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RESEARCH OF THE CHEMICAL HETEROGENEITY DURING CRYSTALLIZATION FOR AlZn5,5Mg2,5Cu1,5 ALLOY BEFORE AND AFTER HOMOGENIZATION

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Abstract. Crystal segregation is taken as chemical heterogeneity under the micro-scale and it develops during the crystallization process. Alloy crystallization does not take place under a particular temperature, as it happens in the case of pure metals, but it runs under a certain temperature interval. When cooling the melt, various places start development among dendritic cells which differ in their chemical composition. Crystal segregation can be generally defined as chemical heterogeneity developing during the alloy crystallization process, and it can be either enriched or in contrast depleted with alloying elements and impurities, which are unevenly segregating over the entire dendritic surface. In the central part of the dendritic cells there is an alloy, which is depleted with alloying elements, while the edge areas of dendritic cells and interdendrite space present higher concentration of alloying elements. This concentration shows a hyperbolic development; when the central part of dendritic cells area has the lowest alloying elements concentration, while the edge part of a dendritic tree and the interdendrite space show the maximum concentration. The distance between two main axes of dendritic cells is affected by the temperature interval running between the liquid and solid phase of the chosen alloy, as well as by melt cooling rate and temperature gradient during the solidification phase. The shorter distance between the axes of dendritic cells appears under faster cooling, which allows very fast heat dissipation and creates very fine structure of the resulting alloy. The longer distance between the main axes of dendritic cells stimulates greater segregation appearing under slow melt cooling.
Keywords: Crystal segregation, dendritic, homogenization annealing, AlZn5,5Mg2,5Cu1,5 alloy, crystal segregation, EDX analysis, image analysis.

1. Introduction

This article deals with the foundry moulds influence (metallic, bentonit sand) on the AlZn5,5MgCu alloy structure and the selected mechanical properties of this alloy. The foundry mould influence can significantly influence the creation and extent of the crystal segregation and the subsequent homogenization temperature process. The research is focused on the creation and extent of the crystal segregation and its removal with homogenization annealing in which is observed the individual factors influence influencing this diffusion process. These factors include the homogenization annealing temperature, the soak temperature length and the diffusion course size. The diffusion course size directly relates with the dendritic cells size which are dependent on the chosen foundry mould for casting namely metallic and bentonit mould.

The AlZnMgCu alloy are the strongest aluminium alloys, superdural (tensile strength 700-720 MPa), they have very good mechanical properties in welds, but their negative qualities are propensity to all kinds of corrosion, lower fracture toughness and higher notch sensitivity. Zinc is the main alloying element of heat treatable alloys which belong to a series 7xxx. In conjunction with Mg creates intermetallic phase $MgZn_2$. Zinc has the greatest solubility in aluminium from all the existing elements (solubility limit is 67 at. % Zn at 381 °C). The disadvantage of these alloys is poor corrosion resistance and susceptibility to large crystal and band segregation. The cause of the crystal segregation is selective solidification of the crystal at a gradual change in the composition of the solid phase. Important parameters influencing the development and degree of crystal segregation are: chemical composition of the alloy, the diffusion in the solid and liquid phase, the crystallization speed and the casting cooling or the intensity of heat removal from the casting which depends on the chosen form of the casting. The crystal segregation significantly influences the mechanical and corrosion properties of the alloy. The crystal segregation is possible to suppress by homogenizing annealing. The homogenization annealing process influences the temperature and the time of the homogenization process, diffusion coefficients of the respective elements in the aluminium matrix and the size of the diffusion course. The temperature and time of the homogenization process is chosen so as to dissolve the non-equilibrium intermetallic phases which formed in the crystallization process and the subsequent diffusion of the respective elements in solid solution α. It is similar by the non-equilibrium eutectics incurred in a higher crystallization rate, than is equilibrium state, which dissolves and the respective elements diffuse into the matrix α. The homogenizing annealing temperature is set in the range from 0.90 to 0.95 M_p. The homogenization annealing can be performed at lower temperatures 0.8 – 0.9 M_p. The reducing of the homogenizing annealing temperature process can significantly extend the homogenization annealing process up to several tens of hours.

The casting method and the correct choice of the casting form have crucial importance to the final structure and properties of alloys. The material of the metallic moulds must withstand relatively high temperatures, in some cases about 400 to 600 °C. The big drawback in their manufacture is their high price and laboriousness and therefore is their usage mainly for large-scale production of castings. For this reason, it is convenient for small-scale production to produce cheaper forms of technologies based on sand. The bentonite sands allow casting in to large moulds for raw because they are binding and good breathable. Compared with casting in metal moulds are cheaper however castings from the metal moulds are characterized by excellent internal compactness, better surface quality, lower roughness, fine-grained structure, good dimensional values and mechanical properties of castings.

