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ASSESSMENT OF THE EFFECT OF TEMPERATURE AND ANNEALING TIME HOMOGENIZATION AlCu4MgMn ALLOYS IN TERMS OF MICROSTRUCTURE IMAGE ANALYSIS METHODS AND EDX

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ОЦЕНКА ВЛИЯНИЯ ТЕМПЕРАТУРЫ И ВРЕМЕНИ ГОМОНИЗАЦИИ ОТЖИГА СПЛАВОВ AlCu4MgMn С ТОЧКИ ЗРЕНИЯ МЕТОДА АНАЛИЗА ИЗОБРАЖЕНИЯ МИКРОСТРУКТУР И EDX АНАЛИЗА

Homogenization is defined as a method of heat treatment, which consists of holding time at high temperature near the liquidus (approx. 0.7 to 0.8 the melting temperature) to eliminate chemical inhomogeneity diffusion processes. Cause of segregation is selective crystal solidification in the gradual change in composition of the solid phase. Melt began to appear after certain of the hypothermia during cooling, and the growth of germs in accordance with the general laws of crystallization. Each rigid layer has a different chemical composition. The first part of the solid phase ingredient low concentration of the element last, on the contrary, very high. Susceptibility to crystal alloy segregation is greater, the greater the temperature interval solidification of alloys and the horizontal distance between the liquidus and solidus lines. Crystal segregation will also increase with increasing content of alloying elements, which in these experimental alloys occurs as containing 6-9% alloying elements. Crystal segregation can be removed by diffusion, for which it is necessary to create conditions by homogenization annealing.
*Keywords: homogenization annealing, AlCu4MgMn alloy, crystal segregation, energy dispersive X-ray analysis (*EDX*), image analysis*

Introduction

The process of homogenizing annealing temperature and time affects the process of homogenization, diffusion coefficients for the elements in the aluminum matrix and the size of the diffusion pathways. The temperature and duration of the process of homogenization is chosen so that there was a dissolution equilibrium intermetallic phases formed in the process of crystallization and subsequent diffusion of the elements into a solid solution α. Likewise for non-equilibrium eutectic resulting in higher crystallization rate than the equilibrium that dissolve and diffuse into relevant components α matrix. Homogenizing annealing temperature is determined in the range from 0.90 to 0.95 Tt. Homogenization annealing can take place at lower temperatures, from 0.8 to 0.9 Tt. Homogenizing annealing temperature reduction can significantly prolong the process of homogenizing annealing up to several tens of hours.

Alloy - Al-Cu-Mg, especially AlCu4Mg duralumin, and AlCu4Mg AlCu4MgMn, reaching considerable strength after you-hardened by heat treatment (Rm up to 530 MPa). The maximum solubility of copper in the aluminum solid solution under equilibrium conditions is 5.7 wt. % Cu eutectic reaction at a temperature of 548.2°C. Alloys AlCuMg achieve considerable strength after curin g, their advantage is the natural aging process. Other elements, alloying in industrial alloys of the Al-Cu-Mg is Mn, which increases strength. AlCu4Mg in the alloy occurs primarily binary eutectic α + $CuAl_2$ a small amount of ternary eutectic α + + $CuAl_2$ $Cu_2Mg_2Al_5$, further Mg_2Si, $FeAl_3$, AlFeMnSi, AlCuFeMn etc. The aim of this paper is to optimize the process of homogenization in terms of its length and the homogenizing annealing temperature alloys AlCu4MgMn.

Experiment

To prepare castings investigated material was used raw material supplied directly by the manufacturer. Casting alloys studied were prepared according to the chemical composition based on EN AW 2024 - EN AW AlCu4Mg1. Melting of the material was held in a furnace at 730°C, the oven temperature was scanned digital thermometer with a precision of ± 2°C. The melt was treated in the melting process and refining salt melt surface was withdrawn smear. Prepared material was gravity casting process into metal molds preheated to a temperature of 220°C. Castings were in the shape of a conical cylinder with dimensions of 40/50 x 100 mm. Chemical composition of experimental alloys prepared in wt. % Is given in Tab. 1.

