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Cooling rate and chemical composition influence on structure of Al-Si-Cu alloys

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Materials

ABSTRACT

Purpose: The aim of this work is to perform the investigation of cooling rate influence as well as rare earth metals modification on microstructure of the AC-AlSi7Cu3Mg and AC-AlSi12CuNiMg cast aluminium alloys. In the work also artificial neural networks were applied for investigations of the influence of the alloying additives on the properties of the AC-4XXX alloy.
Design/methodology/approach: In the work the thermo – derivative analysis was applied for the reason to determine changes occurred in the Al-Si-Cu alloy caused by cooling rate change in a range between 0.1 and 1.4°C/s as well chemical composition of the investigated alloy. Also artificial neural networks were applied for prediction of the chemical composition and heat treatment parameters and influence on mechanical properties of the investigated aluminium alloys.
Findings: The performed investigation are discussed for the reason of an possible improvement of thermal and structural properties of the alloy.
Practical implications: The aim of the carried out investigations was to work out a computer aided tool for prediction of mechanical properties on the basis of registered parameters during the technological process as well as controlling the process in real time, which can be useful for foundry and cast industry for achieving of material with assumed properties.
Originality/value: Chemical composition and cooling rate applied for the alloy influences the crystallisation process of the phases and eutectics, and that fore also the microstructure and determines at the same time the properties of these aluminium alloys. The achieved results can be used also for liquid metal processing in science and industry and obtaining of a required alloy microstructure and properties influenced by a proper production conditions. The determination of the technological process parameters as well chemical composition allows it to predict the material properties.
Keywords: Aluminium alloys; Thermo analysis; Structure; UMSA; Neural network

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1. Introduction

The commonness of appliance of cast elements makes it necessary to increase the amount of scientific investigation projects concerning both a proper alloying additives amount in form of rare earths metals and boron, which improves mechanical properties by microstructure modification, and also the production technology of elements with highest possible strength, wear and corrosion resistance as well as working out and verification of manufacturing technology in industry condition [1-3].

Investigations connected to the elaboration of an optimal chemical composition and manufacturing method for aluminium alloy with addition of chosen metal including rare earths metals and boron with enhanced properties compared to elements manufactured using traditional alloys and methods, will lead to a better understanding of mechanisms leading to improvement of mechanical properties of these new developed alloys. Permanent trend for increasing of the production costs of constructional elements, together with improvement of their mechanical properties, particularly low mass and corrosion and wear resistance has caused an increase of investigation projects about a combination of these properties with new developed constructional materials and production conditions [4,5].

Properties of the new worked out Al alloys depend basically on matrix properties and the alloying additive, as well as the manufacturing conditions of the material, phase morphology, size and distribution of the precipitations in the aluminium matrix [6-11].

Investigation results of rare earths metals and boron influence on microstructure achieved by rapid cooling of the Al-Si alloy show also different transitions types connected to the applied concentration of the rare earths metals, boron and cooling rate. Additionally the seldom occurred ‘‘cell lessening effect’’ can occur. This effect base increasing d-spacing in the AlFeVSiMn alloy after heat treatment together with increasing content of rare earths metals concentration added to the alloy. This effect is probably caused by influence of the complex mechanisms occurred between the rare earths metals and compounds of the aluminium alloys [12].

Modification of hypo and hyper eutectics Al-Si alloys causes change in the silicon crystals structure and it decreases the inter-phase distance in the α+β eutectic. In hypereutectic Al-Si alloys the modification is carried out for the reason of primary silicon particles refinement through addition of crystallizations nuclei [13-14].

An effective way for grain refinement is for example increase of the cooling rate. In has an significant influence on aluminium alloys properties, particularly susceptibility for hot cracking or susceptibility for shrinkage porosity [15-18].

Grin size decreases nearly paraboloidally together with the increase of crystallization rate. Cooling rate has a big influence onto dendritic segregation [19-25]:

- low rate cooling causes an uniform microstructure and dendrite decline,
- by low cooling rate, which is typical for a given alloy, instead of a grained structure, there occurs a dendritic structure, achieving of a defined temperature causes a maximal dendritic segregation,
- by a very high cooling rates a fine-grained microstructure will be achieved by differences in chemical composition of particular grains.

Appliance of a low amount of lanthanum and cerium causes a grand refinement (0.1% addition causes a decrease of the grain size about. 14%, and addition of 0.3% a decrease of ca. 23%). Both the additives work also as antioxidants by building of La_2O_3 and Ce_2O_3 oxides, which are nuclei of the α phase [26].

The novel universal metallurgical simulator and analyzer (UMSA) technology platform is capable of collecting in situ and analyzing on-line, the thermal characteristics of metallurgical treated melts and solidifying and heat-treated test samples, using precision-controlled heating and cooling rates while simulating industrial conditions. For this reason on the crystallization curve occurs some characteristic inflexion points coming from exothermic or endothermic reactions of the crystallizing phase transformations. It is difficult to determine unequivocally the crystallization temperature of the phases occurred on the crystallization curve. The determination is possible using the first derivative curve of the cooling line in function of time, that mines using the differential ATD curve called also derivative curve [27-29].