2. Experimental part

To prepare castings of the researched material was used aluminium of the purity 99.8% and the necessary alloying metals. The castings of the researched alloys were prepared according to the chemical composition of the original standards CSN 42 4222 - EN AW 7075. The melting of the material took place in an electric resistive furnace K 70/13 at the temperature 730 °C, the temperature of the furnace was captured using a digital thermometer with an accuracy of ± 2 °C. The melt was treated with the refining salt during the melting and the scrapings were withdrawn from the surface of the melt. The prepared material was gravity-cast into a metal mould preheated to a temperature of 220 °C and to the bentonite mould prepared from the bentonite sand. The castings were in the shape of a conical cylinder with dimensions of 40/50 x 100 mm. Chemical compositions of prepared experimental alloys in wt. % is shown in (Table 1).

Table 1
Chemical composition of the experimental alloy AlZn5,5Mg2,5Cu1,5

Chemical composition [wt. %] metallic preheated mould		Chemical composition [wt. %] bentonite mould	
Zn	5,21	Zn	5,25
Mg	2,08	Mg	2,13
Cu	1,47	Cu	1,59
Si	0,05	Si	0,05
Ti	0,002	Ti	0,001
Fe	0,06	Fe	0,06
Al	91,12	Al	90,90

Surface roughness of the prepared AlZn5,5Mg2,5Cu1,5 alloy castings was evaluated using a confocal laser microscope OLYMPUS LEXT OLS 3100. Scanning of the castings surface took place in confocal mode with the help of the analysis of roughness on the line. For purposes of evaluating the surface roughness of the castings were measured most commonly used height parameters Ra, Rz and Rq (Table 2).

Table 2
Evaluation of surface roughness based on the parameter Ra, Rz, Rq

Foundry mould	Ra [μm]	Rz [μm]	Rq [μm]
Metallic	13,94	187,76	22,25
Bentonite	34,30	596,70	59,79

To evaluate the microstructure was prepared from the castings of the alloy AlZn5,5Mg2,5Cu1,5 metallographic thin sections. The microstructure of the prepared casting was observed as in the central region so in the edge of the casting in the state of etching using a phosphoric acid solution (Fig. 1).

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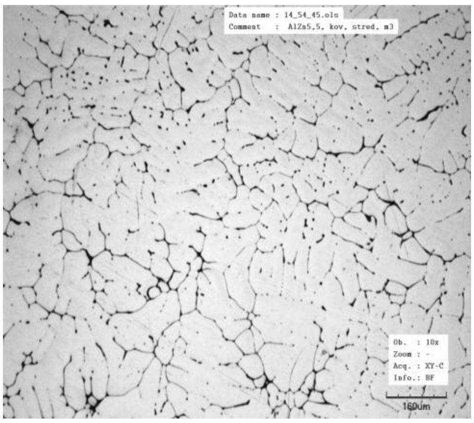

Fig. 1 The AlZn5,5Mg2,5Cu1,5 alloy microstructures – metal mould edge mag. ×100

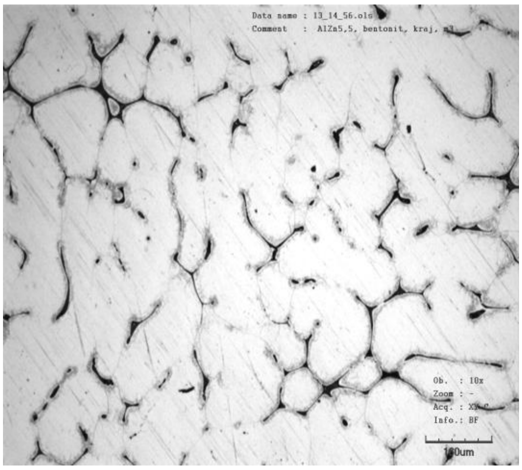

Fig. 2 The AlZn5,5Mg2,5Cu1,5 alloy microstructures – bentonite mould, mag. ×100

AlZn5,5Mg2,5Cu1,5 alloy structure was accentuated by the method of colour etching, when a colour contrast appears as a response of a specimen to an etching agent ($KMnO_4$). By the means of that method the inhomogeneity of chemical composition of dendritic cells was revealed (Fig.3, Fig.4).

