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Assessment of the Effect of Temperature and Annealing Time on Homogenization of AlCu4MgMn Alloys

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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 of 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 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 is characterized with low concentration of the element, the last one, on the contrary, is characterized with very high concentration. Susceptibility to crystal alloy segregation is greater, when the temperature interval solidification of alloys and the horizontal distance between the liquidus and solidus lines are larger. Crystal segregation will also increase with increasing content of alloying elements, which occurs in these experimental alloys as containing 6–9% alloying elements. Crystal segregation can be removed by diffusion, for which it is necessary to create conditions by homogenization annealing.

Гомогенізація визначається як метод оброблення, який полягає у витримці при високій температурі поблизу ліквідуса (від 0,7 до 0,8 температури топлення) з метою усунення процесів дифузії, пов'язаних з хімічною неоднорідністю. Причиною сеґреґації є вибіркове твердіння кристалу при поступовому змінюванні складу твердої фази. Розтоп починає з'являтися після деякого переохолодження під час остигання та росту зародків відповідно до загальних законів кристалізації. Кожний жорсткий шар має різний хімічний склад. Перша частина твердої фази мала низьку концентрацію леґувальних елементів, остання — навпаки, високу. Сприйнятливість до сеґреґації кристалічного стопу тим більша, чим більший температурний інтервал твердіння стопів та відстань по горизонталі між лініями ліквідуса і солідуса. Сеґреґація кристалу також зростає зі збільшенням кількості леґувальних елементів, яка в цих експериментальних стопах складала 6–9%. Сеґреґація кристалу може бути усунена дифузією, для чого необхідно створити умови шляхом гомогенізувального відпалу сталі.

Гомогенизация определяется как метод термообработки, который состоит в выдержке при высокой температуре вблизи ликвидуса (от 0,7 до 0,8 температуры плавления) с целью устранения процессов диффузии, связанных с химической неоднородностью. Причиной сегрегации является избирательное отвердевание кристалла при постепенном изменении состава твёрдой фазы. Расплав начинает появляться после некоторого переохлаждения во время остывания и роста зародышей в соответствии с общими законами кристаллизации. Каждый жёсткий слой имеет различный химический состав. Первая часть твёрдой фазы имела низкую концентрацию легирующих элементов, последняя — наоборот, высокую. Восприимчивость к сегрегации кристаллического сплава тем больше, чем больше температурный интервал отвердевания сплавов и расстояние по горизонтали между линиями ликвидуса и солидуса. Сегрегация кристалла также возрастает с увеличением содержания легирующих элементов, которое в этих экспериментальных сплавах составляло 6–9%. Сегрегация кристалла может быть устранена диффузией, для чего необходимо создать условия путём гомогенизирующего отжига стали.

Key words: crystal segregation, AlCu4MgMn alloy, homogenization annealing, EDX analysis, image analysis.

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1. INTRODUCTION

The homogenizing annealing temperature and time affect the process of homogenization, diffusion coefficients for the elements in the aluminium matrix, and the size of the diffusion pathways. The temperature and duration of the process of homogenization is chosen to provide dissolution equilibrium of 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 one, it dissolves and diffuses into relevant components of α matrix. Homogenizing annealing temperature is determined in the range from $0.90T_m$ to $0.95T_m$. Homogenization annealing can take place at lower temperatures, from $0.8T_m$ to $0.9T_m$. Homogenizing annealing temperature reduction can significantly prolong the process of homogenizing annealing up to several tens of hours.

Alloys Al–Cu–Mg, especially, AlCu4Mg duraluminium, and AlCu4Mg, AlCu4MgMn, reach considerable strength after self-hardening by heat treatment (R_m up to 530 MPa). The maximum solubility of copper in the aluminium solid solution under equilibrium conditions is 5.7 wt.% Cu in eutectic reaction at the temperature of 548.2°C. AlCuMg alloys achieve considerable strength after curing, their advantage is the natural aging process. Other alloying element in industrial alloys of the Al–Cu–Mg is Mn, which increases strength. AlCu4Mg in the alloy occurs primarily as binary eutectic $\alpha + CuAl_2$ with a small amount of ternary eutectic $\alpha + CuAl_2$ $Cu_2Mg_2Al_5$, further Mg_2Si, $FeAl_3$, AlFeMnSi, AlCuFeMn, *etc*. The aim of this paper is to optimize the process of homogenization in terms of its duration and the homogenizing annealing temperature of AlCu4MgMn alloys.