Table 1

The chemical composition of AlCu4MgMn alloy

AlCu4MgMn [hm. %]	Cu	Mn	Mg	Fe	Si	Al
	3,83	0,31	1,78	0,07	0,06	93,93

Alloy thus prepared was divided into two sets of samples were subjected to homogenization annealing. Homogenization annealing furnace was held in LAC, the temperature was scanned digital thermometer with accuracy of ± 2°C. The first set of samples was homogenized at various temperature regimes in the temperature range from 450 to 570°C in 20°C intervals (450, 470, 490, 510, 530, 550, 570°C) at a constant time of homogenization 8 hours. A second set of samples were prepared at a constant temperature of homogenization annealing T = 490°C, but with different time of the thermal process. Homogenizing annealing time was 2 to 24 hours (2, 4, 6, 8, 10, 12, 16, 20, 24 hours). After homogenization, the samples were about the size of 3x5 / 3 cm in the air cooled. Microstructure of the prepared samples before and after homogenizing annealing was analyzed on thin sections prepared metallographic LEXT confocal laser microscope at a magnification of 100x (Fig. 1 - Fig.4).

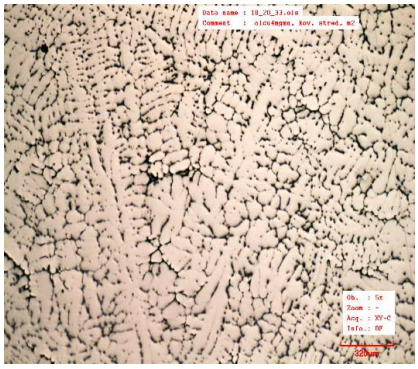

Fig. 1. AlCu4MgMn alloy microstructure before homogenization, enl.:100x

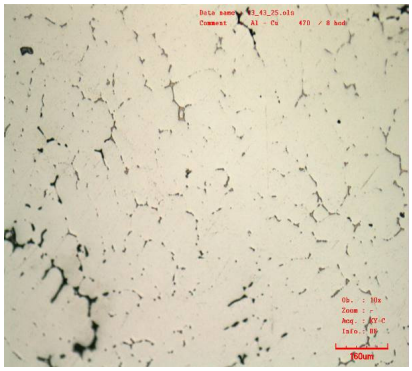

Fig. 2. AlCu4MgMn alloy microstructure at constant annealing lasting 8 hours. 470°C, enl.:100x

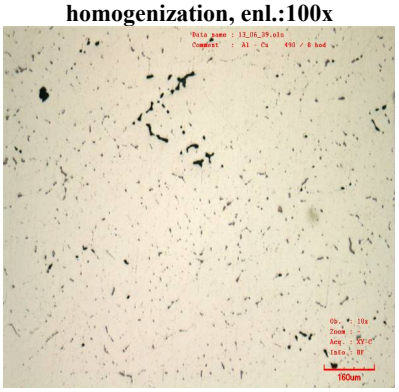

Fig. 3. AlCu4MgMn alloy microstructure at constant annealing lasting 8 hours. 490°C, enl.:100x

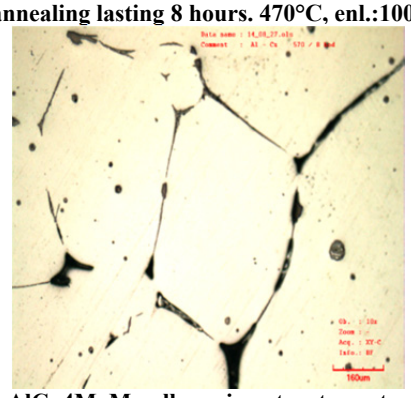

Fig. 4. AlCu4MgMn alloy microstructure at constant annealing lasting 8 hours. 570°C, enl.:100x

A second set of samples were prepared at a constant temperature homogenization annealing T = 490°C, but at different times of the heating process (Fig. 5 - Fig. 7). Homogenizing annealing time was chosen in the range of 2 to 24 hours [t = 2, 4, 6, 8, 10, 12, 16, 20, 24 hours].

