Arch. Metall. Mater., Vol. 61 (2016), No 3, p. 1663–1670

DOI: 10.1515/amm-2016-0269

A.WOŹNICKI*,#, D. LEŚNIAK*, G. WŁOCH*, P. PAŁKA*, B. LESZCZYŃSKA-MADEJ*, A. WOJTYNA*

THE EFFECT OF COOLING RATE AFTER HOMOGENIZATION ON THE MICROSTRUCTURE AND PROPERTIES OF 2017A ALLOY BILLETS FOR EXTRUSION WITH SOLUTION HEAT TREATMENT ON THE PRESS

The influence of cooling rate after homogenization on the 2017A alloy microstructure was analysed. The capability of the θ (Al_2Cu) particles, precipitated during various homogenization coolings, for rapid dissolution was estimated. For this purpose, the DSC test was used to determine the effect of the cooling rate after homogenization on the course of melting during a rapid heating. Moreover, the samples after solution heat treatment (with short time annealing) and ageing, were subjected to the microstructure investigations and the microhardness of grains interiors measurements. It was found that cooling after homogenization at 160 °C/h is sufficient for precipitation of fine θ phase particles, which dissolve during the subsequent rapid heating. The cooling at 40 °C/h, causes the precipitation of θ phase in the form of large particles, incapable of further fast dissolution.

Keywords: 2017A alloy, cooling rate after homogenization, θ (Al_2Cu) phase particles precipitation

1. Introduction

An extrusion with solution heat treatment on the runout table is a very advantageous technology of aluminium alloys profiles production. The elimination of a separate operation of extrudates solution heat treatment, leads to a reduction of manufacturing costs and improvement of productivity. Moreover, it contributes to enhancement of profiles properties due to retaining of the fibrous structure after the extrusion (the press-effect). It also reduces the risk of extruded product defects such as peripheral coarse grain layer (PCG) and shape distortions [1-4]. In industrial practice, this technology is commonly applied for 6xxx and some 7xxx alloys. The extrudates of the hard-deformable 2xxx alloys are generally solution heat treated in a separate operation, although some grades of this series are solution heat treated on the runout table [1]. It results from the metallurgical features of AlCuMg alloys. They are characterized by a narrow range between the solidus and solvus temperature [5]. Thereby, obtaining at the die exit the microstructure with fully dissolved hardening phases particles is hindered. These alloys are also quench sensitive [6,7] and during solution heat treatment high cooling rates must be applied, which is difficult when profiles are quenched on the runout table. Because of low exit speed, which is characteristic of these alloys [5], the time of uncontrolled cooling between the die exit and quenching installation (“water wave”) is elongated. This creates a risk of initiation of undesirable solid solution decomposition during cooling.

The difficulties described above are less pronounced, when the alloys with low content of main alloying additions (within the range defined by standards) are extruded. In the case of commonly used, high-strength 2024 and 2014 grades, application of low-alloyed compositions increases the solidus temperature. In consequence, as it was shown by numerical simulations and experiments, it is possible to preform extrusion process at higher exit temperature and speed, with lowered extrusion force, compared to high-alloyed billets [2, 3]. The reduced Cu and Mg content simultaneously decreases quench sensitivity of the alloys [8]. Thus, the extrusion with solution heat treatment on the press is facilitated. It is worth to mention here that the investigations, aimed at the application of the solution heat treatment directly from hot deformation temperature for the alloys with high additions concentrations are also performed [9, 10].

The effective solution heat treatment of the extruded profiles on the runout table is possible, only if the billets are previously suitably prepared for extrusion during the homogenization process. A particular attention should be paid to the billets cooling from homogenization temperature. In the case of standard technology, with separate heating of extruded products to the solution heat treatment temperature, there is no need for controlling the cooling rate. When the homogenization process is performed in batch-type furnaces, the billets located in the centre of the batch are often cooled significantly slower than the billets at the batch edge [11]. The slow billets cooling from the homogenization temperature results in precipitation of alloying elements in the form of large particles [12-14].

If the billets are intended for the extrusion with solution heat treatment on the press, such a slow cooling from homogenization temperature cannot be applied. The coarsened particles, present in the billets microstructure after this manner of homogenization cooling, do not fully dissolve during subsequent billets preheating and extrusion process. The concentration of alloying elements in the solid solution after press quenching is then reduced and strength properties of the material after final ageing are inadequate. This problem

* AGH – UNIVERSITY OF SCIENCE AND TECHNOLOGY, FACULTY OF NON-FERROUS METALS, AL. A. MICKIEWICZA 30, 30-059 KRAKÓW, POLAND

Correspondence author: woznicki@agh.edu.pl

is described in the literature in details for 6xxx alloys [15- 18], but it is also reported for AlCuMg(Si) alloy [19]. Therefore, in the case of billets intended for the extrusion with solution heat treatment on the runout table, cooling from homogenization temperature must be controlled and realized in repeatable manner. The cooling rate should be high enough to cause precipitation of the fine hardening phase particles, capable for further fast dissolution. It is necessary for obtaining the required mechanical properties of the profiles extruded with solution heat treatment and aged. The presented paper describes the influence of cooling rate from homogenization temperature, on the structure of 2017A alloy billets intended for extrusion with solution heat treatment on the runout table.

2. Experimental work

The billets with chemical composition presented in TABLE 1 were DC cast in semi-industrial conditions.

TABLE 1

Chemical composition of the investigated 2017A alloy, mass %

Element	Si	Fe	Cu	Mn	Mg	Zr+Ti	Al
Concentration	0.47	0.1	3.54	0.71	0.61	0.1	bal.