2. EXPERIMENT

To prepare castings, raw material supplied directly by the manufacturer is used as investigated material. Casting alloys studied are prepared according to the chemical composition based on EN AW 2024–EN AW AlCu4Mg1. Melting of the material is carried out in a furnace at 730°C, the oven temperature is scanned by digital thermometer with a precision of ±2°C. The melt is treated in the melting process and refining salt melt surface is withdrawn. Prepared material is subjected to gravity casting process into metal moulds preheated to a temperature of 220°C. Castings are in the shape of a conical cylinder with dimensions of 40/50 × 100 mm. Chemical composition of experimental alloys prepared is presented in Table 1 in wt.%.

Prepared alloy is divided into two sets of samples, which are subjected to homogenization annealing. Homogenization annealing furnace is held in LAC, the temperature is scanned by digital thermometer with accuracy of ±2°C. The first set of samples is homogenized at various temperature regimes in the temperature range from 450 to 570°C in 20°C intervals (450, 470, 490, 510, 530, 550, and 570°C) at a constant time of homogenization 8 hours. A second set of samples is prepared at a constant temperature of homogenization annealing $T = 490$°C, but with different time of the thermal process. Homogenizing annealing time is 2 to 24 hours (2, 4, 6, 8, 10, 12, 16, 20, and 24 hours). After homogenization, the samples had the size of 3×5/3 cm after the air-cooling. Microstructure of the prepared samples before and after homogenizing annealing is analyzed on thin slices using metallographic LEXT confocal laser microscope at magnification of ×100 (Figs 1–4).

A second set of samples is prepared at a constant temperature ho-

TABLE 1. The chemical composition of AlCu4MgMn alloy.

	Cu	Mn	Mg	Fe	Si	Al
AlCu4MgMn (wt.%)	3.83	0.31	1.78	0.07	0.06	93.93

Fig. 1. AlCu4MgMn alloy microstructure before homogenization; enl: ×100.

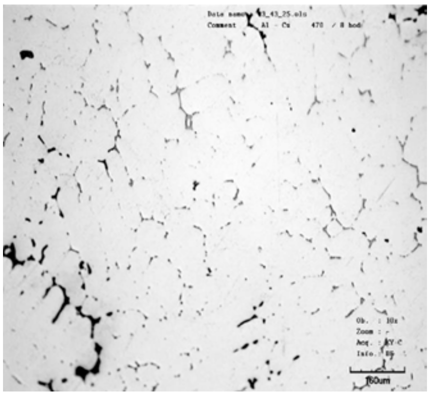

Fig. 2. AlCu4MgMn alloy microstructure at constant annealing lasting 8 hours; 470°C, enl: ×100.

mogenization annealing $T = 490$°C, but at different times of the heating process (Figs 5–7). Homogenizing annealing time is chosen in the range of 2 to 24 hours ($t = 2, 4, 6, 8, 10, 12, 16, 20, 24$ hours).

Optical microscope in conjunction with PC software Buehler Omnimet Image Analysis system allows evaluating the quality of alloy AlCu4MgMn homogenizing annealing using image analysis of the samples (5.40-nil). This analysis allows determining the heterogeneities (particles) and the percentage occurring in the studied alloy by statistically evaluated microscopic image size. Microscope image acquisition is performed using metallographic optical microscope on thin slices at

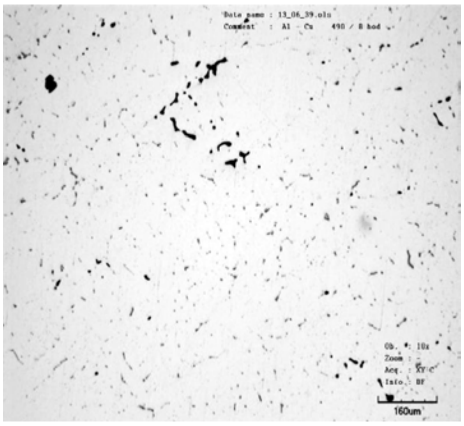

Fig. 3. AlCu4MgMn alloy microstructure at constant annealing lasting 8 hours; 490°C, enl: ×100.