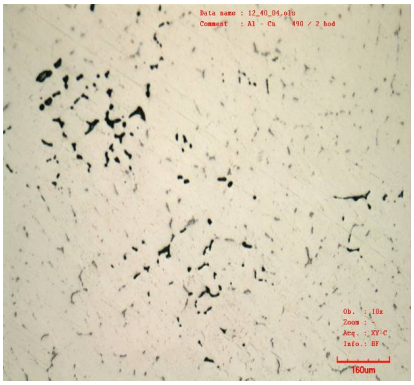

Fig.5. AlCu4MgMn alloy microstructure at constant annealing temperature of 490°C, 2 hours, enl.:100x

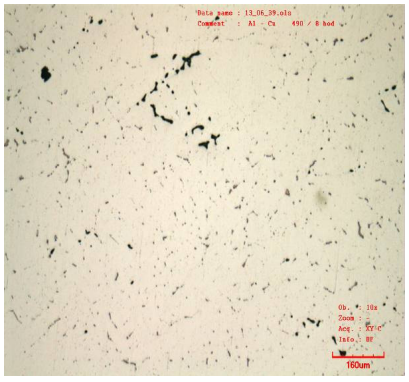

Fig. 6. AlCu4MgMn alloy microstructure at constant annealing temperature of 490°C, 8 hours, enl.:100x

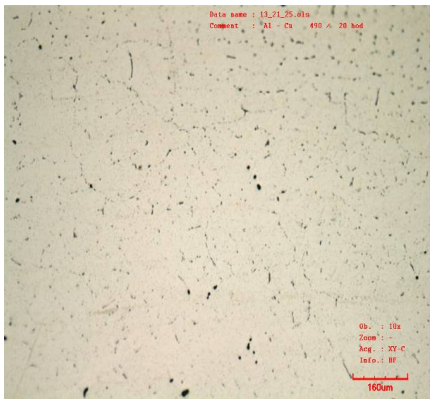

Fig. 7. AlCu4MgMn alloy microstructure at constant annealing temperature of 490°C, 20 hours, enl.:100x

Optical microscope in conjunction with PC software Buehler Omnimet Image Analysis system allows 5.40-nil at the samples to evaluate quality alloy AlCu4MgMn homogenizing annealing using image analysis. This analysis enabled by statistically evaluated microscopic image size heterogeneities (particles) and the percentage occurring in the studied alloy. Microscope image acquisition was performed metallographic optical microscope on thin sections at a magnification of 100x. Image analysis of allow use to determine the influence of homogenizing annealing time on the quality of homogenizing annealing. To evaluate the percentage of the phase represented by the selected samples investigated alloy AlCu4MgMn prior to homogenizing annealing and after homogenizing annealing at 490°C for 4, 8 and 12 hours. All samples were performed imaging analysis of five different sites examined. The results were the above-mentioned program mathematically processed.

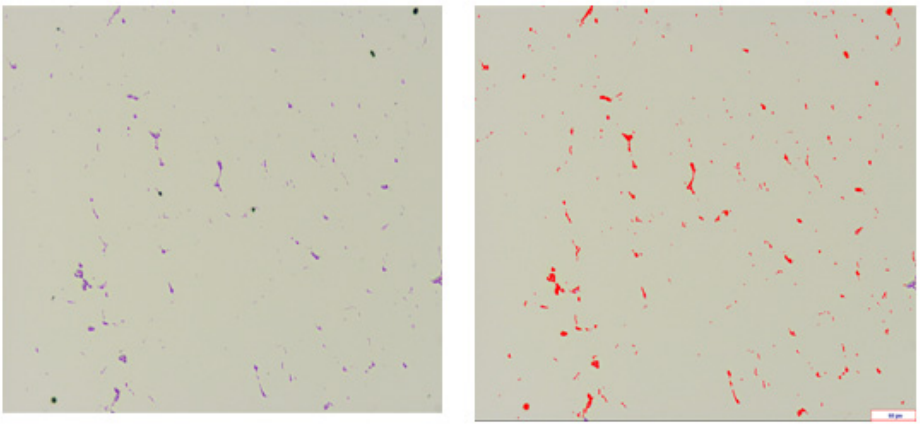

Fig. 8. Image analysis AlCu4MgMn alloy homogenization annealing 490°C, 8 hours, enl.:100x