The UMSA system is equipped with an special computer program for control of the simulation process. It makes it possible in an elastic way to program the heat flow simulation itself, taking into account the heating rate, cooling rate and isothermal hold time. Moreover it is also equipped with modulus for interpretation of the collected data using the thermo analysis, which allows it to determine the characteristic temperature of the phase transitions occurred during the melting or solidification process [30].

Appliance of artificial intelligence method allows it to perform a prediction of properties of the cast aluminium alloys, based on the entrance data, for example the chemical composition or heat treatment parameters. Neural network is a system of programs and data structures that approximates the operation of the human brain. A neural network usually involves a large number of processors operating parallel, each with its own small sphere of data and access to data in its local memory. Normally a neural network is initially trained or fed large amounts of data and rules about data relationships. A program can then tell the network how to behave in response to an external stimulus, for example, to input from a computer user who is interacting with the network, or can initiate activity on its own, within the limits of its access to the external world [31,32].

During the homogenisation process of as-cast Al-Mg-Si alloys a phase transformation takes place transforming single plate-like β-AlFeSi particles into multiple, more rounded, α-AlFeMnSi particles. This intermetallic phase transformation is of technological interest since it improves the extrudability of the aluminium considerably. Therefore this transformation will be analysed in more detail in this thesis. In this introduction, first the scope of the thesis will be given, detailing the focal points of this research. Subsequently some background will be given about the intermetallic phase transformations as well as the approaches to model intermetallic transformations. Finally the outline of this thesis is presented, explaining briefly the contents and conclusions of each separate chapter [33].

One important microstructural change during homogenisation is the transformation of plate-like intermetallic β-Al5FeSi particles to multiple rounded α-AlFeMnSi type of intermetallic particles. The change in morphology shows that the plate-like β particles in the as-cast state appear as needles in a two dimensional micrograph. After homogenisation these β plates are transformed into a string of separate coarse α particles. The β-to-α phase transformation considerably improves the extrusion process of the aluminium since the transformed α-particles in the homogenised material improve the ductility of the material and the surface quality of the extruded material. Often, the as cast microstructure is not extrudable at all as many cracks appear when as cast material is extruded anyway. Additional microstructural changes, such as the dissolution of Mg_2Si or Si particles, also occur during homogenisation. However, since the Mg_2Si or Si particles dissolve rather fast, it is the β-to-α transformation kinetics which determines the minimum homogenisation time that is needed to get the material in a suitable state for extrusion [20,21,33-36].

2. Materials and experimental procedure

For evaluation for the dependence between chemical composition and cooling temperature in the range between 0.1 and 1.4°C/s of the cast aluminium alloys of the AC-AlSi7Cu3Mg and AC-AlSi12CuNiMg type named according to the EN 1706:2001 standard, following investigations were performed:

- Microstructure of the alloy was investigated using MEF4A optical microscope supplied by Leica together with the image analysis software as well electron scanning microscope (SEM) using Zeiss Supra 25 device with SE and BSE electron detection. The samples for optical microscope investigations were electro etched using 30% HBF_4 solution,
- EBSD technique was applied for phase composition investigation,
- Chemical composition of the Al alloy was investigated using qualitative and quantitative X-Ray analysis, as well EDS microanalysis,
- derivative thermo analysis using the UMSA device (Universal Metallurgical Simulator and Analyser),
- also artificial neural networks were applied for the reason of prediction of mechanical properties of the AC-4XXX type alloys. The independent variables of this network were put in in form of chosen chemical elements: Si, Mn, Mg, Cu as well temperature of solution heat treatment and ageing, where as the dependent variables of the network were set in form of the following mechanical properties: tensile strength, hardness and elongation.

Chemical composition of the investigated alloys was presented in Tables 1 and 2. To the alloy were added rare earths metals in form of lanthanum and cerium of 0.1 mass per cent. The range of the taking in values into the neural network for the reason to determine the dependence between chemical composition and temperature of the heat treatment was presented in Table 3.

For thermo-derivative analysis samples of ϕ30 mm in diameter and 35 mm in high were used. The samples were melted in graphite crucible and 0.025 mm thick steel foil. For cooling argon gas was applied. For temperature measurement a chromel-alumel thermocouple of the K type was applied with a reaction time of 250 ms. Two pieces of thermocouples were used for temperature measurement. The first thermocouple was placed 5 mm from the wall of the samples and the second one in the centre axis of the sample in place were the highest temperature occurs (Fig. 1).

Table 1.
Chemical composition of AC-AlSi7Cu3Mg aluminium alloy

Mass concentration of the element, %							
Si	Cu	Mg	Mn	Fe	Ti	Zn	Ni
7.5	3.5	0.3	0.25	0.36	0.11	0.13	0.04

Table 2.
Chemical composition of AC-AlSi12CuNiMg aluminium alloy

Mass concentration of the element, %							
Si	Cu	Mg	Mn	Fe	Ti	Zn	Ni
11.8	1.05	1	0.14	0.5	0.1	0.13	0.95

Table 3.
Values of the input and output variables chosen for analysis using artificial neural networks

Value	Mass fraction of the element, %			
	Si	Cu	Mg	Mn
Min.	4	0	0	0
Max.	23	5	1.5	0.8
Value	Saturation	Aging	Tensile	Hardness
	°C	°C	MPa	HB
Min.	490	155	200	60
Max.	535	230	250	100

3. Results and discussion

The presented investigation results show influence of the rare earths metals (0.1% mass concentration) on microstructure refinement as well change of the crystallization process, what can be seen comparing the derivative curves of the non-modified (Figs. 2 and 3) as well the modified alloy (Fig. 4), and comparing also the liquidus and solidus points of the investigated materials.