Fig. 3 AlZn5,5Cu2,5Mg1,5 alloy microstructure before homogenizing annealing, metallic form, enl.: ×100

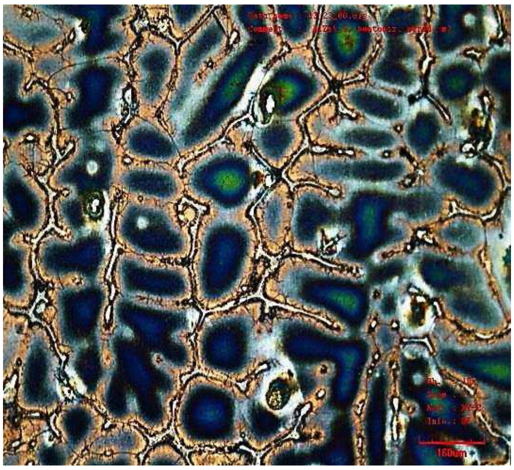

Fig. 4 AlZn5,5Cu2,5Mg1,5 alloy microstructure before homogenizing annealing, bentonic form, enl.: ×100

From the microstructures of the prepared samples cast into a metal and bentonite mould were numerically evaluated the sizes of dendritic cells (20 measurements) using a confocal laser microscope OLYMPUS LEXT OLS 3100.

Table 3
Evaluation of measured sizes of dendritic cells alloys AlZn5,5Mg2,5Cu1,5 [μm]

Foundry mould	Min.	Max.	Average
Metallic preheated, the centre	171	279	210
Bentonite, the centre	274	756	490

The prepared samples of the AlZn5,5Mg2,5Cu1,5 alloy were further heat treated with homogenization annealing at a constant temperature T = 470 °C for annealing time from 2 to 24 hours. Homogenization annealing was carried out in a furnace with a digital thermometer reads the temperature with an accuracy of ± 2 °C. From the samples of AlZn5,5Mg2,5Cu1,5 alloy after homogenization annealing were prepared grindings on which were analyzed the chemical compositions of the occurring phases and the solid solution with the EDX method.

The chemical composition of the basic structural components and the relative distribution of the parent metal and the main alloying elements were made on scanning electron microscope using EDX analysis. EDX is a very suitable method for the identification of all present elements, foreign particles and the different structural components in the alloy. Using this method was determined the chemical composition of a eutectic and the inner region of dendrite AlZn5,5Mg2,5Cu1,5 alloy for casting.

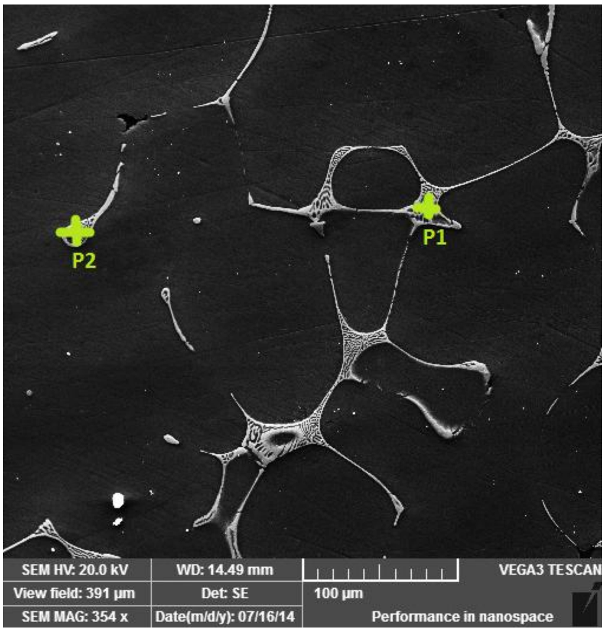

Fig. 5 SEM of the alloy AlZn5,5Mg2,5Cu1,5 analysed by surface method EDS, before annealing, the places of analysis are marked as P1, P2

Table 4
Pattern EDX analysis values of AlZn5,5Mg2,5Cu1,5 alloy's marked places Fig.5, P1, P2

Analyzed elements	P1	P2
Cu [wt.%]	20,90	10,10
Zn [wt.%]	26,65	12,82
Mg [wt.%]	20,14	7,34
Al [wt.%]	32,31	69,73