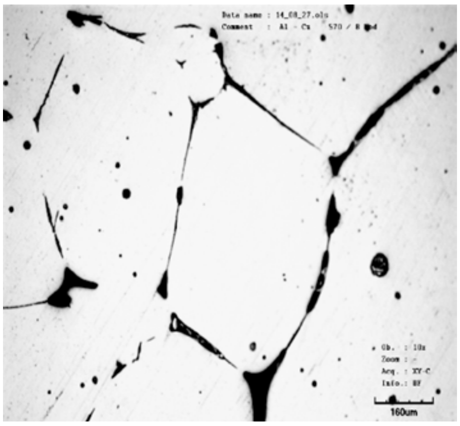

Fig. 4. AlCu4MgMn alloy microstructure at constant annealing lasting 8 hours; 570°C, enl: ×100.

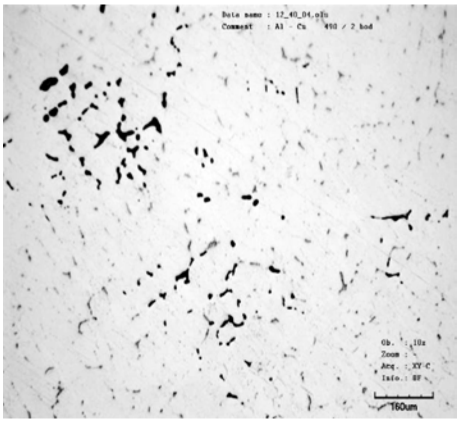

Fig. 5. AlCu4MgMn alloy microstructure at constant annealing temperature of 490°C; 2 hours, enl: ×100.

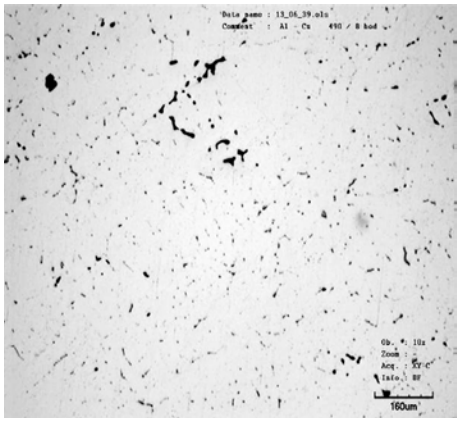

Fig. 6. AlCu4MgMn alloy microstructure at constant annealing temperature of 490°C; 8 hours, enl: ×100.

the magnification of ×100. Image analysis of alloy is used to determine the influence of the annealing time on the quality of homogenizing annealing.

To evaluate the percentage of the phase represented by the selected samples, AlCu4MgMn alloy is investigated before homogenizing annealing and after homogenizing annealing at 490°C for 4, 8 and 12 hours. All samples are subjected to imaging analysis of five different sites examined. The results are mathematically processed by the abovementioned program (see Figs 8, 9 and Tables 2, 3).

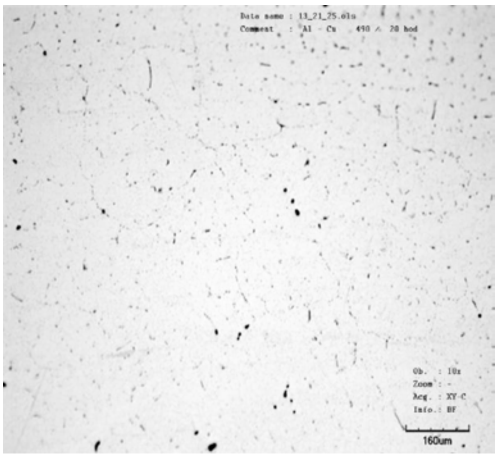

Fig. 7. AlCu4MgMn alloy microstructure at constant annealing temperature of 490°C; 20 hours, enl: ×100.

