The Crystal Segregation During Casting of the Alloy AlZn5.5Mg2.5Cu1.5

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Abstract

In the course of homogenizing annealing of aluminium alloys being cast continually or semi-continually it appears that chemical inhomogenity takes off within separate dendritic cells (crystal segregation). It is about a diffusion process that takes place at the temperature which approaches the liquid temperature of the material. In that process the transition of soluble intermetallic compounds and eutectic to solid solution occurs and it suppresses crystal segregation significantly [1]. The temperature, homogenization time, the size of dendritic cells and diffusion length influences homogenizing process. The article explores the optimization of homogenizing process in terms of its time and homogenizing annealing temperature which influence mechanical properties of AlZn5,5Mg2,5Cu1,5 alloy.

Keywords: Theory of crystallization, Heat treatment, Homogenizing annealing, AlZn5,5Mg2,5Cu1,5 alloy, Crystal segregation

1. Introduction

Homogenization is defined as a method of heat treatment that happens at high temperature approaches liquid (approximately 0,7 -0,8 of melting temperature) with the purpose of eliminating chemical inhomogeneity by the means of diffusion processes. In the process of crystallization a long interval between a liquid and a solid state causes the difference in chemical composition in the middle and on the boarders of the dendrite. The presence of balanced and not balanced eutectics which appears in the structure of AlZnMgCu alloy casts deteriorates mechanical properties of the alloy. It is possible to minimize or even to eliminate completely chemical inhomogeneity in crystal segregation by the means of homogenizing annealing. The article describes heat treatment process optimization in the terms of the structure and chosen mechanical properties of AlZn5,5Mg2,5Cu1,5 alloy[2, 3].

2. Experiment

Raw material that was given by a producer was used for making a cast of a tested material. Casts of tested alloys were prepared taking into consideration chemical composition defined by CSN 42 4222 standard. The material was melted in a furnace at the temperature of 730 °C, the furnace temperature was read by a digital thermometer with ± 2°C accuracy. In the process of melting the melt was treated with a refining salt and melt surface skimming was done. A prepared material was cast into a preheated (at the temperature of 220 °C) mould in a gravitational way. The casts were in a form of a conical cylinder in size of 40 /50 x 100 mm. The chemical composition of a prepared tested alloy (mass percentage) is shown in Tab.1.

Being composed in such a way the alloy was divided into two sets of specimens which were subjected to homogenizing annealing.

Table 1.
The chemical composition of AlZn5,5Mg2,5Cu1,5 alloys

AlZn5,5Mg2,5Cu1,5 metallic form [mass %]	Zn	Mg	Cu	Si	Fe	Al
	5,21	1,89	1,47	0,05	0,06	91,30

Annealing was carried out in a LAC furnace, the temperature was read by a digital thermometer with ± 2°C accuracy. The first set of specimen was subjected to different temperature conditions with temperature ranges of 410-530 °C with 20°C intervals (410; 430; 450; 470; 490; 510; 530°C), annealing lasted 8 hours.

The second set of specimen was made at a constant temperature of homogenizing annealing (T=470°C), but with a different time period of a heat treatment process. The time of homogenizing annealing was from 2 hours to 24 hours (2; 4; 6; 8; 10; 12; 16; 20; 24 o´clock). For microstructure and micro-hardness analysis, according to Vickers, with a load of 20 g. with a 5-second duration metallographic grinds were made out of the specimen before and after homogenizing annealing.

AlZn5,5Mg2,5Cu1,5 alloy structure was accentuated by the method of color etching, when a color contrast appears as a response of a specimen to an etching agent (KMnO$_4$) [4]. By the means of that method the inhomogeneity of chemical composition of dendritic cells was revealed.
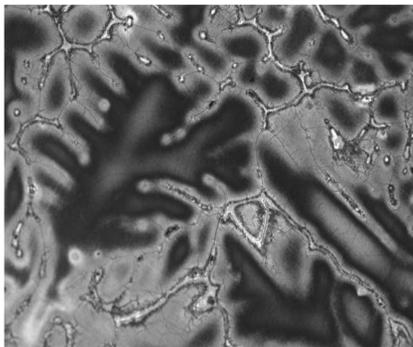

Fig. 1. AlZn5,5Cu2,5Mg1,5 alloy microstructure before homogenizing annealing, mag. 200x

After homogenizing annealing the microstructure of the specimens (of their metallographic grinds) was analyzed with a confocal laser microscope LEXT with magnification 100x.

