sample F was used. An obtained microstructure with homogeneously dispersed globular structure was achieved, Fig. 4 f. Furthermore, according to the Gecu et al. [23], for aluminum alloy A380 low melting eutectic phase should be surrounding globular solid α – Al. Furthermore, for same alloy, after thixoforming process, usual formed phases are $\alpha(\text{Al})$, eutectic and polyhedral Si phases and additionally Fe-rich and Cu-rich phases [23]. Microstructure of the sample F with 500 x magnification is shown in figure 5.



a) Sample A (1 min) b) Sample B (2 min) c) Sample C (2 min and 40 s)

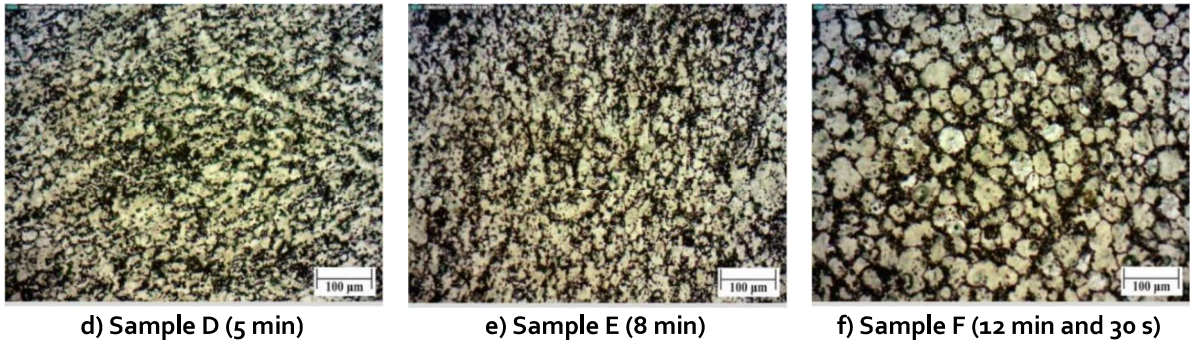


Fig. 4: Microstructure evolution of the aluminum alloy A380 during different holding times at semi-solid temperature

Morphology of the as cast needle like eutectic is changed into smaller size eutectic which surrounding globular solid $\alpha - \text{Al}$. There is no sign of any large-scale silicon or Fe- rich based particles. Therefore, sample F with obtained microstructure should be excellent feedstock material for thixoforming process for A380 aluminum alloy. For future work, obtained phases determination with scanning electron microscopy and energy dispersive x-ray spectroscopy should be done. Furthermore, influence of heat treatment on obtained samples microstructure should be investigated.



Fig. 5: Globular microstructure of the sample F with 500 x magnification

4. CONCLUSION

In the present work, equal channel angular pressing (ECAP) technique is used to produce thixo feedstock. By this thixo-route method, a high plastic strain is applied to a solid feedstock. By heating in semi-solid range, this deformed structure is changed to the structure consisted of globular solid particles surrounded with liquid. The homogenous distribution of $\alpha - \text{Al}$ and eutectic phase of A380 alloy is achieved by heating ECAPed samples at semisolid temperature for 12 min and 30 sec. Hence, the explained process is good enough for producing thixo feedstock.

REFERENCES

- [1] Wang, J., Phillion, A. B., & Lu, G., 2014, Development of a visco-plastic constitutive modeling for thixoforming of AA6061 in semi-solid state. *Journal of Alloys and Compounds*, 609, p. 290-295.
- [2] Salleh, M. S., Omar, M. Z., Syarif, J., & Mohammed, M. N., 2013, An overview of semisolid processing of aluminium alloys. *ISRN Materials Science*, 2013.