The material in as-cast state was subjected to microstructure observations, XRD and DSC analyses. A Hitachi SU-70 scanning electron microscope equipped with EDS detector was used for microstructure observations. The samples for SEM investigations were prepared using standard metallography methods, including mechanical grinding and polishing with diamond suspensions and colloidal silica. EDS analyses were applied to determine the chemical composition of the observed particles and to measure Cu and Mg content in the grains interiors after homogenization. A Rikagu diffractometer, with $Cu_{K\alpha}$ radiation was used to execute XRD analysis of the powdered specimens. DSC test was performed using a Mettler Toledo 821^e heat flux type calorimeter. The disc shaped samples were inserted in ceramic pans into the cell with the temperature of 470 °C and heated 20 °C/min to the temperature of 700 °C. The solidus temperature and heat of the incipient melting reactions were determined.

The samples, mechanically sectioned from billets, were subjected to homogenization in laboratory conditions. The influence of cooling rate from the homogenization temperature on the billets microstructure was investigated. The material was heated to the temperature of 495 °C during 6h, soaked for 12 h and cooled to the room temperature in three ways: water quenched, cooled at 160 °C, and cooled at 40 °C/h. This cooling rates are the average values, estimated on the basis of samples temperature measurements recorded during the cooling cycles (Fig. 1), in the temperature range of 495-200 °C.

The samples after homogenization were subjected to SEM observations, XRD and DSC analyses, as described above.

In order to evaluate the θ phase particles dissolution ability during rapid heating, which occurs in the extrusion process, the following experiment was realized: the homogenized samples were annealed at the temperature of 500 °C for 5 min., quenched in water and naturally aged for 30 days. Afterwards, Vickers microhardness of grains interiors of the age-hardened samples was measured using a Shimadzu MHV hardness tester (nominal test force: 0.9807 N, dwell time 10 s.) and SEM/EDS microstructure investigations were performed. It should be noted that this experiment is only an approximation of the industrial conditions. During the experiment, similarly as in the extrusion process, the material was heated within a few minutes to the temperature of the alloy at a die exit (500 °C). In the laboratory test, however, the deformation did not take place and the temperature course during billet preheating (dependent on the heater type, extrusion speed etc.) was not simulated.

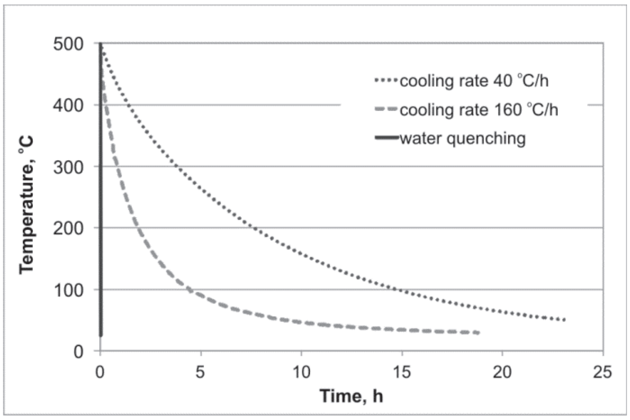
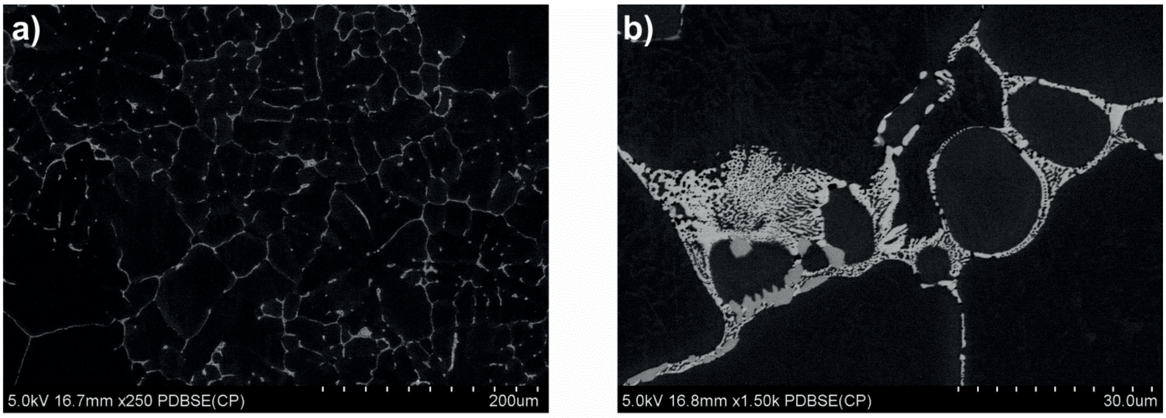
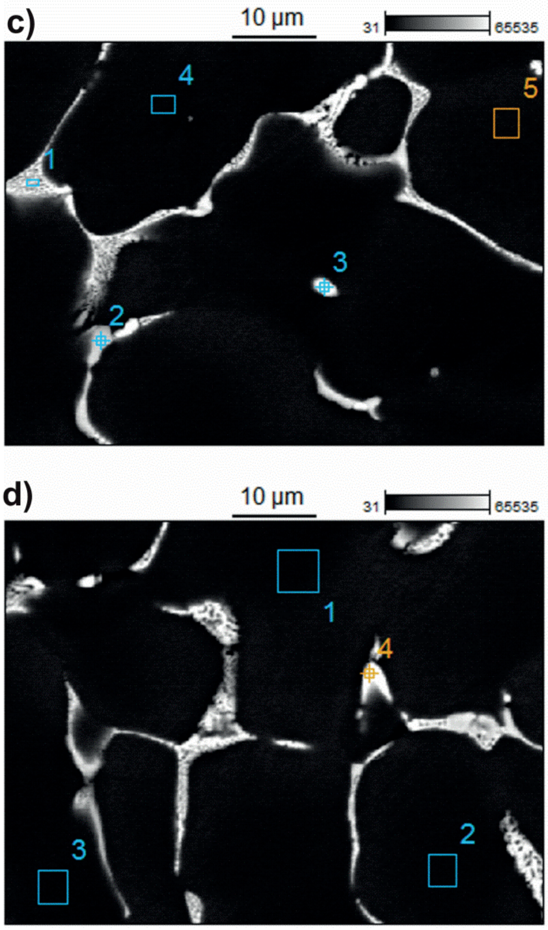