The AlZn5,5Cu2,5Mg1,5 alloy microstructures at constant annealing lasting 8 hours are shown above, magnification 100x.

The analysis of microstructure of an experimental alloy being heat treated constantly with a changeable temperature shows that the temperature of 430 – 450°C is insufficient for crystal segregation suppression. It is obvious according to images (Fig. 2b, Fig. 2c) of AlZn5,5Mg2,5Cu1,5 structure that not all present soluble eutectics and intermetallic compounds have melted. At the temperature of 510°C it is possible to observe the structure and it is evident that at such a high temperature melting eutectics, which are arranged along dendritic cells, and spheroid melting eutectics appear. After AlZn5,5Mg2,5Cu1,5 alloy structure observation it is possible to state that homogenizing annealing at the temperature of 430 – 530°C is the optimal condition for homogenization at the temperature range of 470 – 490°C.

After microstructure evaluation all these AlZn5,5Mg2,5Cu1,5 alloy specimens, particularly their micro-hardness, that were subjected to constant 8-hour annealing were analyzed according to Vickers with a load of 20 g. with a 5-second duration, that determines the optimal temperature of 470°C for homogenizing annealing (Fig. 4). The same temperature was chosen to treat another set of specimens which were subjected to annealing lasting from 2 up to 24 hours.
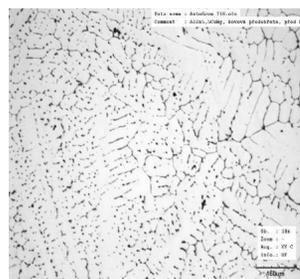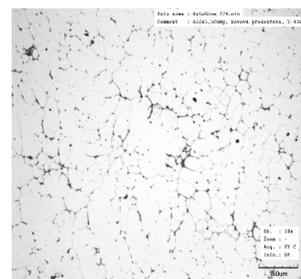

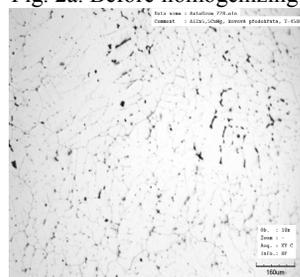Fig. 2a. Before homogenizing   Fig. 2b. 430°C
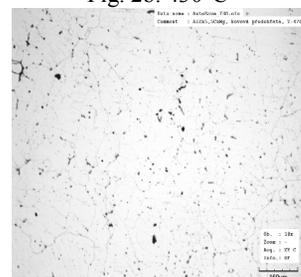
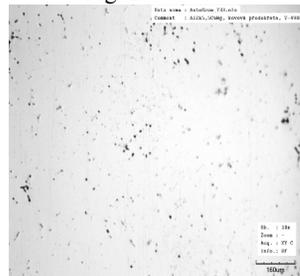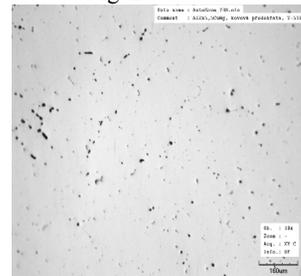Fig. 2c. 450°C   Fig. 2d. 470°C

Fig. 2e. 490°C   Fig. 2f. 510°C

The AlZn5,5Cu2,5Mg1,5 alloy microstructures at constant annealing temperature of 470°C are shown above, magnification 100x.

It is known that homogenizing time depends on the structure of a tested alloy. As it is seen from the Fig. 3a, showing the structure of AlZn5,5Mg2,5Cu1,5 alloy before homogenizing, the structure of a prepared alloy which is poured into a preheated mold is finely reticular and the melting of intermetallic compounds and the diffusion of presented elements inside dendritic cells will not be time-consuming. From the Fig. 3b and Fig. 3c it is possible to state that annealing time at the temperature

of 470°C is not sufficient. Considering the structure it is possible to specify an optimal homogenizing time ranging from 8 to10 hours when the melting of melting eutectics and the diffusion of elements into a solid solution α is completed. With a longer homogenizing time the elimination of precipitate occurs and pores appear as a result of melting and eutectics solidification of a tested alloy, it is obvious from the Fig. 3f.