In Fig. 1 there is also presented the microstructure of the investigated alloys as well places were the thermocouples were placed.

a) b)

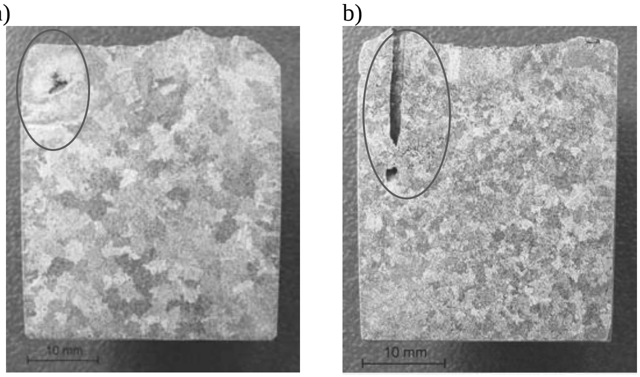


Fig. 1. Microstructure of the AC-AlSi7Cu3Mg alloy, cooling rate ~0.3°C/s: a) non-modified alloy, b) modified alloy with rare earths metals addition

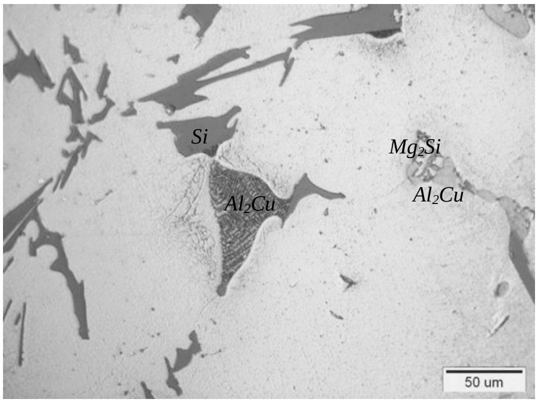


Fig. 2. Microstructure of the AC-AlSi7Cu3Mg cooled with ~ 0.2ºC/s

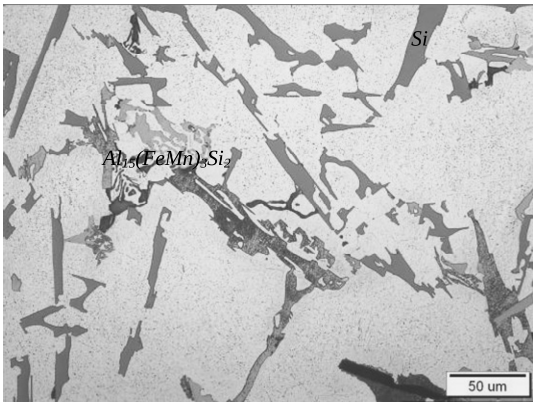


Fig. 3. Microstructure of the AC-AlSi12CuNiMg cooled with 0.7ºC/s

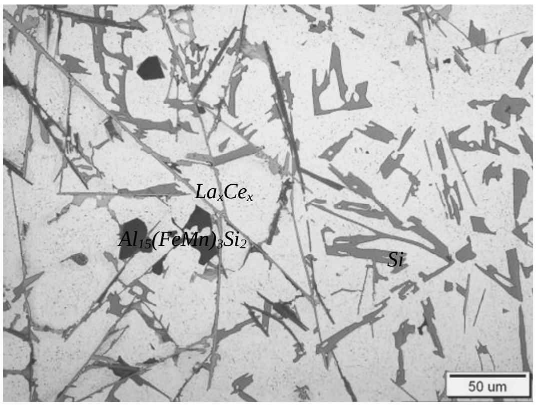


Fig. 4. Microstructure of the AC-AlSi12CuNiMg cooled with 1.4ºC/s, with addition of select rare earth metals

Point-wise X-Ray microanalysis performed in the marked place in Fig. 5 shows the chemical composition in place of the investigated point, presented in Figs. 6, 7 and Table 4.