Fig. 1. Cooling curves recorded during homogenization experiments

3. Results and discussion

The alloy in as-cast state is characterized by dendritic microstructure (Fig. 2). In the interdendritic areas, significant amount of the eutectic containing Al, Cu, Mg and Si is present. Based on the XRD pattern (Fig. 3) it can be stated that the θ (Al_2Cu) phase is one of the components of this eutectic. SEM/EDS examination revealed that outside the eutectic regions, two types of particles are also present. The first one, with Al, Cu, Mn, Fe, Si in chemical composition (Fig 2c point 2), is probably $Al_{15}Si_2(CuFeMn)_3$ [14, 19, 20]. The second type, containing and Al, Mg, Si and Cu (Fig. 2c point 1 and 2d point 4) is probably the Q phase, in the literature designated also as h-AlCuMgSi, W or λ. The chemical composition of this phase is described as $Al_5Cu_2Mg_8Si_6$, $Al_4CuMg_5Si_4$, $Al_4Cu_2Mg_8Si_7$ or $Al_3Cu_2Mg_9Si_7$ [21]. The particles described above were identified on the basis of the EDS analyses results (Fig. 2 c, d) and literature data, as the peaks corresponding to these phases were not found on XRD pattern recorded for the alloy in as-cast state (Fig. 3). As can be seen, considerable fraction of alloying additions is located in interdendritic and intergranular areas. In consequence, the concentrations of Cu and Mg in the dendrites interiors are small, the mean values from ten measurements in randomly selected areas are only 1.6 and 0.15 mass % respectively (Fig. 4). On the DSC curve for the alloy in as-cast state a large peak with the onset temperature of 515 °C is observed (arrowed in the Fig. 5, described in TABLE 2). This is the result of melting of the eutectic mixtures present in the alloy's microstructure.





Point	Mg	Al	Si	Mn	Fe	Cu
1	3.2	66.8	2.2	0.2	0.1	27.5
2	0.2	71.2	5.5	9.4	5.7	8.0
3	0.9	81.6	1.1	1.5	1.1	13.9
4	0.2	97.8	-	0.6	0.1	1.4
5	0.3	96.9	-	0.7	-	2.1

Point	Mg	Al	Si	Mn	Fe	Cu
1	0.2	97.6	-	0.6	-	1.6
2	0.2	97.3	-	0.7	0.1	1.7
3	0.2	98.0	-	0.6	-	1.2
4	1.4	63.4	1.8	0.1	-	33.3

EDS analysis results in mass%

Fig. 2. Typical microstructure of investigated billets in as-cast state

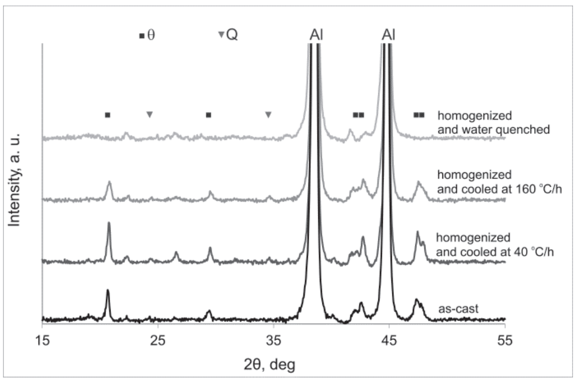


Fig. 3. The XRD patterns of the investigated alloy in as cast state and after homogenization

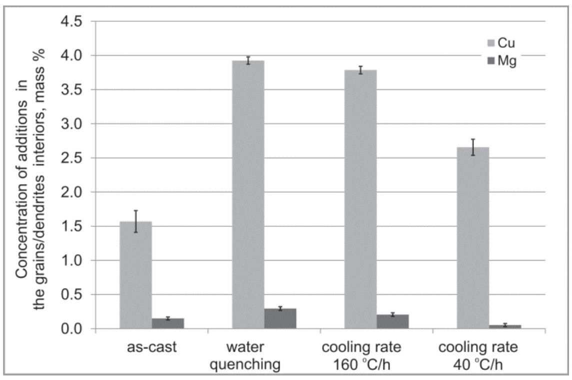


Fig. 4. Concentration of Cu and Mg in the dendrites/grains interiors in as-cast state and after homogenization (mean values from ten measurements in randomly selected areas)