The hardness of all the specimens of a tested alloy was defined according to Vickers, it characterizes material hardness properties and is influenced by the chemical composition and structure of the material. Micro-hardness of metallographic grinds in the centre of dendritic cells with a load of 20 g with a 5 - second duration was defined [5, 6]. The dependence of tested alloy hardness on the temperature and homogenizing time is presented graphically on the Fig. 4. and Fig. 5.
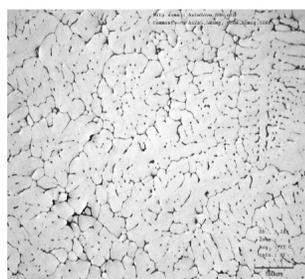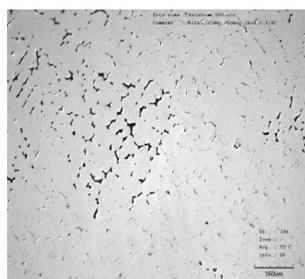

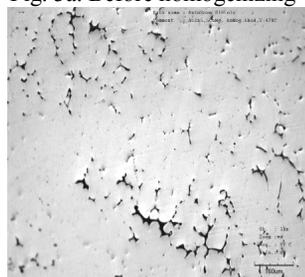Fig. 3a. Before homogenizing

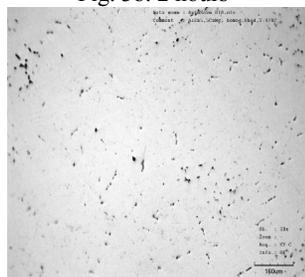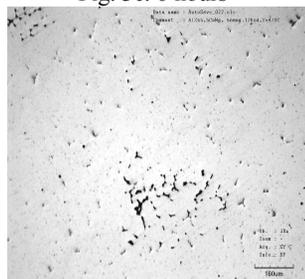Fig. 3b. 2 hours

Fig. 3c. 6 hours

Fig. 3d. 8 hours

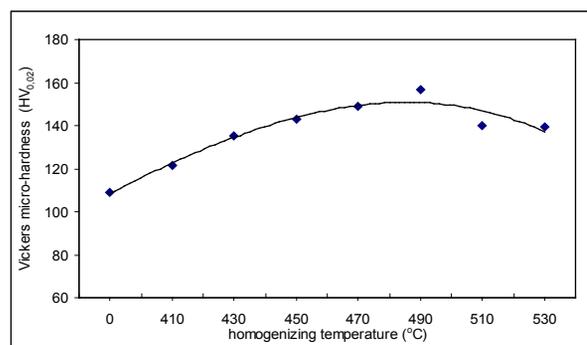Fig. 3e. 12 hours

Fig. 3f. 20 hours

Fig. 4. The dependence of AlZn5,5Mg2,5Cu1,5 alloy hardness on homogenizing temperature along 8 hours according to Vickers

From the Fig. 4 it is possible to state that with the increase of homogenizing annealing temperature (T = 410 – 490°C) the micro-hardness of a tested AlZn5,5Mg2,5Cu1,5 alloy, according to Vickers, in the centre of dendritic cells is growing. The specimens of a tested alloy which were homogenized with the temperature of 470 up to 490°C during 8 hours demonstrate the maximum micro-hardness value according to Vickers. With further temperature increase the hardness is getting lower, it occurs because of a melting eutectic which appears on the borders of the grains and because of a spheroid melting eutectic which appears inside the grains.
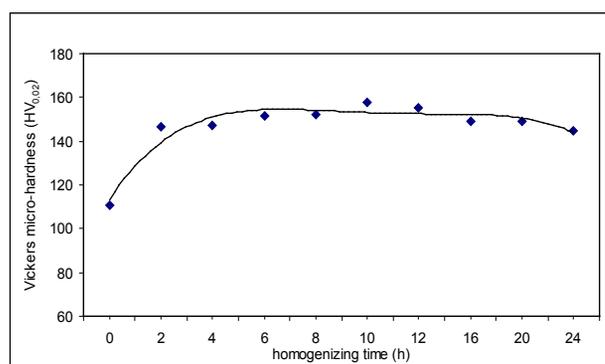

Fig. 5. The dependence of AlZn5,5Mg2,5Cu1,5 alloy hardness, according to Vickers, on homogenizing time with a constant temperature T = 470°C