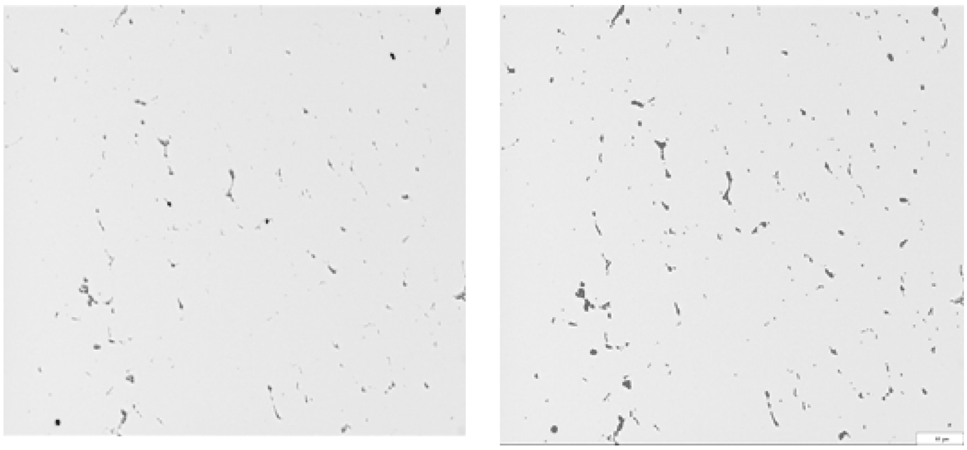

Fig. 8. Image analysis of AlCu4MgMn alloy homogenization annealing at 490°C; 8 hours, enl: ×100.

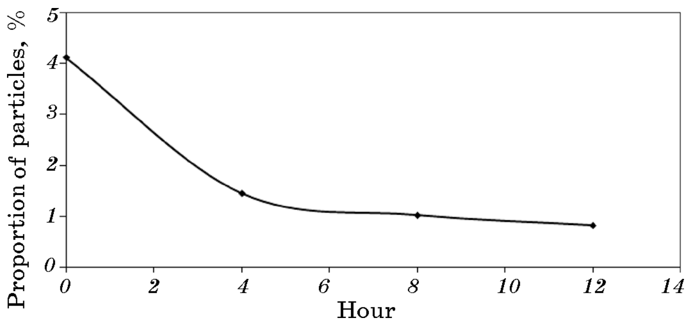

Fig. 9. Dependence of the percentage of particles during homogenization annealing in AlCu4MgMn alloy.

For the evaluation of homogenizing annealing of the AlCu4MgMn alloy, EDX analysis was performed before and after homogenization (see Fig. 10, Table 4 and Fig. 11, Table 5).

TABLE 2. The percentage of particles in 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 in 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

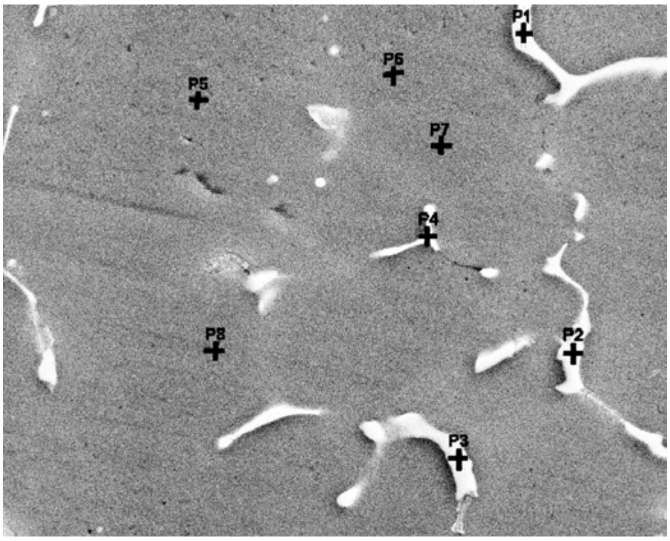

Fig. 10. Pattern of EDX analysis of AlCu4MgMn alloy before annealing; the places of analysis are marked as P1–P8; enl: ×1000.

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

TABLE 4. Pattern EDX analysis values of AlCu4MgMn alloy marked places in Fig. 10 as P1–P8.

Analyzed elements	P1	P2	P3	P4	P5	P6	P7	P8
Cu (wt.%)	18.61	27.00	11.60	22.80	1.12	1.14	1.57	3.31
Mn (wt.%)	0.06	0.03	0.10	0.10	0.09	0.13	0.10	0.13
Mg (wt.%)	2.25	1.72	1.74	1.55	2.33	1.79	2.85	3.79
Al (wt.%)	79.08	71.25	86.56	75.56	96.47	96.94	95.48	92.77