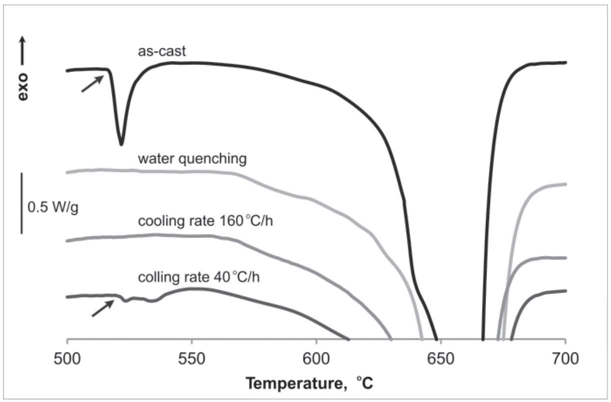


Fig. 5. DSC curves for the alloy in as cast state and after homogenization

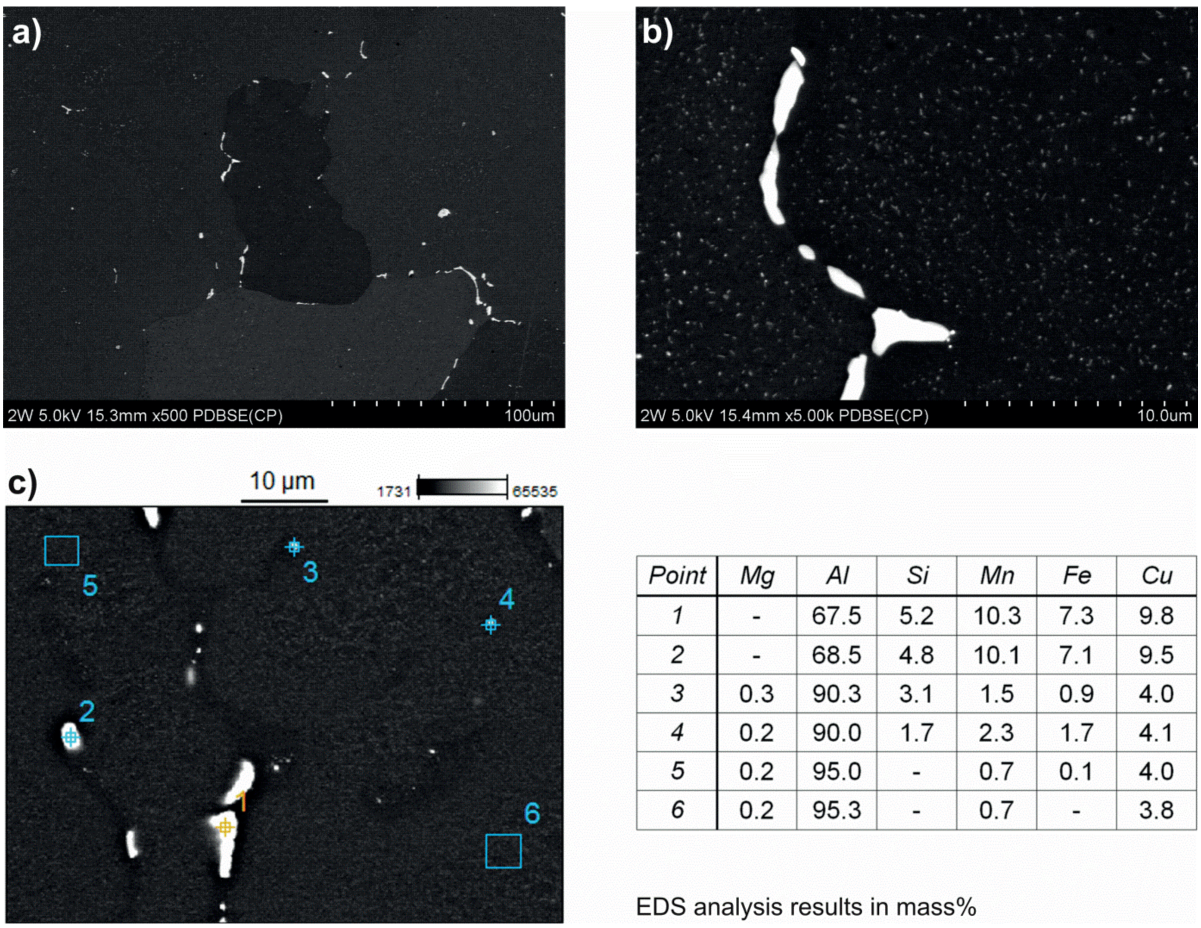
TABLE 2

DSC analysis results

Homogenization cooling conditions	Solidus temperature, °C	Incipient melting heat, J/g
as-cast	515	11.9
water quenching	565	-
cooling rate 160 °C/h	563	-
cooling rate 40 °C/h	518	3.0

In the microstructure of the homogenized and water quenched alloy (Fig. 6), the eutectic is not observed. On the DSC curve for the alloy in that state, the incipient melting peak is not found (Fig. 5) and solidus temperature is 565 °C. It indicates that the presence of the eutectic in as-cast state was an effect of the non-equilibrium solidification conditions and it fully dissolved during the subsequent homogenization. After homogenization and water quenching, on the grains boundaries, particles of the unsoluble $Al_{15}Si_2(CuFeMn)_3$ phase are found. In the grain interiors, fine dispersoids, precipitated during the homogenization annealing, are present. The Cu and Mg concentrations in the grains interiors are 3.8 and 0.3 mass % respectively, they are two times higher than in as-cast state. It is worth to notice that the differences between the results obtained in individual microanalysis areas are small. These results allow to conclude that the homogenization annealing conditions for the investigated alloy were selected properly and the microsegregation observed in the as-cast state was removed.