As it is shown graphically, in the terms of AlZn5,5Mg2,5Cu1,5 alloy micro-hardness, according to Vickers, and its dependence on homogenizing time with a constant temperature of 470°C, it is possible to state the following: after 2 hours of homogenizing annealing the micro-hardness of the material is growing expressively; with a longer homogenizing (8-12 hours) of a tested material the hardness of a tested material, according to Vickers, is growing gently; homogenizing annealing with the temperature of 470°C and which lasts more than 20 hours causes a gentle decrease of a tested material hardness that occurs because of eliminated precipitate coarsening inside dendritic cells in a solid solution α. In general, it is possible to state that 2 hours of homogenizing annealing of that experimental alloy with the

temperature of T = 470°C is enough to gain maximal micro-hardness value, according to Vickers, which is analyzed in the central part of dendritic cells.
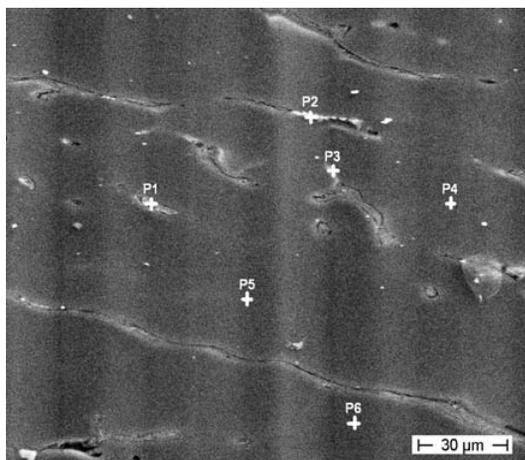

Fig. 6. Pattern EDX analysis of AlZn5,5Mg2,5Cu1,5 alloy before annealing, the places of analysis are marked as P1-P6

Table 2.
Pattern EDX analysis values of AlZn5,5Mg2,5Cu1,5 alloy's marked places Fig. 6 P1-P6

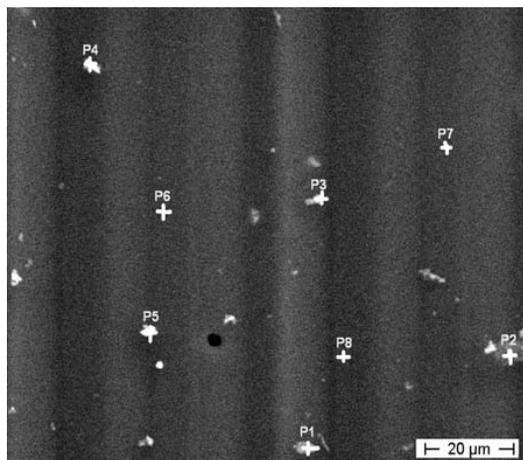| wt. %	Mg	Al	Cu	Zn
P1	1,77	76,14	7,43	14,67
P2	1,42	64,00	22,60	11,98
P3	2,17	76,66	4,68	16,49
P4	2,16	85,03	1,20	11,60
P5	2,17	85,07	1,28	11,48
P6	2,10	81,06	2,56	14,27

Fig. 7. Pattern EDX analysis of AlZn5,5Mg2,5Cu1,5 alloy after annealing with the temperature of T = 470°C during 8 hours, the places of analysis are marked as P1-P8

Table 3.
Pattern EDX analysis values of AlZn5,5Mg2,5Cu1,5 alloy's marked places Fig. 7 P1-P8

wt. %	Mg	Al	Cu	Zn
P1	2,05	77,51	3,92	16,52
P2	1,83	75,85	6,90	15,41
P3	2,03	77,28	3,93	16,75
P4	1,91	76,80	5,50	15,79
P5	2,12	77,17	4,18	16,53
P6	1,91	77,40	4,17	16,52
P7	2,01	77,19	4,06	16,75
P8	2,20	77,27	3,84	16,70