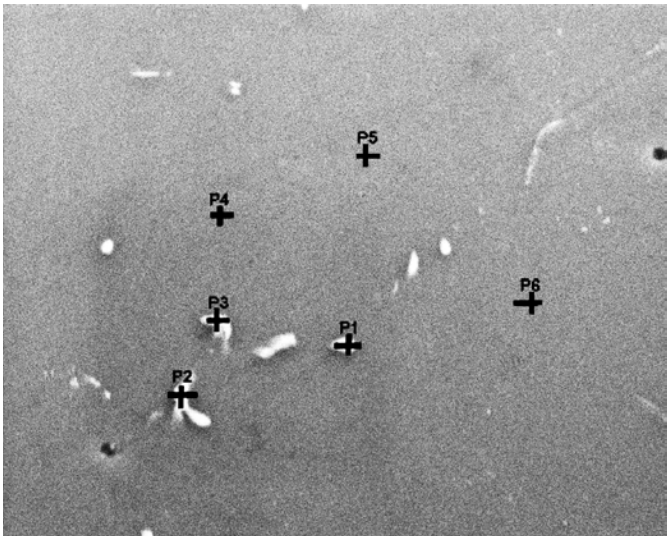

Fig. 11. Pattern of EDX analysis of AlCu4MgMn alloy after annealing with the temperature of $T = 490°C$ during 8 hours; the places of analysis are marked as P1–P6; enl: ×1000.

TABLE 5. Pattern of EDX analysis values of AlCu4MgMn alloy places marked in Fig. 11 as P1–P6.

Analyzed elements	P1	P2	P3	P4	P5	P6
Cu (wt.%)	16.98	16.28	4.09	2.97	4.10	6.18
Mg (wt.%)	2.16	2.15	1.98	1.21	1.68	2.88
Mn (wt.%)	0.05	0.10	0.20	0.77	1.59	2.89
Al (wt.%)	80.82	81.48	93.72	95.06	92.63	88.05

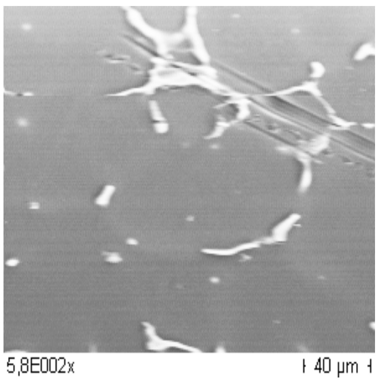

Fig. 12. Analyzed area of EDX analysis of AlCu4MgMn alloys (as-cast condition, scanning electron microscope).

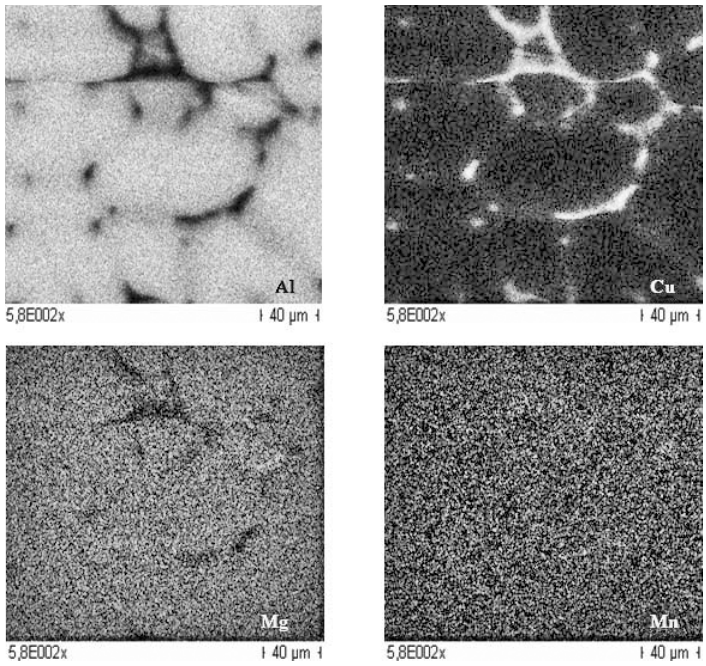

Fig. 13. EDX analysis of AlCu4MgMn alloy (Fig. 12) (distribution of aluminium, copper, magnesium and manganese, scanning electron microscope).

To evaluate the quality of homogenizing annealing in addition to point EDX analysis, EDX analysis of surface is used to distribute alloying elements in the selected area of the sample alloy AlCu4MgMn homogenized at 490°C for 8 hours in typified aluminium parent metal (see Figs 12, 13 and Figs 14, 15).