- [3] Fabrizi, A., Capuzzi, S., De Mori, A., & Timelli, G., 2018, Effect of T6 Heat Treatment on the Microstructure and Hardness of Secondary AlSiCu₃ (Fe) Alloys Produced by Semi-Solid SEED Process. *Metals*, 8(10), 750.
- [4] Mohammed, M. N., Omar, M. Z., Salleh, M. S., Alhawari, K. S., & Abdelgnei, M. A., 2014, An overview of semi-solid metal processing. *Australian Journal of Basic and Applied Sciences*, 8(19), p. 369-373.
- [5] Nafisi, S., & Ghomashchi, R., 2016, *Semi-solid processing of aluminum alloys*. Springer International Publishing.
- [6] Júnior, M. V., de Souza Neto, E. A., & Munoz-Rojas, P. A. (Eds.). (2011). *Advanced computational materials modeling: from classical to multi-scale techniques*. John Wiley & Sons, p. 205-256.
- [7] Fu, J. L., Jiang, H. J., & Wang, K. K., 2018, Influence of processing parameters on microstructural evolution and tensile properties for 7075 Al alloy prepared by an ECAP-based SIMA process. *Acta Metallurgica Sinica (English Letters)*, 31(4), p. 337-350.
- [8] Azimi-Yancheshmeh, D., & Aghaie-Khafri, M., 2010, Improvement of Mechanical Properties and Microstructure of Al-Fe-Si Alloy by ECAP Semisolided Method. In *ASME 2010 10th Biennial Conference on Engineering Systems Design and Analysis*, American Society of Mechanical Engineers p. 717-722.
- [9] Nedjad, S. H., Meidani, H., & Ahmadabadi, M. N., 2008, Effect of equal channel angular pressing on the microstructure of a semisolid aluminum alloy. *Materials Science and Engineering: A*, 475(1-2), p. 224-228.
- [10] Meidani, H., Hossein Nedjad, S., & Nili-Ahmadabadi, M., 2008, A novel process for fabrication of globular structure by equal channel angular pressing and isothermal treatment of semisolid metal. In *Solid State Phenomena Vol. 141*, Trans Tech Publications p. 445-450.
- [11] Moradi, M., Nili-Ahmadabadi, M., Poorganji, B., Heidarian, B., Parsa, M. H., & Furuvara, T., 2010, Recrystallization behavior of ECAPed A356 alloy at semi-solid reheating temperature. *Materials Science and Engineering: A*, 527(16-17), p. 4113-4121.
- [12] Ashouri, S., Nili-Ahmadabadi, M., Moradi, M., & Iranpour, M., 2008, Semi-solid microstructure evolution during reheating of aluminum A356 alloy deformed severely by ECAP. *Journal of Alloys and Compounds*, 466(1-2), p. 67-72.
- [13] Moradi, M., Nili-Ahmadabadi, M., Heidarian, B., & Habibi-Parsa, M., 2008, Study of ECAP processing routes on semi-solid microstructure evolution of A356 alloy. In *Solid State Phenomena, Vol. 141*, Trans Tech Publications, p. 397-402.
- [14] Campo, K. N., Proni, C. T. W., & Zoqui, E. J., 2013, Influence of the processing route on the microstructure of aluminum alloy A356 for thixoforming. *Materials Characterization*, 85, p.26-37.
- [15] Aghaie-Khafri, M., & Azimi-Yancheshme, D., 2012, The study of an Al-Fe-Si alloy after equal-channel angular pressing (ECAP) and subsequent semisolid heating. *Jom*, 64(5), p.585-592.
- [16] Azimi-Yancheshmeh, D., & Aghaie-Khafri, M., 2010, Improvement of Mechanical Properties and Microstructure of Al-Fe-Si Alloy by ECAP Semisolided Method. In *ASME 2010 10th Biennial Conference on Engineering Systems Design and Analysis*, American Society of Mechanical Engineers p. 717-722.

- [17] Meshkabadi, R., Faraji, G., Javdani, A., & Pouyafar, V., 2016, Combined effects of ECAP and subsequent heating parameters on semi-solid microstructure of 7075 aluminum alloy. *Transactions of Nonferrous Metals Society of China*, 26(12), p. 3091-3101.
- [18] Torres, L. V., Proni, C. T. W., & Zoqui, E. J., 2016, Comparison of morphological evolution of Al-7wt% Si-2.5 wt% Cu alloy produced by direct chill casting/electromagnetic stirring and ECAP. In *Solid State Phenomena*, Vol. 256, Trans Tech Publications, p. 17-24.
- [19] Torres, L. V., Torres, L. F., & Zoqui, E. J., 2016, Electromagnetic stirring versus ECAP: morphological comparison of Al-Si-Cu alloys to make the microstructural refinement for use in SSM processing. *Advances in Materials Science and Engineering*, 2016.
- [20] Iwahashi Y., Wang J., Horita Z., Nemoto M., Langdon T.G., 1996, Principle of equal-channel angular pressing for the processing of ultra-fine grained materials, *Scripta Materialia*, Vol. 35, p. 143-146.
- [21] Birol, Y., 2008, Semisolid processing of near-eutectic and hypereutectic Al-Si-Cu alloys. *Journal of Materials Science*, 43(10), p. 3577-3581.
- [22] Kirkwood, D. H., Suéry, M., Kapranos, P., Atkinson, H. V., & Young, K. P., 2010, *Semi-solid processing of alloys* (Vol. 124). Berlin: Springer.
- [23] Ridvan, G. E. C. U., Serhat, A. C. A. R., & Alptekin KISASOZ, K. A. G., 2018, Influence of T6 heat treatment on A356 and A380 aluminium alloys manufactured by thixoforging combined with low superheat casting. *Transactions of Nonferrous Metals Society of China*, 28(3), p. 385-392.