7,3E002x   ⊢ 40 µm ⊣
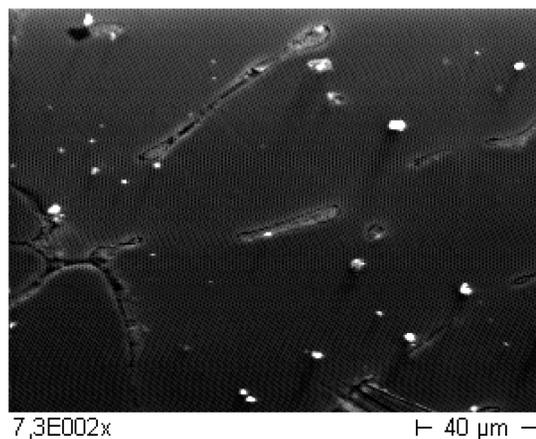
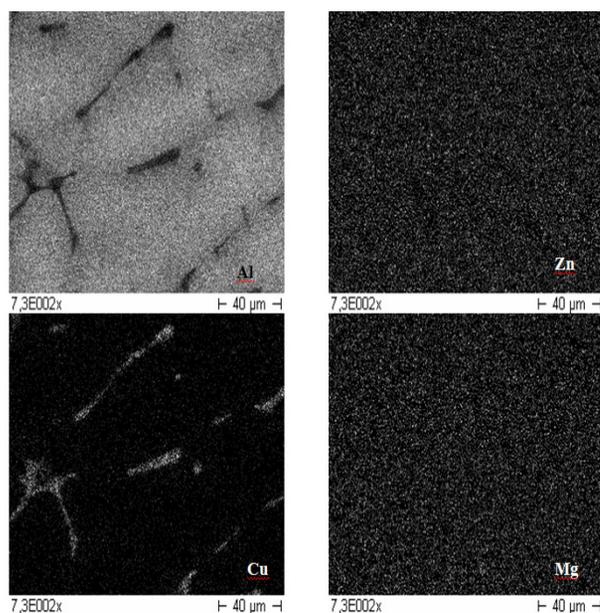Fig. 8. Analyzed area EDX analysis AlZn5,5MgCu, homogenized T = 470°C/8 h, scanning electron microscope

Fig. 9. EDX analysis AlZn5,5MgCu alloy (Fig.8), distribution of Al, Zn, Mg, Cu, scanning electron microscope

Fig. 10. Analyzed area EDX analysis AlZnMgCu, homogenized T = 470°C/8 h, scanning electron microscope

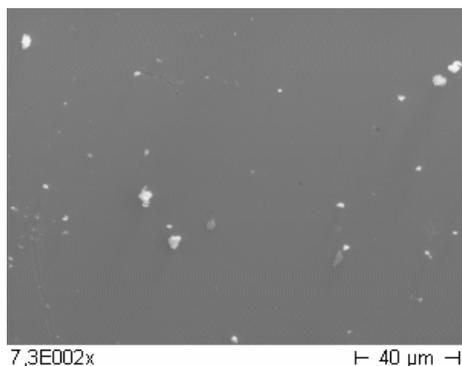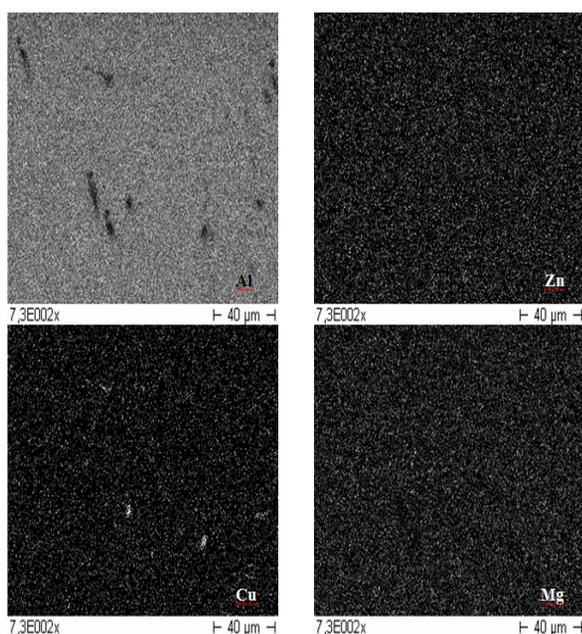Fig. 11. EDX analysis AlZn5,5MgCu alloy (Fig.10), distribution of Al, Zn, Mg, Cu, scanning electron microscope

Considering pattern EDX analysis values of an experimental AlZn5,5Mg2,5Cu1,5 alloy before homogenizing annealing it is possible to state the following:

– the structure of an experimental AlZn5,5Mg2,5Cu1,5 alloy before homogenizing is composed of α – solid solution and a soluble and insoluble eutectic of a type $\alpha + Mg_3Zn_3Al_2$ (quasibinary) or an insoluble eutectic of a type $\alpha + CuMgAl_2$. It is also possible to reveal the presence of soluble tertiary eutectics $\alpha + Mg_5Al_8 + Mg_3Zn_3Al_2$ and $\alpha + Mg_3Zn_3Al_2 + MgZn_2$. The presence of copper causes the growth of eutectic of a type $\alpha + CuMgAl_2 + Mg_3Zn_3Al_2 + MgZn_2$, the most part of it will dissolve into $MgZn_2$ or $Mg_3Zn_3Al_2$.