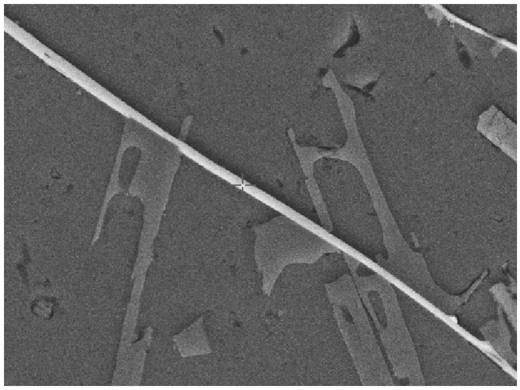


Fig. 5. SEM micrograph of the investigated AC-AlSi12CuNiMg alloy

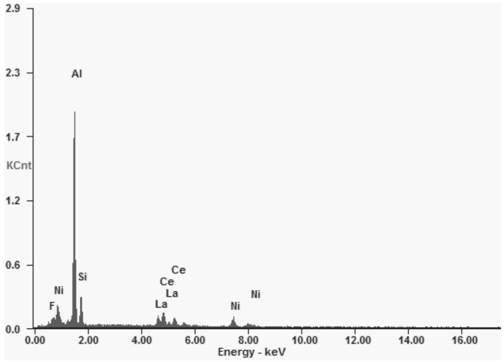


Fig. 6. EDS point wise analysis of the investigated aluminium cast alloy, marked in Figure 5

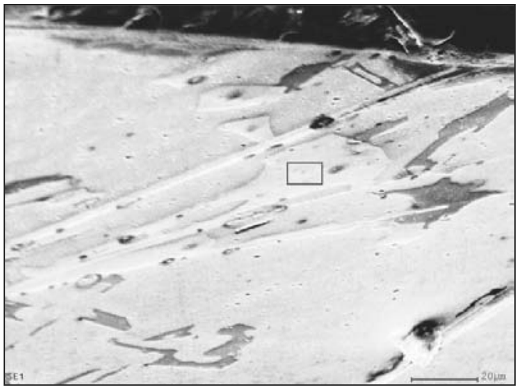
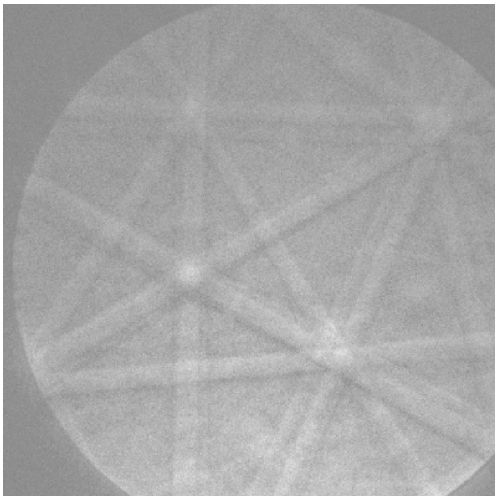


Fig. 7. Surface EDS microanalysis

Table 4.
Mass and atomic chemical composition of the La and Ce containing phase presented in Figs. 4 and 5

Element	Wt%	At%
AlK	63.99	72.71
SiK	13.51	14.75
MnK	04.41	02.46
FeK	14.56	07.99

a)



b)

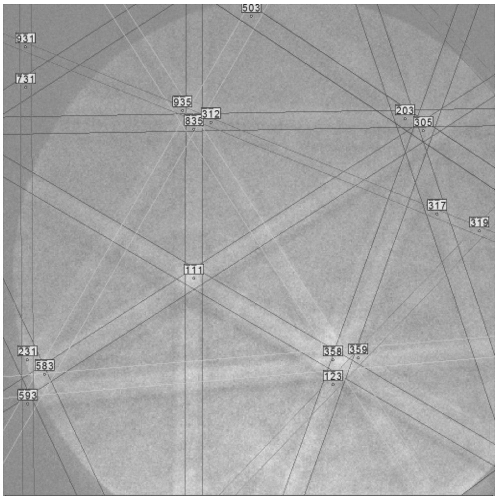


Fig. 8. EBSD investigations results of the Fe and Mn containing phase: a) obtained Kikuchi lines, b) calculated Kikuchi lines

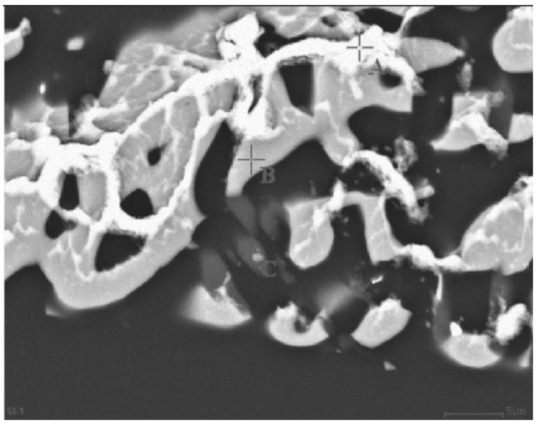


Fig. 9. SEM micrograph of the investigated Al_2Cu and Mg_2Si phase in AlSi7Cu3Mg alloy