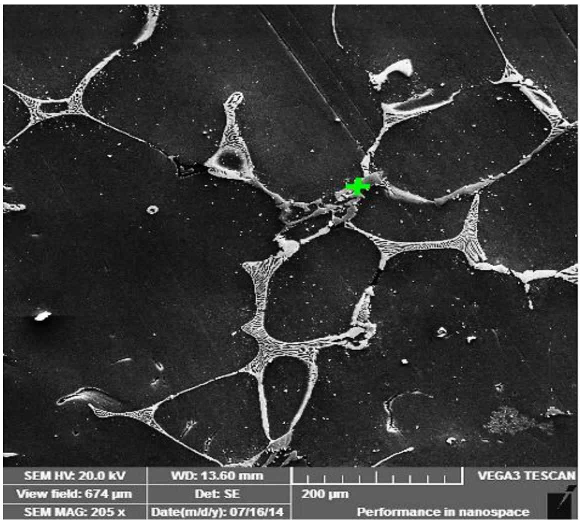

Fig. 6 SEM of the alloy AlZn5,5Mg2,5Cu1,5 analysed by surface method EDS, before annealing

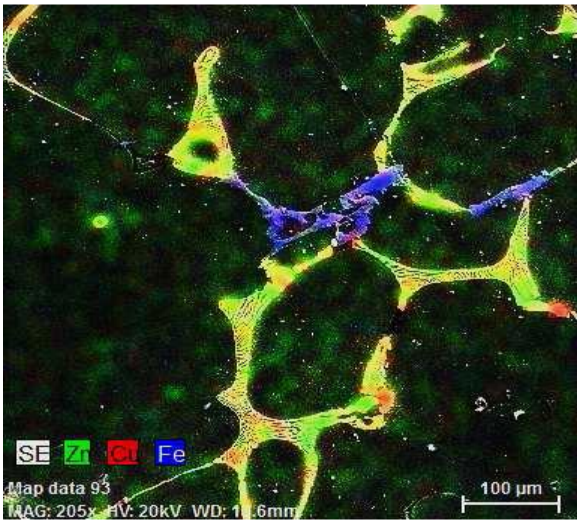

Fig. 7 Concentration maps of selected elements of the alloy AlZn5,5Mg2,5Cu1,5, before annealing

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

Analyzed elements	P1
Cu [wt.%]	1,89
Fe [wt.%]	18,89
Ni [wt.%]	11,07
Al [wt.%]	68,15

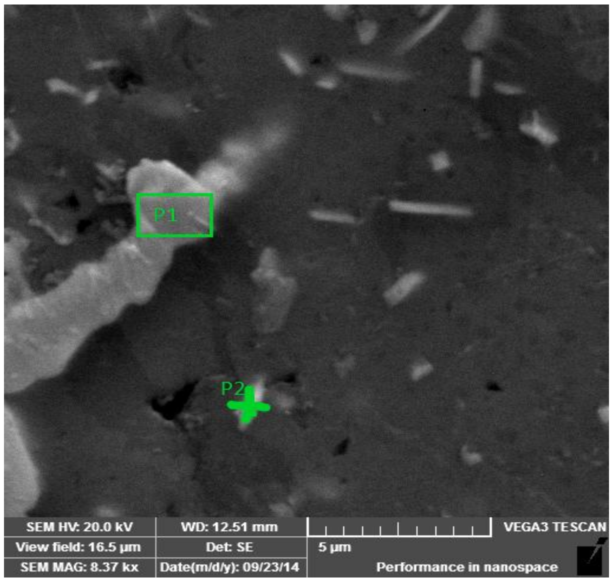

Fig. 8 SEM of the alloy AlZn5,5Mg2,5Cu1,5 analysed by surface method EDS, after annealing with the temperature of T = 470°C during 8 hours, the places of analysis are marked as P1, P2.

Table 6
Pattern EDX analysis values of AlZn5,5Mg2,5Cu1,5 alloy's marked places Fig.8, P1, P2

Analyzed elements	P1	P2
Cu [wt.%]	28,30	3,61
Fe [wt.%]	12,71	-
Mg [wt.%]	1,02	6,87
Zn[wt.%]	-	18,12
Al [wt.%]	57,97	71,39

Vickers micro hardness HV0,02 of the given material before and after homogenization were analyzed in a central region of dendritic cells at 20 g load for the duration of 5 seconds.

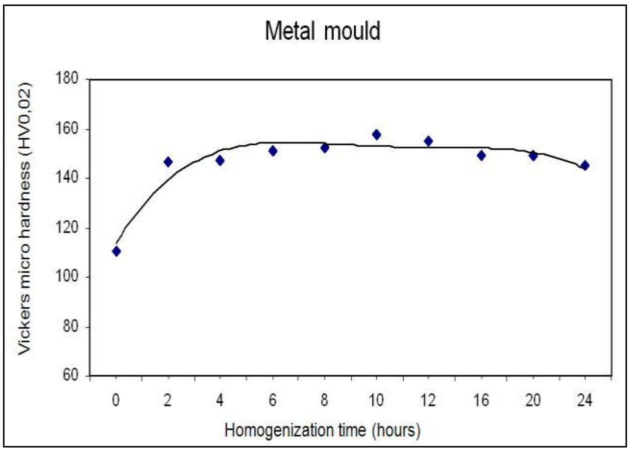

Fig. 9 Dependence of Vickers micro hardness of the AlZn5,5Mg2,5Cu1,5 alloy at the time of homogenization at a constant temperature T = 470 °C - metal mould