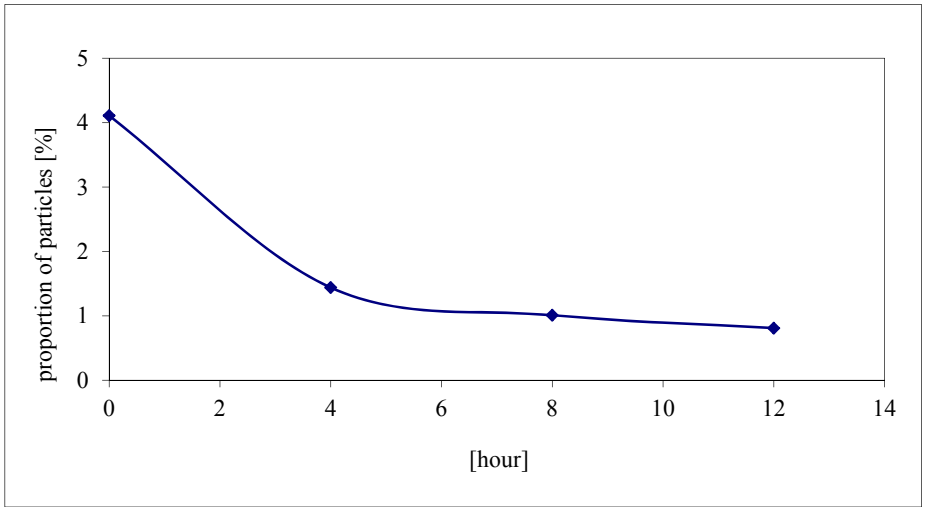

Fig. 9. Dependence of the percentage of particles in the during homogenization annealing AlCu4MgMn alloy

Table 2

The percentage of particles AlCu4MgMn alloy

Pattern	Min. [%]	Max. [%]	Average [%]	Standard deviation [%]
Before homogenization	3,74	4,66	4,11	0,36
4 hours/490°C	1,03	1,85	1,44	0,29
8 hours/490°C	0,92	1,14	1,01	0,79
12 hours/490°C	0,57	1,11	0,81	0,21

Table 3

Analysis of particle size AlCu4MgMn alloy

Pattern	Max. [μm]	Min.[μm]	Average [μm]	Total number of particles	Standard deviation [%]
Before homogenization	64,66	0,46	9,32	373	9,23
12 hours/490°C	23,81	0,93	4,23	253	3,46

For the evaluation of homogenizing annealing of the alloy AlCu4MgMn was performed before and after homogenization EDX analysis. Energy dispersive spectroscopy allowed to analyze chemical composition of the phases present, and eutectic α - solid solution. EDX analysis AlCu4MgMn alloys was performed on the sample prior to homogenization annealing after heat processing and homogenizing annealing at 490°C for 8 hours. Analyzed sites are indicated by symbols P1 to P8.

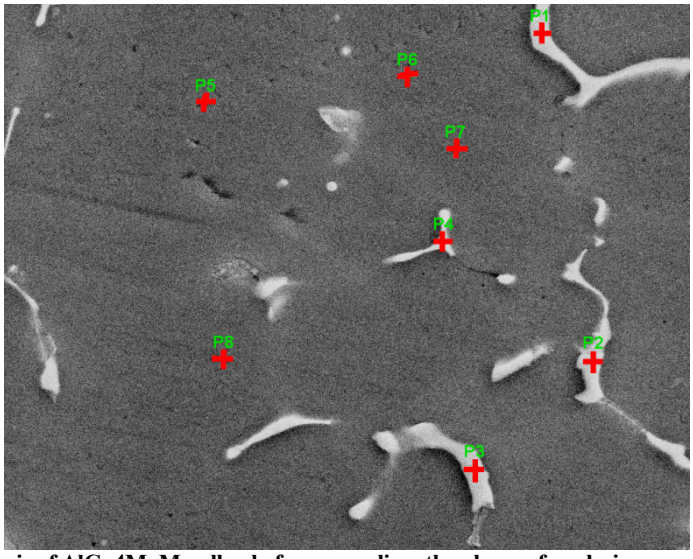

Fig. 10. Pattern EDX analysis of AlCu4MgMn alloy before annealing, the places of analysis are marked as P1 -P8, enl. 1000x

Table 4

Pattern EDX analysis values of AlCu4MgMn alloy's marked places Fig.10 P1 - P8

Analyzed elements	P1	P2	P3	P4	P5	P6	P7	P8
Cu [hm%]	18,61	27,00	11,60	22,80	1,12	1,14	1,57	3,31
Mn [hm%]	0,06	0,03	0,10	0,10	0,09	0,13	0,10	0,13
Mg [hm%]	2,25	1,72	1,74	1,55	2,33	1,79	2,85	3,79
Al [hm%]	79,08	71,25	86,56	75,56	96,47	96,94	95,48	92,77