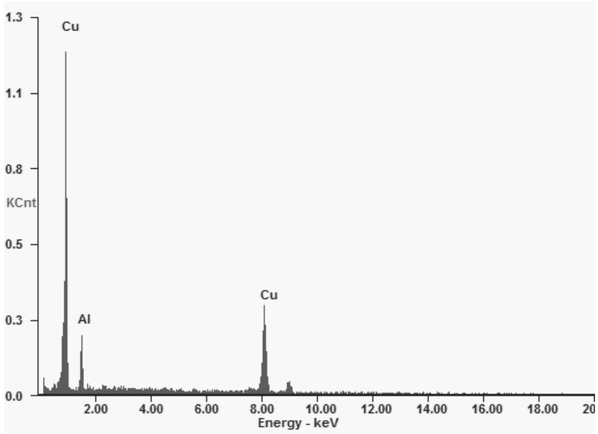


Fig. 10. EDS point wise analysis of the investigated aluminium cast alloy, marked A in Figure 9

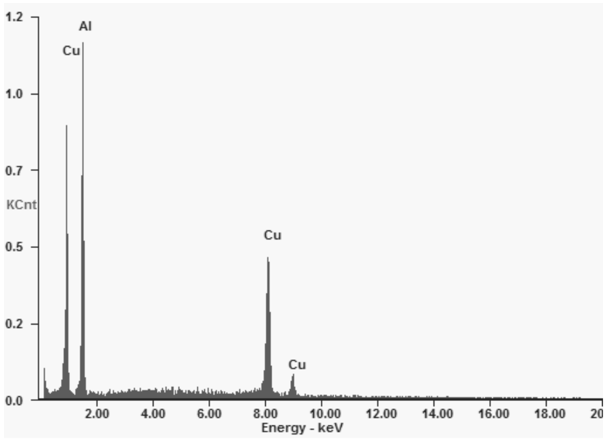


Fig. 11. EDS point wise analysis of the investigated aluminium cast alloy, marked B in Figure 9

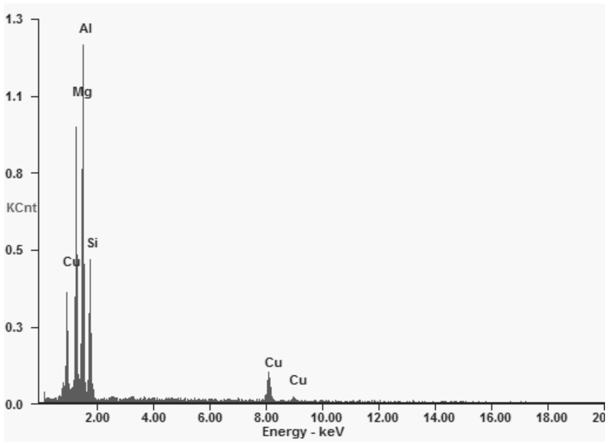


Fig. 12. EDS point wise analysis of the investigated aluminium cast alloy, marked C in Figure 9

Table 5.
Crystallisation sequence of the phases and eutectics of the AlSi7Cu3Mg alloy with the correspondent temperature of the reaction

Summary of reactions	Reaction temperature of the alloy cooled with a cooling rate of ~0.1°C/s	Reaction temperature of the alloy cooled with a cooling rate of ~1.2°C/s	Reaction temperature of the modified alloy cooled with a cooling rate of ~0.3°C/s
1. Start of solidification and formation of α-Al	605	605	607
2. Precipitation of iron intermetallic phase Liq → α + $Al_{15}(FeMn)_3Si_2$	592	591	597
3. Start of main eutectic reaction Liq → α + β	561	570	559
4. Precipitation of hypereutectic phase Liq → α + Al_2Cu + Mg_2Si + β	501	500	504
5. End of solidification	482	431	479