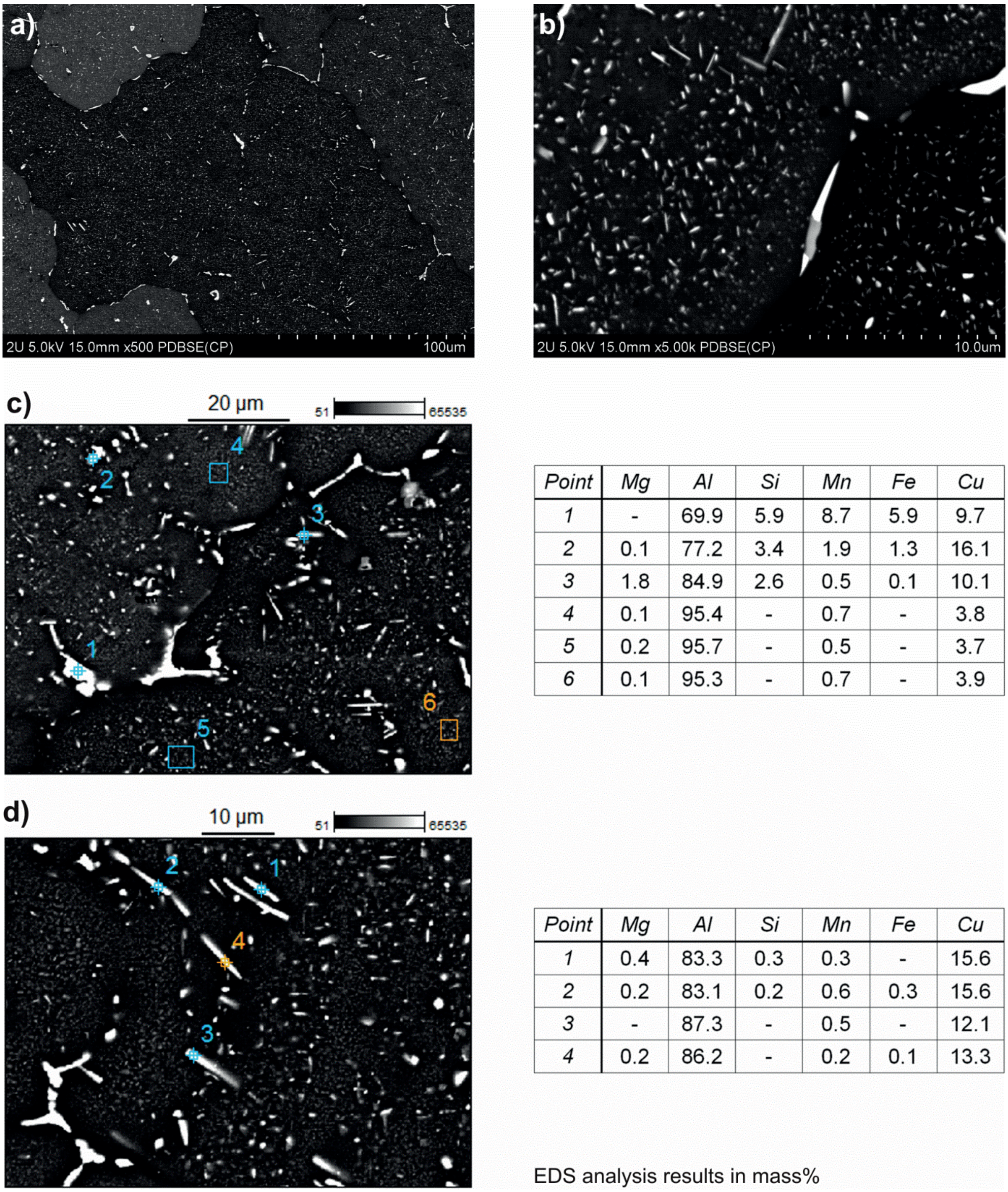
Cooling from homogenization temperature at 160 °C/h, causes precipitation of θ phase mainly in the form of fine, elongated particles numerously observed in the grains interiors (Fig. 7). Their small size justifies the expectation that they will easily dissolve during the subsequent reheating and extrusion processes. However, on the grains boundaries some coarser (a few μm in size) particles are also found. During the cooling from homogenization temperature at 40 °C/h, the θ phase precipitates as large particles, with about 10 μm in size (Fig. 8), frequently plate-shaped. The $Al_{15}Si_2(CuFeMn)_3$ and Q phases particles are also found in the microstructure of the investigated alloy after homogenization cooling at 160 and 40 °C/h.



Point	Mg	Al	Si	Mn	Fe	Cu
1	-	67.5	5.2	10.3	7.3	9.8
2	-	68.5	4.8	10.1	7.1	9.5
3	0.3	90.3	3.1	1.5	0.9	4.0
4	0.2	90.0	1.7	2.3	1.7	4.1
5	0.2	95.0	-	0.7	0.1	4.0
6	0.2	95.3	-	0.7	-	3.8

EDS analysis results in mass%

Fig. 6. The microstructure of the investigated alloy after homogenization and water quenching



Point	Mg	Al	Si	Mn	Fe	Cu
1	-	69.9	5.9	8.7	5.9	9.7
2	0.1	77.2	3.4	1.9	1.3	16.1
3	1.8	84.9	2.6	0.5	0.1	10.1
4	0.1	95.4	-	0.7	-	3.8
5	0.2	95.7	-	0.5	-	3.7
6	0.1	95.3	-	0.7	-	3.9