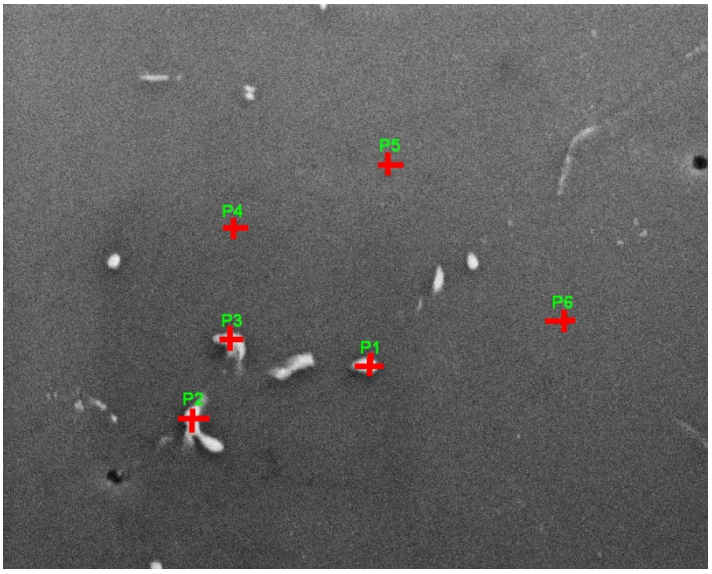

Fig. 11. Pattern EDX analysis of AlCu4MgMn alloy after annealing with the temperature of T = 490°C during 8 hours, the places of analysis are marked as P1 - P6, enl. 1000x

Table 5

Pattern EDX analysis values of AlCu4MgMn alloy's marked places Fig. 11, P1 - P6

Analyzed elements	P1	P2	P3	P4	P5	P6
Cu [hm%]	16,98	16,28	4,09	2,97	4,10	6,18
Mg [hm%]	2,16	2,15	1,98	1,21	1,68	2,88
Mn [hm%]	0,05	0,10	0,20	0,77	1,59	2,89
Al [hm%]	80,82	81,48	93,72	95,06	92,63	88,05

To evaluate the quality homogenizing annealing in addition to point EDX analysis of EDX analysis of surface used to distribute typified aluminum parent metal, and of the alloying elements in the selected area of the sample alloy AlCu4MgMn homogenized at 490°C for 8 hours. The images captured EDX analysis shows the different areas of concentration of the analyzed elements. Higher concentrations of the analyzed element, is represented by a light gray to white.

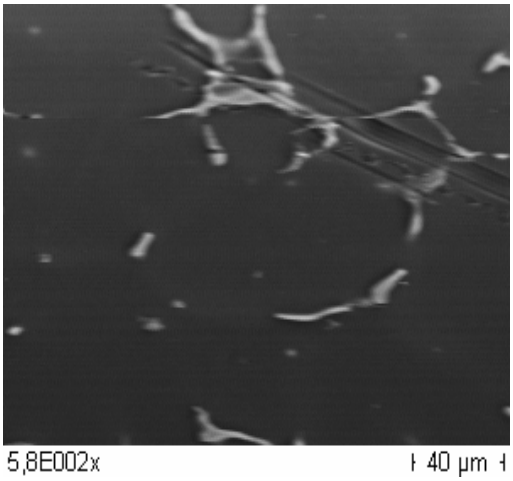

Fig. 12. Analyzed area EDX analysis AlCu4MgMn alloys, cast condition, scanning electron microscope