– from the Fig.6 it is possible to define the presence of eutectic of a type $\alpha + CuAl_2$, which can also appear after a short period of ageing.

Due to pattern EDX analysis of specimens after homogenizing annealing with the temperature of T = 470°C during 8 hours it is possible to conclude the following:

– the solubility of copper and zinc under homogenizing is extremely high, and during homogenizing very sound and fast diffusion of that elements into a solid solution α occurs;

– from the Fig. 7 it is evident that the most number of presented soluble eutectics had dissolved at the temperature of 470°C after 8 hours. It is about a soluble low-meltable eutectic $\alpha + Mg_5Al_8 + Mg_3Zn_3Al_2$, which dissolves at the temperature over 450 °C. It is possible to state that even eutectic $\alpha + Mg_3Zn_3Al_2 + MgZn_2$, which can dissolve at the temperature of 475°C, has dissolved under those homogenizing conditions.

– from the structure and EDX analysis values of an experimental alloy under the homogenizing temperature of 470 °C and time of 8 hours Fig. 7 it is evident that existing eutectics $\alpha + CuMgAl_2$ are not dissolved (P2,P4).

3. Conclusions

After the experiments with AlZn5,5Mg2,5Cu1,5 alloy it is possible to conclude the following:

1. From the microstructure of AlZn5,5Mg2,5Cu1,5 alloy that was homogenized during a constant time of 8 hours with a changeable temperature it is possible to state that considering the microstructure of AlZn5,5Mg2,5Cu1,5 alloy, homogenizing annealing with the temperature of 430 up to 530 °C is an optimal homogenizing condition with the temperature range of 470 up to 490°C. At the higher temperature over 510°C as it is seen from the microstructure melting eutectics set along the borders of dendritic cells and spheroid melting eutectics appear.

2. After microstructure evaluation of experimental alloy sets being made with the constant temperature of T=470°C it is possible to conclude that annealing time of 2-6 hours is not sufficient. Considering the microstructure after treatment with 470°C the optimal homogenizing time is 8 up to 10 hours, when the dissolution of soluble eutectics and the diffusion of elements into a solid solution α are completed.

3. Considering the micro-hardness of a solid solution α, according to Vickers, with a load of 20g. with a 5-second duration (the micro-hardness of all the specimens was analyzed in the middle part of dendritic cells) it is possible to determine the optimal annealing condition - it is constant annealing time of 8 hours with the temperature range of 470 up to 490°C.

4. From an experimental alloy made with the constant temperature of 470°C (Fig. 5) it is possible to observe a considerable hardness growth of a solid solution α, according to Vickers, after 2 hours of homogenizing annealing. Homogenizing annealing which lasts 20 hours causes a gentle micro-hardness decrease of a tested material, it takes place because of eliminated precipitate coarsening inside dendritic cells in a solid solution α.

5. The results of EDX analysis of an experimental AlZn5,5Mg2,5Cu1,5 alloy before and after homogenizing with the temperature of 470°C lasting 8 hours show that the

structure of AlZn5,5Mg2,5Cu1,5 alloy before homogenizing is made with a solid solution α and a soluble and insoluble eutectic of a type α + $Mg_3Zn_3Al_2$ (quasibinary) or an insoluble eutectic of a type α + $CuMgAl_2$. It is possible to presuppose the presence of soluble tertiary eutectics α + Mg_5Al_8 + $Mg_3Zn_3Al_2$ and α + $Mg_3Zn_3Al_2$ + $MgZn_2$. Copper influences the growth of eutectic of a type α + $CuMgAl_2$+ $Mg_3Zn_3Al_2$ + $MgZn_2$, the most part of it will dissolve into $MgZn_2$ or $Mg_3Zn_3Al_2$. From the the Fig. 6 (P2) it is possible to disclose the presence of eutectic of a type α + $CuAl_2$, which can also appear after a short period of ageing. From the Fig. 7 of a homogenized alloy it is evident that the most number of presented soluble eutectics had dissolved at the temperature of 470°C after 8 hours. It is about the eutectic of a type α + Mg_5Al_8 + $Mg_3Zn_3Al_2$ and the eutectic of a type α + $Mg_3Zn_3Al_2$ + $MgZn_2$. The eutectics of a type α+$CuMgAl_2$ under the temperature of 470°C during 8 hours are not dissolved (P2, P4).

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