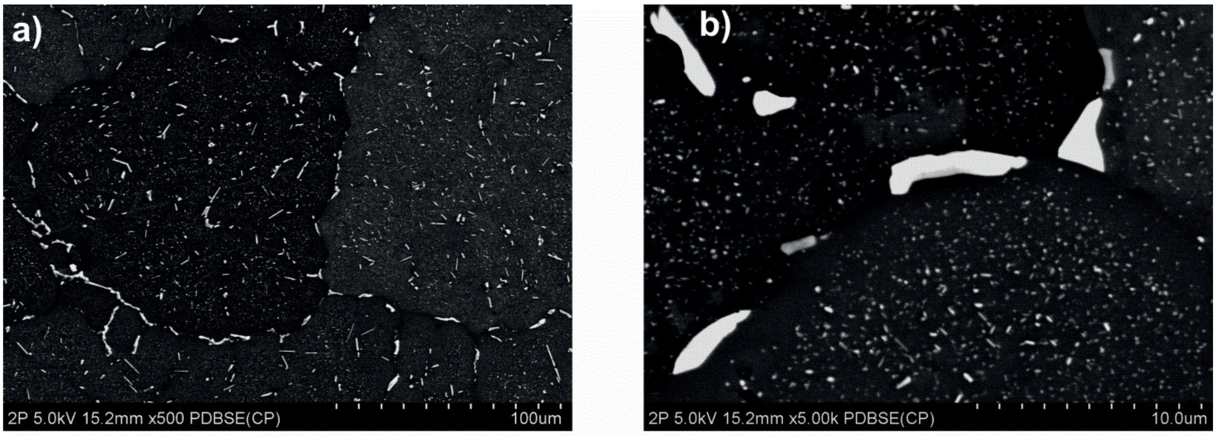
Point	Mg	Al	Si	Mn	Fe	Cu
1	0.4	83.3	0.3	0.3	-	15.6
2	0.2	83.1	0.2	0.6	0.3	15.6
3	-	87.3	-	0.5	-	12.1
4	0.2	86.2	-	0.2	0.1	13.3

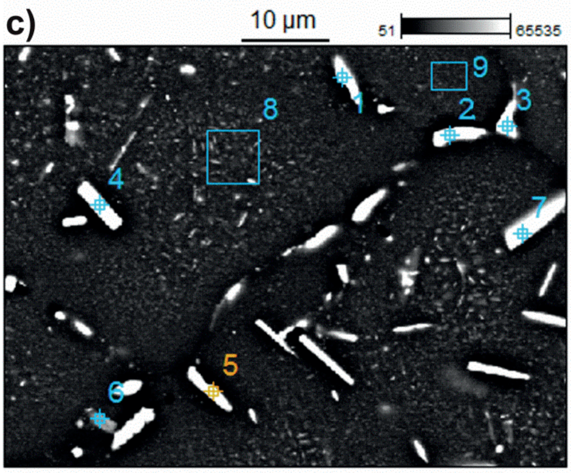
EDS analysis results in mass%

Fig. 7. The microstructure of the investigated alloy after homogenization and cooling at 160 °C/h

The precipitation of θ and Q phases particles during cooling from the homogenization temperature at 160 and 40 °C/h is confirmed by XRD analysis results (Fig. 3). In should be noted that the peaks resulting from the presence of the θ phase are significantly greater after slower cooling than after faster one, which indicates a larger fraction of this phase in the alloys microstructure in the former case. The peaks corresponding to the Q phase are similar for both cooling rates.

The cooling rate from homogenization temperature influences the concentration of Cu and Mg in the grains interiors (Fig. 4). After water quenching and cooling at 160 °C/h, Cu concentrations are very similar (3.8 and 3.7 mass % respectively). It indicates that a predominant amount of Cu, present in solid solution at the end of soaking stage, during cooling at 160 °C/h precipitated as fine particles in the grains interiors. After cooling at 40 °C/h this concentration is lower (2.7 mass %), because a considerable amount of Cu precipitated in the form of coarse particles on the grains boundaries. The tendency to lower concentration in the grains interiors with decreasing cooling rate was also observed for Mg. On the basis of SEM and XRD investigations it may be assumed that this results from the precipitation of the Q phase during cooling.





Point	Mg	Al	Si	Mn	Fe	Cu
1	-	70.7	0.1	0.5	-	28.7
2	-	65.8	-	0.3	-	33.9
3	-	65.6	-	0.2	-	34.2
4	-	78.6	0.5	1.1	0.8	19.0
5	-	93.3	-	0.5	-	6.2
6	5.7	77.3	10.5	0.4	-	6.1
7	0.1	76.6	0.1	0.7	0.1	22.5
8	0.1	96.2	-	0.8	-	3.0
9	-	96.5	-	0.8	-	2.6

EDS analysis results in mass%

Fig. 8. The microstructure of the investigated alloy after homogenization and cooling at 40 °C/h

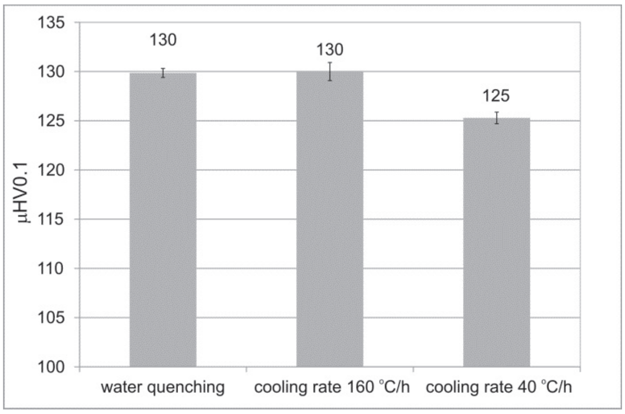
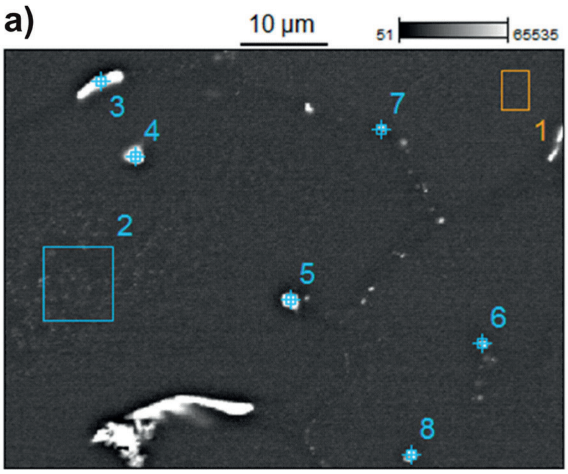