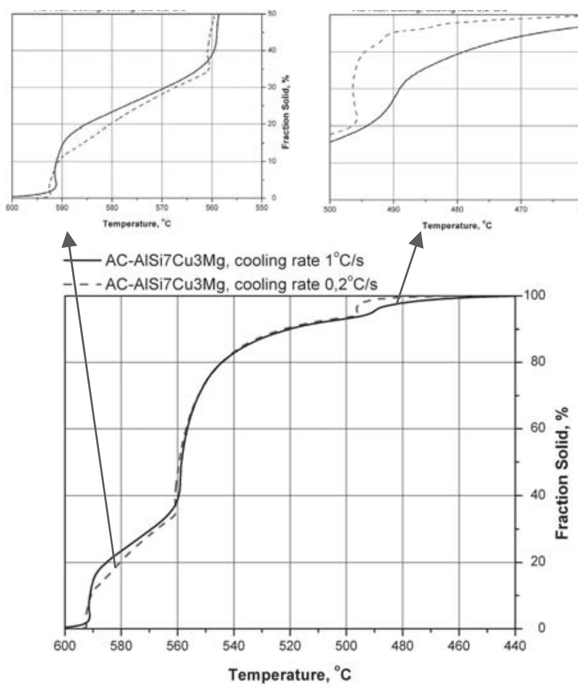


Fig. 13. Fraction solid for the AlSi7Cu3Mg alloy

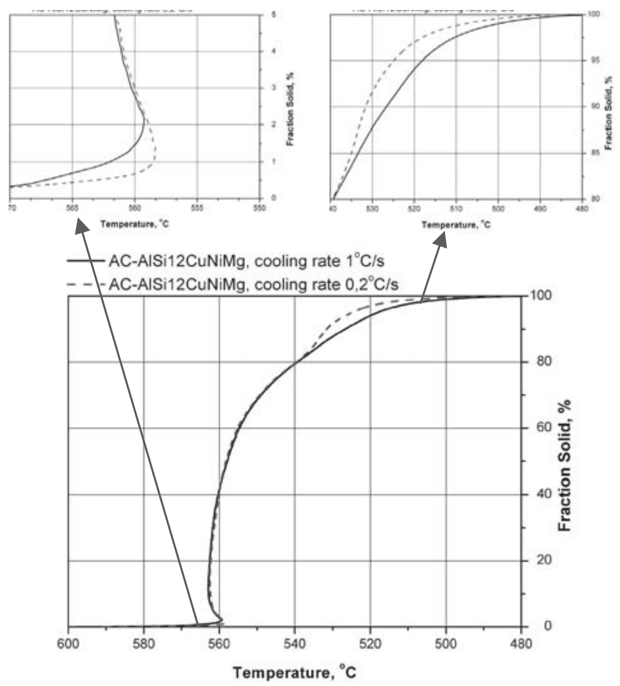


Fig. 14. Fraction solid for the AlSi12CuNiMg alloy

In Fig. 7 there is marked the EDS measurement area with the data presented in Table 4. This analysis was preformed to collect more data foe adequately atomic chemical composition determination. Kikuchi lines obtained by mind of EBSD technique (Fig. 8) have allow it to determine the occurrence of the iron manganese containing phase, more accurate determination is possible using electron diffraction method. In Fig. 9 there is presented a micrograph of the Al_2Cu phase in the BSE mode which allows it to distinguish between to form of this phase a bright (white) core and grey surrounding edge. So there is an evidence for non-uniformity of the Al_2Cu phase, probably the stechiometry of Al and Cu changes not in a fluently way but steep -like, rapidly, the reason for this should be the theme for future investigations. In Figures 10 to 12 there are presented the chemical composition measurements of the Al_2Cu are, where significant differences can be detected in the Cu concentration in the measurement points marked in Fig. 9.

Change of the cooling rate of ~0.8°C/s causes a crystallisation kinetic change of the Al alloy, as a result of a slightly different fraction solid curve, what was presented of the AlSi7Cu3Mg alloy in Fig. 13 and for the AlSi12CuNiMg alloy in Fig. 14.

In Figs. 15 to 20 there is presented the influence of cooling rate and modification with lanthanum and cerium onto the crystallization kinetics of the investigated aluminium alloys.

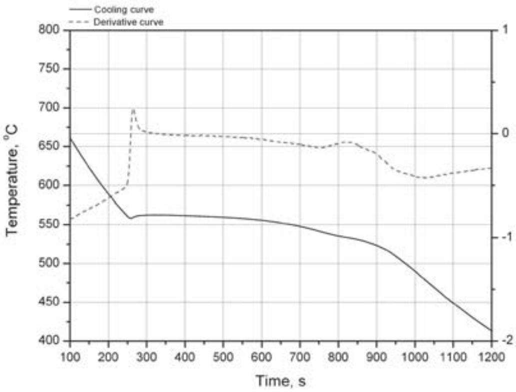


Fig. 15. Cooling curve and derivative curve of the AlSi12CuNiMg alloy cooled with a rate of 0.1°C/s

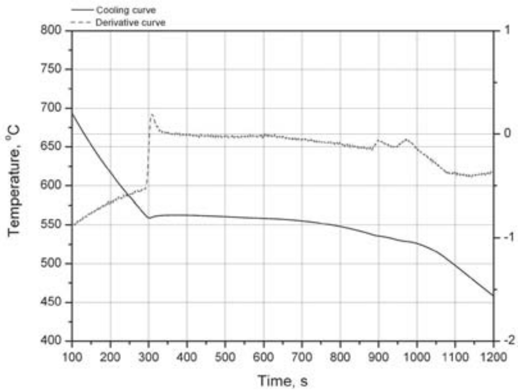


Fig. 16. Cooling curve and derivative curve of the AlSi12CuNiMg alloy cooled with a rate of 0.3°C/s modified with rare earths metals

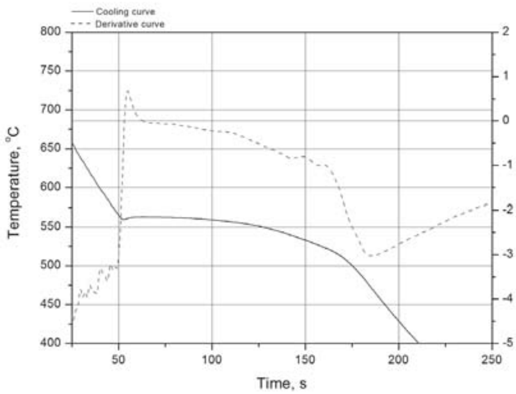


Fig. 17. Cooling curve and derivative curve of the AlSi12CuNiMg alloy cooled with a rate of 1.2°C/s