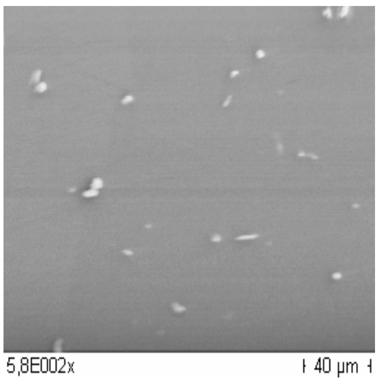

Fig. 14. Analyzed area of EDX analysis of AlCu4MgMn (homogenized $T = 490°C/8$ h, scanning electron microscope).

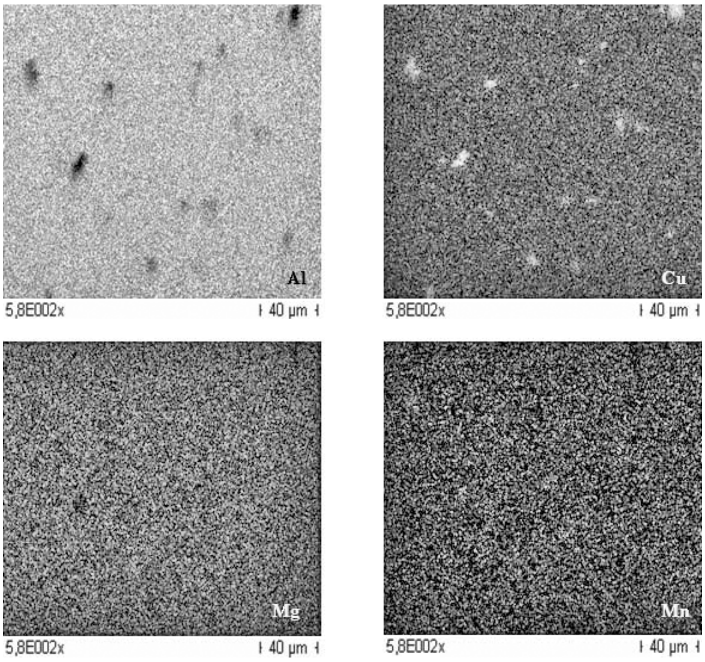

Fig. 15. EDX analysis of AlCu4MgMn alloy (Fig. 14) (distribution of aluminium, copper, magnesium and manganese, scanning electron microscope).

The images obtained during EDX analysis show the different areas of concentration of the analyzed elements. Higher concentrations of the analyzed element are represented by a light grey to white colours.

3. CONCLUSION

Optimal conditions of homogenizing annealing of AlCu4MgMn alloys are evaluated in terms of microstructure image analysis using energy dispersive spectroscopy. The selected parameters are homogenizing annealing time at a constant temperature and annealing temperature of homogenizing annealing at constant duration of this process. In terms of microstructure of optimal homogenization, annealing temperature at constant time of 8 hours for AlCu4MgMn alloys lies within the range from 490 to 510°C. Annealing at higher temperature leads to homogenization of the melted eutectic grain boundaries and globular bodies of melted eutectic grains inside.

Influence of homogenizing annealing on microstructure of the investigated alloy is observed after two hours of this process. Optimum annealing time of AlCu4MgMn alloy homogenization can be determined in the range of 8–10 hours at the temperature of homogenization annealing 490°C.

Quantitative evaluation method of image analysis is used to determine the effect of homogenizing annealing time on the percentage of produced particles and their size in AlCu4MgMn alloy cast in a metal mold. The graphical dependence of the average percentage of particles on the time of homogenization at the temperature of 490°C shows that with increasing time of homogenization annealing the percentage decreases from 4.11% to 0.81%. The most intense homogenizing annealing effect is observed after 4-hour soak at homogenization annealing temperature 490°C, the percentage of particles decreased from 4.11% to 1.85%. After the following eight hours the percentage of particles decreased to 1.14%. Size of particles, which were observed in the alloy after 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 of AlCu4MgMn alloys show that the process of homogenizing annealing caused dissolution of the intermetallic phase and subsequent diffusion of the elements into solid solution α. Data obtained by EDX suggest the presence of heterogeneities in the form of the increased undissolved copper content (16.63 wt.%). It can confirm the presence of insoluble phase AlCuFeMn, AlSiMnCuFe, and Al12Mn2Cu or undissolved residual phases and eutectic $CuAl_2$, $CuAl_2 + \alpha$, $\alpha + CuAl_2 + Mg_2Si$, which melt at 517°C.

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