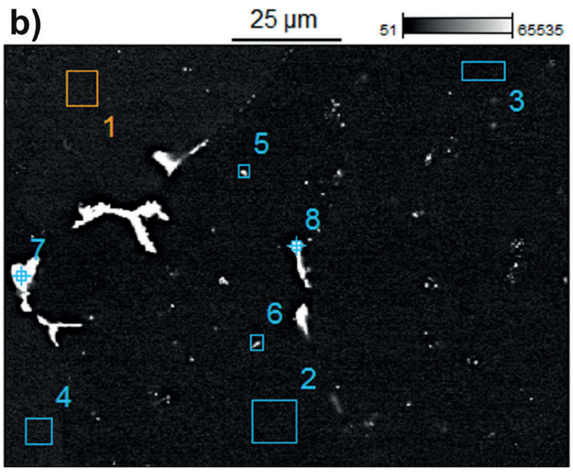
Fig. 9. Microhardness of the grains interiors after solution heat treatment (500 °C/5 min.) water quenching and natural ageing (30 days)

The capacity of θ phase particles, precipitated during homogenization cooling, for rapid dissolution was estimated by microhardness of the grains interiors, of solution heat treated (with short time annealing) water quenched and naturally aged samples. Final microhardness obtained for samples water quenched after homogenization, and samples cooled from homogenization temperature at 160 °C/h is the same. The microhardness of the samples cooled from homogenization temperature at 40 °C/h is lower by about 5HV (Fig. 9). It results from the difference in dissolution of θ phase particles during solution heat treatment (Fig. 10). In the microstructure of the material previously water quenched from homogenization temperature, no θ phase particles are found. In the case of homogenization cooling at 160 °C/h, only small undissolved θ phase particles can be found and for the cooling at 40 °C/h, θ phase particles with a few µm in size are visible.

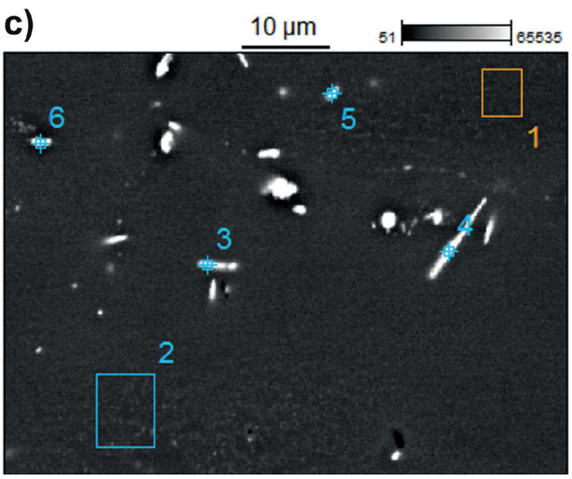
This explanation is confirmed by the results of DSC test with rapid heating, performed on the samples after different homogenization cooling variants (Fig. 5, TABLE 2). The curves obtained after homogenization with water quenching and cooling at 160 °C/h, are very similar, without incipient melting, whereas after cooling at 40 °C/h an incipient melting peak with the onset at the temperature of 518 °C occurs (arrowed in the Fig. 5). By comparison to the literature data [15, 22] it can be explained as follows: the large θ phase particles, present in the billets microstructure after slow homogenization cooling, do not fully dissolve during the rapid heating and cause local enrichment of the surrounding matrix with Cu. In consequence, unequilibrium melting reaction takes place. On the other hand, the fine θ phase particles, which precipitated during homogenization cooling at 160 °C, dissolve completely during DSC run, thus no incipient melting is observed. On the basis of the results presented above it can be stated that cooling from homogenization temperature at 160 °C/h is sufficient and at 40 °C/h it is too slow for 2017A alloy billets intended for extrusion with solution heat treatment at the press output.



Point	Mg	Al	Si	Mn	Fe	Cu
1	0.3	95.0	-	0.7	-	4.0
2	0.3	95.2	-	0.7	-	3.7
3	0.3	74.1	4.1	9.1	6.4	6.1
4	0.2	75.5	4.5	7.9	5.3	6.6
5	0.3	80.9	3.6	5.0	3.5	6.9
6	0.3	83.5	3.2	5.2	3.5	4.4
7	0.3	91.5	1.2	2.1	0.8	4.2
8	0.3	83.7	3.9	5.4	3.2	3.6



Point	Mg	Al	Si	Mn	Fe	Cu
1	0.2	95.5	-	0.7	-	3.5
2	0.3	95.5	-	0.6	0.1	3.6
3	0.3	95.3	-	0.7	-	3.8
4	0.2	95.5	-	0.7	-	3.7
5	0.3	93.5	0.1	0.8	0.1	5.2
6	0.3	94.3	-	0.6	0.1	4.8
7	-	72.7	5.3	8.0	5.2	8.7
8	0.1	68.4	5.1	10.4	6.7	9.2



Point	Mg	Al	Si	Mn	Fe	Cu
1	0.3	96.0	-	0.6	-	3.1
2	0.3	95.5	-	0.5	-	3.6
3	0.4	87.7	-	0.5	-	11.4
4	0.4	84.3	0.1	0.6	0.1	14.5
5	0.3	84.0	0.1	0.5	0.1	15.1
6	0.4	89.4	0.1	0.6	0.1	9.4

EDS analysis results in mass%

Fig. 10. The microstructure of the investigated alloy after homogenization with water quenching (a), cooling at 160 °C/h (b), cooling at 40 °C/h (c), subjected to subsequent solution heat treatment (500 °C/5 min.) water quenching and natural ageing (30 days)

It should be pointed here that suitable billet preparation is only one of conditions, necessary for achieving expected mechanical properties of the extruded products, after solution heat treatment on the press and ageing. As is was mentioned earlier, during the extrusion process, the special attention must be paid on the selection of temperature-speed parameters. In order to enable full dissolution of θ phase particles, the materials temperature at the die exit must exceed the alloy's solvus temperature and simultaneously, acceptable extrudates surface quality and extrusion process efficiency must be ensured. Moreover, high cooling rate is necessary for the effective quenching of the extruded product on the runout table.