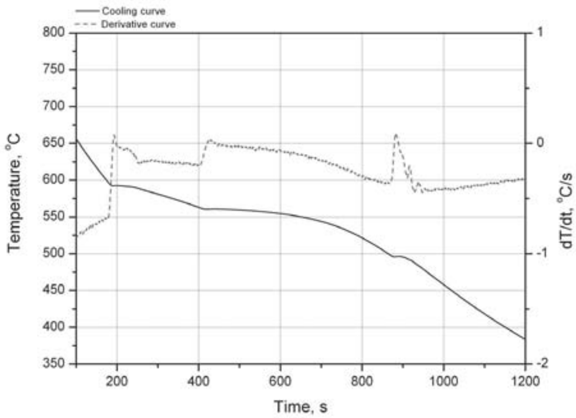


Fig. 18. Cooling curve and derivative curve of the AlSi7Cu3Mg alloy cooled with a rate of 0.1°C/s

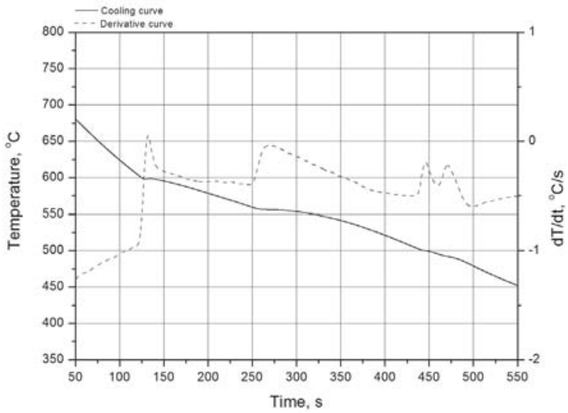


Fig. 19. Cooling curve and derivative curve of the AlSi7Cu3Mg alloy cooled with a rate of 0.3°C/s modified with rare earths metals

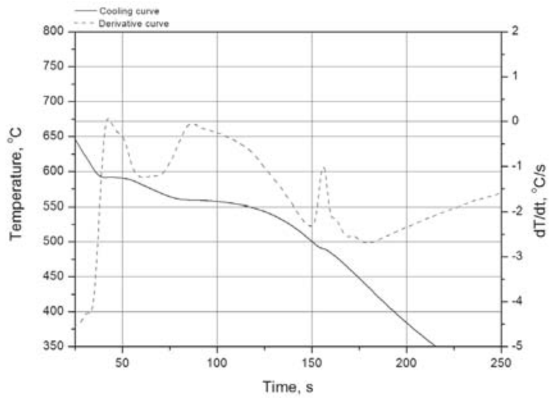


Fig. 20. Cooling curve and derivative curve of the AlSi7Cu3Mg alloy cooled with a rate of 1.2°C/s

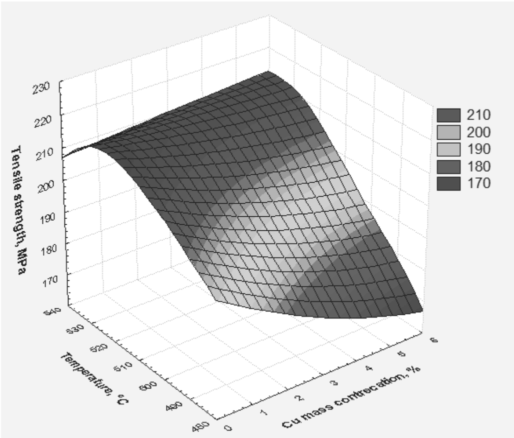


Fig. 21. Mapping diagram of Cu and saturation temperature influence on tensile strength

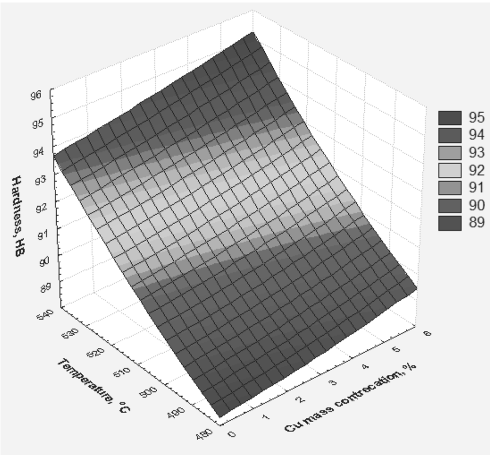


Fig. 22. Mapping diagram of Cu and saturation temperature influence on hardness

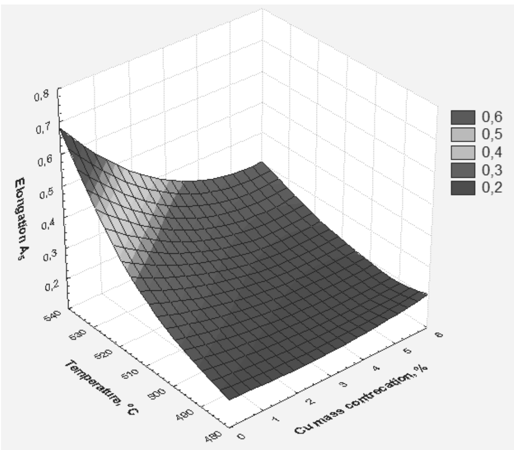


Fig. 23. Mapping diagram of Cu and saturation temperature influence on elongation