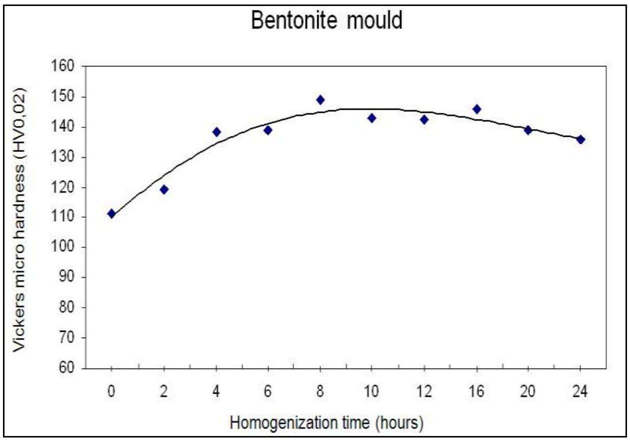

Fig. 10 Dependence of Vickers micro hardness of the AlZn5,5Mg2,5Cu1,5 alloy cast into the bentonite mould at the homogenization time at a constant temperature T = 470 °C.

3. Conclusions

From the performed experiments by the alloy AlZn5,5Mg2,5Cu1,5 is possible to formulate the following partial results. From the viewpoint of performed macrostructures and microstructures prepared castings of the alloy AlZn5,5Mg2,5Cu1,5 is apparent that the size of the dendrite in the alloy cast to the bentonite mould is on average twice greater than in the alloys cast into metal moulds. To evaluate the surface roughness were used most commonly used height parameters Ra, Rz and Rq. Low surface roughness reached alloy cast into a metal mould, the higher surface roughness was measured by the casting from the bentonite mould.

The average size of dendritic cells by the alloys cast into a metal preheated mould was 210 microns. By the alloy cast in the bentonite mould was determined the average size of dendritic cells 490 microns. It follows that the size of the dendritic cells is by the casting of the examined alloy cast into the bentonite mould twice greater than by the cast of the alloy cast in to the metal mould.

From the results of EDS analysis of experimental alloy AlZn5,5Mg2,5Cu1,5 before homogenization is evident that the structure of this alloy is formed with α - solid solution and with a soluble and insoluble eutectics of the type α + $Mg_3Zn_3Al_2$ (quasibinary) and insoluble eutectic α + $CuMgAl_2$. We can assume the presence of soluble tertiary eutectic α + Mg_5Al_8 + $Mg_3Zn_3Al_2$ a α + $Mg_3Zn_3A_{l2}$ + $MgZn_2$. Copper causes the formation of eutectic type α + $CuMgAl_2$+ $Mg_3Zn_3Al_2$ + $MgZn_2$, the majority part dissolves during the heat treatment in $MgZn_2$ or $Mg_3Zn_3Al_2$. The concentration maps of the elements Al, Zn, Mg, Cu shows an increased concentration of copper in the interdendritic spaces (bright places) where there is evident low concentration of aluminium. EDS spot analysis showed the occurrence of phases rich in iron and nickel, the iron concentration (18.9 wt.%) and nickel (11 wt.%), copper up to 2 wt. % (Fig.6). It is an intermetallic phase and AlFeCuNi AlCuNi comprised of nickel and iron, which are insoluble. The analysis shows a relatively even distribution of the zinc and it is in the whole surface of dendritic cells. From the record is apparent that the distribution of magnesium is in the interdendritic spaces poorer.

Form the Vickers micro hardness of the investigated alloy in the central area of dendritic cells was found significant effect of the casting mould on the micro hardness HV0,02 of the given material. From the graphical dependence can be concluded that the alloy cast in a preheated metal mould occurs after two hours of homogenizing annealing to a significant increase in micro hardness HV0,02. This is related to the smaller size of dendritic cells (shorter diffusion path) during the homogenization process, during which occurs to the dissolution of the non equilibrium intermetallic phases and eutectic in α matrix and subsequent precipitation during cooling from the α matrix. In the case of samples of alloys cast in the bentonite mould is required the homogenizing annealing process was longer because the maximum values of micro hardness HV0,02 thus prepared alloy occurs up to 8 hours after the homogenizing annealing.

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