4. Conclusions

On the basis of the investigation results presented above, the following conclusions can be drawn:

Homogenization at the temperature of 495 °C for 12h was found to be appropriate for the investigated alloy. It results in dissolution of unequilibrium eutectic observed in the as-cast state, the increase of solidus temperature from 515 to 565 °C and enrichment of grains interiors in the main alloying additions.

Cooling rate after homogenization at 160 °C/h is sufficient for precipitation of fine θ phase particles, whereas the cooling at 40 °C/h causes precipitation of considerable fraction of θ phase in the form of large particles on the grains boundaries.

The fine θ phase particles, precipitated during the cooling after homogenization at 160 °C/h, dissolve during the subsequent rapid heating. This is concluded on the basis of the high grains interiors microhardness of the alloy cooled in this manner, subjected to solution heat treatment with short time annealing (500°C/5 min) water quenching and ageing, as well as on the basis of the microstructure investigations. It can be expected that in the case of application of this cooling rate in industrial conditions, the particles dissolution will also take place during the subsequent billets reheating and extrusion.

The large θ phase particles, present in the microstructure of the investigated alloy after homogenization cooling at 40 °C/h, do not fully dissolve during the subsequent rapid heating. It is confirmed by the microstructure investigations, lower grains interiors microhardness after the solution heat treatment, water quenching and ageing, as well as DSC test results, showing incipient melting in the case of alloy subjected to this kind of homogenization cooling.

Acknowledgements

The financial support from National Centre of Research and Development under grant No: PBS2/B5/26/2013 entitled New material and technological solutions for extrusion process of high-strength thin-walled hollow shapes from aluminium alloys is kindly acknowledged.

REFERENCES

[1] J. Senderski, L. Pierewicz, B. Płonka, Rudy Metale **47** (6), 284-288 (2002).
[2] D. Leśniak, A. Woźnicki, Arch. Metall. Mater. **57** (1), 19 – 31 (2012).
[3] D. Leśniak, J. Zasadziński, M. Galanty, A. Woźnicki, A. Rękas, Proc. of 10th International Aluminum Extrusion Technology Seminar & Exposition (2012).
[4] D. Leśniak, Arch. Metall. Mater. **54** (4), 1135 – 1145 (2009).
[5] B. Dixon, Proc. of 7th International Aluminum Extrusion Technology Seminar, **281-294** (2000).
[6] G. Beck, J. C. Chevrier, Int. J. Heat Mass Tran. **14** (10), 1731-1745, (1971).
[7] J.E. Hatch (ed.), Aluminum Properties and Physical Metallurgy, American Society for Metals 1984.
[8] I. I. Nowikow, Diagrammy Izotermicieskogo raspada rastwora w aliuminijewych spławach, Metallurgija, Moskwa 1973.
[9] A. Woźnicka, Określenie warunków technologicznych intensywnego przesycenia stopu AlCu4MgSi z temperatur przeróbki plastycznej na gorąco, PhD thesis, AGH University of Science and Technology, Kraków (2012).
[10] J. Richert, A. Woźnicka, Rudy Metale **58** (2), 90-95 (2013).
[11] M. Bronicki, A. Woźnicki, Rudy Metale **52** (12), 907-909 (2007).
[12] A. Łatkowski, J. Gryziecki, M. Bronicki, Aluminium **63** (2), 193-195 (1987).
[13] G.R. Ebrahimi, A. Zarei-Hanzaki, M. Haghshenas, H. Arabshahi, J. Mater. Process. Tech. **206**, 25–29 (2008).
[14] M. Gonçalves, M.G. Martins, W.Z. Misiołek, W.H. VanGeertruyden, Mater. Sci. Forum **396-402**, 393-398 (2002).
[15] O. Reiso, J.E. Hafsås, O. Sjothun, U. Tundal, Proc. of 6th International Aluminum Extrusion Technology Seminar vol.1, 1 – 10 (1996).
[16] J. Richert, M .Richert, J. Woźnicka, Z. Stec, Proc. of 7th International Aluminum Extrusion Technology Seminar, vol. 2, 105 – 113 (2000)
[17] E.B. Bjørnbakk, J.A. Sæter, O. Reiso, U. Tundal, Mater. Sci. Forum **396-402**, 405-410 (2002).
[18] A. Woźnicki, J. Richert, M. Richert, J. Woźnicka, Rudy Metale **48** (10-11), 468-473 (2003).
[19] G. Szilagyi, M. Goncalves, Mater. Forum **28**, 558-563 (2004).
[20] L.F. Mondolfo, Aluminum alloys. Structure and properties, Butterworths 1979.
[21] D.J. Chakrabarti, D.E. Laughlin, Prog. Mater. Sci. **49**, 389–410 (2004).
[22] M. Lefstad, O. Reiso, Proc. of 6th International Aluminum Extrusion Technology Seminar vol.**1**, 11 – 21 (1996).