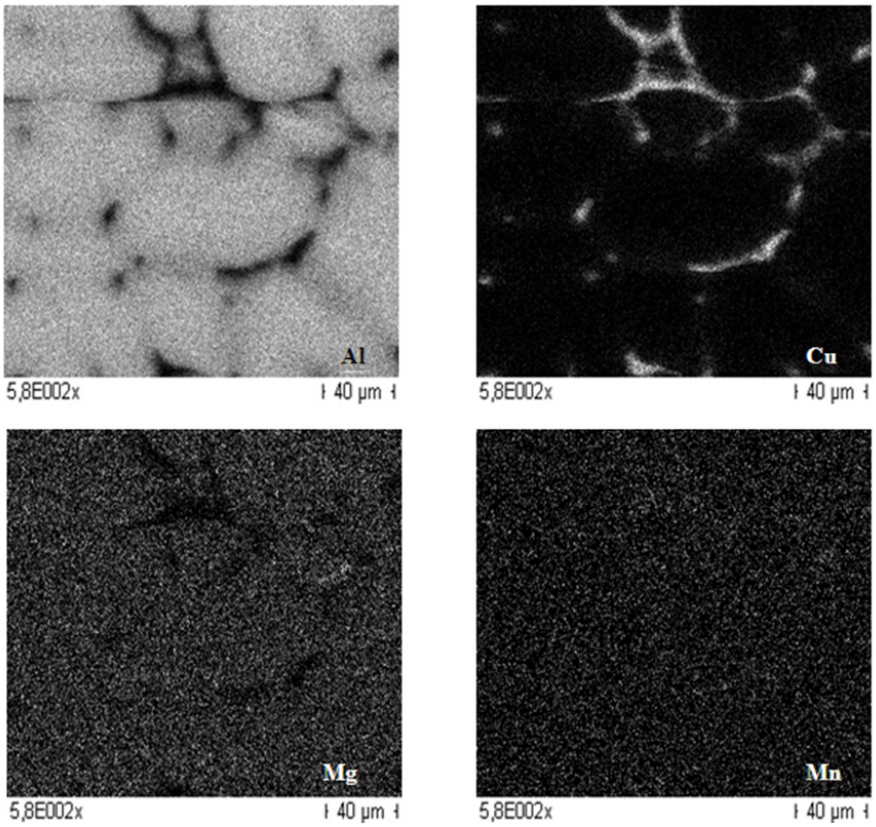

Fig. 13. EDX analysis AlCu4MgMn alloy (Fig. 12), distribution of aluminum, copper, magnesium and manganese, scanning electron microscope

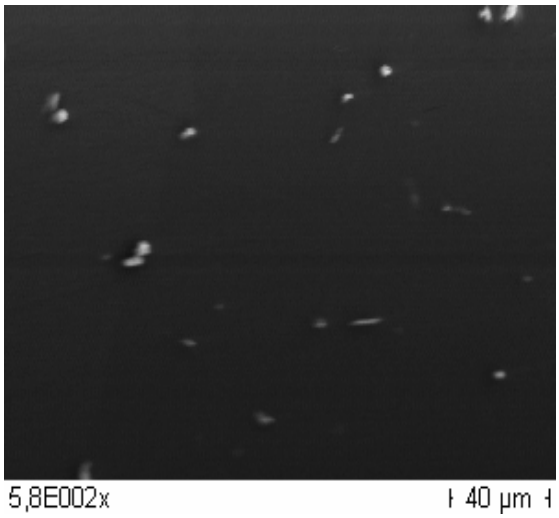

Fig. 14. Analyzed area EDX analysis AlCu4MgMn, homogenized T = 490°C/8 h, scanning electron microscope