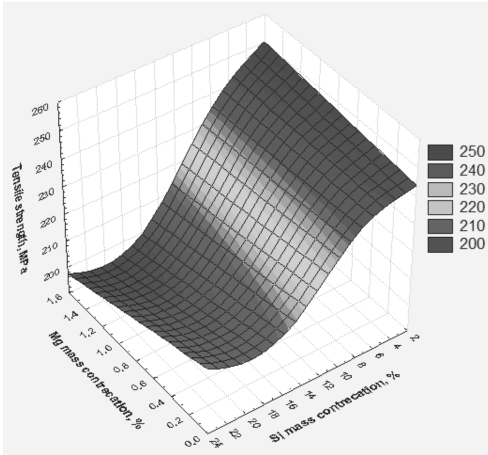


Fig. 24. Mapping diagram of Mg and Si influence on tensile strength

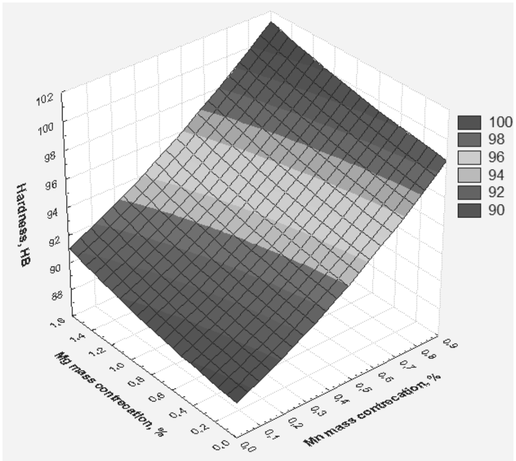


Fig. 25. Mapping diagram of Mg and Mn influence on hardness

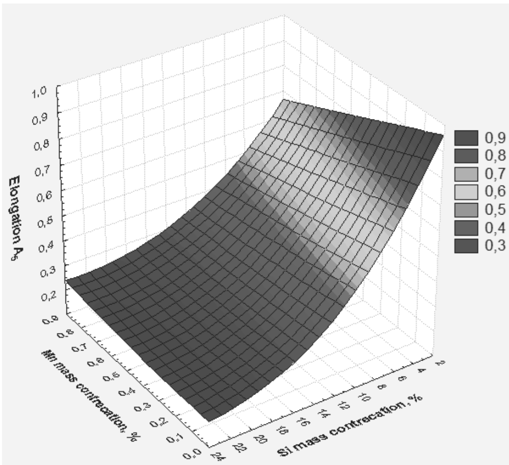


Fig. 26. Mapping diagram of Mn and Si influence on elongation

In Figures 15, 16 as well 18, 19 there is presented the cooling curve and the derivative curve of the freely cooled alloys as well as in Figs. 17 and 20 for forced cooled alloys. Modification of the alloy causes change in the derivative curve, what influences also the microstructure of the investigated alloys, and that fore also their mechanical properties. Increase of the cooling rate causes an enhancement of the overcooling of the investigated alloys as well a change of phase morphology. In Table 5 there is presented the crystallisation sequence of the phases and eutectics of the AlSi7Cu3Mg alloy with the correspondent temperature of the reaction. There are presented both the freely cooled as well the forced cooled lanthanum and cerium modified alloy.

It was found by mind of the chemical analysis using artificial neural networks that the hardness of the alloy increases together with the Cu content increase (Fig. 22), simultaneously the strength decreases - with a content over 2% Cu (Fig. 21) - because of the occurrence of brittle cracking.

Cu content in the Al-Si alloy causes the occurrence of the Al_2Cu phase and gives itself the possibility for precipitation hardening and that fore for material strengthening.

Also with a higher Cu content an increase of the elongation occurs (Fig. 23). Strength increase accrues also as a result of Mg mass concentration increase In form of Mg_2Si phase occurrence (Fig. 24). An addition of magnesium in aluminium alloys make sit possible to carry out of precipitation hardening, if causes also a shift of the crystallization point of the multi phase eutectic to lower temperature. An increase of the mass manganese and silicon content (lower then 8%) causes an increase of the elongation of the analysed aluminium alloys (Fig. 26).

Together with the increase of magnesium and manganese mass per cent content there occurs also a hardness increase of the analysed aluminium alloys (Fig. 25).

4. Conclusions

Properties of the cast alloys of the AC-4XXX series are depending not only on chemical composition but also on cooling rate. Modification of the aluminium alloys with rare earths metals including lanthanum and cerium causes microstructure refinement as a reason of heterogenic crystallisation nuclei occurrence in aluminium alloys.

Phase morphology changes visible using light microscopy, can be detected also on the basis of the derivative curves as changes in the diagram form.

Analysing the interdependences between microstructure and phases or eutectics coming into existence during the crystallisation process, which are connected to the chemical composition, cooling rates and mechanical properties, a prediction of these properties is possible, of course with known chemical composition and registered technological parameters of the cast process. As a tool for this kind of predictions models can be applied which bases on artificial neural networks.

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