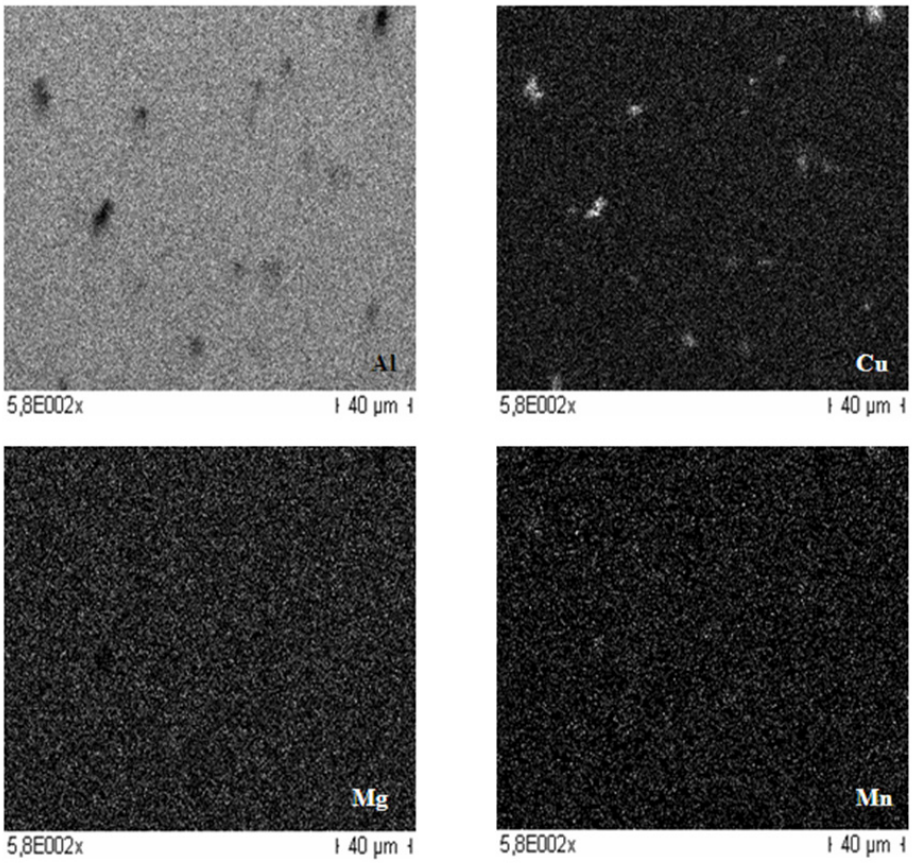

Fig. 15. EDX analysis AlCu4MgMn alloy (Fig. 14), distribution of aluminum, copper, magnesium and manganese, scanning electron microscope

Conclusion

Optimal conditions homogenizing annealing AlCu4MgMn alloys were evaluated in terms of microstructure image analysis and help energy dispersive spectroscopy. The selected parameters were homogenizing annealing time homogenizing annealing at a constant temperature, annealing temperature and homogenizing annealing at a constant length of this process. In terms of microstructure optimal homogenization annealing temperature at constant time of 8 hours alloys AlCu4MgMn provide in the range of 490 to 510°C. At higher temperature annealing leads to a homogenization of the melted eutectic grain boundary and globular bodies melted eutectic grains inside Influence of homogenizing annealing on microstructure of the investigated alloy was observed after two hours of this process. Optimum annealing time homogenization AlCu4MgMn alloy can be determined in the range of 8 - 10 hours at a temperature of homogenization annealing 490°C. Quantitative evaluation method of image analysis to help determine the effect of time allowed homogenizing annealing on the percentage occurring particles and their size at AlCu4MgMn alloy cast in a metal mold. The graphical dependence of the average percentage of particles at the time of

homogenization at a temperature of 490°C is evident that, with increasing time of homogenization annealing decreases the percentage of from 4.11% to 0.81%. The most intense period of homogenizing annealing effect is observed as early as 4 hour soak at temperature 490 ° C homogenization annealing, the percentage of particles occurring decreased from 4.11% to 1.85%. Over the next few hours (8 hours) decreased the percentage of particles present up to 1.14%. Size of particles present in the alloy due to AlCu4MgMn homogenizing annealing at 490°C for 12 hours decreased from 9.32 micron to 4.23 micron. The results of the EDX point analysis AlCu4MgMn alloys show that the process of homogenizing annealing was present to dissolve the intermetallic phase and a eutectic naked on the copper content and subsequent diffusion of the elements into a solid solution α. From the data obtained by EDX suggests heterogeneities present undissolved noted an increased copper content (16.63 wt.%), From which one can infer the presence of insoluble phase AlCuFeMn, AlSiMnCuFe and Al12Mn2Cu or undissolved residual phases and eutectic $CuAl_2$, $CuAl_2$ + α, α + $CuAl_2$ + Mg_2Si, which melts at 517°C.

Анотація: *Гомогенізація визначається як метод термічної обробки, який складається з тимчасової витримки при високій температурі поблизу ліквідуса (приблизно від 0,7 до 0,8 температури плавлення), щоб усунути хімічну неоднорідність дифузії. Температура та тривалість процесу гомогенізації обрана так, щоб вийшло розчинення рівноваги інтерметалевих фаз, що утворюються в процесі кристалізації і подальшого розповсюдження елементів у твердих α рішеннях.*
Ключові слова: гомогенізація, AlCu4MgMn сплав, EDX аналіз, дифузія

Аннотация: *Гомогенизация определяется как метод термической обработки, который состоит из временной выдержки при высокой температуре вблизи ликвидуса (примерно от 0,7 до 0,8 температуры плавления), чтобы устранить химическую неоднородность диффузии. Температура и длительности процесса гомогенизации выбрана так, чтобы получилось растворение равновесия интерметаллических фаз, образующихся в процессе кристаллизации и последующего распространения элементов в твердых α решениях.*
Ключевые слова: гомогенизация, AlCu4MgMn сплав, EDX анализ, диффузия

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