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Crystallisation kinetics of the Zn-Al alloys modified with lanthanum and cerium

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Materials

ABSTRACT

Purpose: The aim of the investigation is to determine the influence of modification on crystallisation kinetics on microstructure of the cast zinc alloy. This research work presents also the investigation results of derivative thermoanalysis performed using the UMSA device. The material used for investigation was the ZnAl8Cu1 alloy.
Design/methodology/approach: The UMSA device (Universal Metallurgical Simulator and Analyser) allows it to determine the characteristic points of the crystallised alloy including: determination of the influence of alloy modifiers, alloying additives, melting process parameters, cooling rate influence on phase and eutectics crystallisation of the investigated alloys. In was fund that cooling rate has an influence an microstructure and mechanical properties of the cast zinc alloys.
Findings: Crystallisation kinetics change makes it possible to produce materials with improved properties, which are obtained by: microstructure refinement and decrease or elimination of the segregation phenomenon.
Research limitations/implications: The material was examined metallographic and analysed qualitatively using light and scanning electron microscope as well as the area mapping and point-wise EDS microanalysis. The performed investigation are discussed for the reason of an possible improvement of thermal and structural properties of the alloy.
Practical implications: The investigated material can find its use in the foundry industry; an improvement of component quality depends mainly on better control over the production parameters.
Originality/value: value Investigation concerning the elaboration of optimal chemical composition and production method of zinc-aluminium alloys modified with chosen rare earths metals with enhanced properties compared to elements performed from traditional alloys and production methods, makes it possible to achieve a better understanding of mechanisms influencing improvement of mechanical properties of the new developed alloys.
Keywords: Metallic alloys; Thermo analysis; Zn-Al alloy; Microstructure

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1. Introduction

Progress in the field of technique make sit necessary to apply modern engineering materials with more and more improved mechanical properties. Parallel also the improvement of quality and production efficiency will be expected, for the reason of increasing market competitiveness, what makes it necessary to reduce production costs on the production level, but also on the level of quality control [1-6].

Cast zinc alloys hale very similar properties to aluminium or copper alloys. Differences are connected to a lover melting temperature and higher density [1,4,5].

Copper is the main alloying additive used for zinc alloys, which influence strength increase, hardness and corrosion resistance. Copper addition causes a shift of the eutectic point in the Zn-Al-Cu equilibrium diagram into the higher Al content [1,4,5].

Copper increases also the ageing liability of the Zn-Al. alloys and connected to this dimension changes. This has a low influence onto the eutectoid transformation of the β(β') phase and onto the solubility changes in the solid state in low temperature range. But first of all it influence the phase transformations In the solid state regarding the hexagonal η phase, face centred cubic phase β(β'), the hexagonal ε (CuZn4) phase and the hexagonal τ phase (intermetallic compound CuZn3) [1,8,9,10].

This transformation is the main reason for dimension changes of the material samples. For occurrence of the τ phase there is necessary a copper content of 0.6-0.7% in the alloy [1,6,7].

Magnesium is addend to the Zn-Al. alloy for the reason of limitation of the impurities influence, particularly Pb and Sn as well the limitation of intercrystalline corrosion. Addition of more than 0.1% mg deteriorates the castability, increases hot cracking and leads to casting cracks occurrence. In the ZnAl4Cu1 alloy a changeable content of Mg as well the modification additives Be, Ce and Ti cause Basic deference in change of dimensions during ageing [11-15].

Beryllium (Be) added to the alloy in amount of 0.005% increases the fatigue strength of the high pressure casts about 17-88%. Beryllium addition work also as an antioxidant which increases the oxidation resistance. In a range of 0.004-0.0045% Be Content in the ZnAl4Cu1 alloy there is remaining only ca. 25$ of the natural slag compared to alloy without beryllium addition.

This additive imitates also the diffusion of Fe coming from the crucible and furnace tools (made of steal or cast iron) [1-5].

In case of cast alloys the crystallization process proceeds in a range of temperature of the begin and end of the crystallization. The free energy value for the liquid and solid phase depend on the concentration of the second additive (in case of binary alloys).

The driving energy in case of alloys is the difference between the free energy of liquid and energy of the liquid and solid solution in a range of the second compound concentration for the liquid and solid phase [16-18].

Solidification can be characterised as linear or volume, In dependence of processes occurred on the crystallisation front. Crystallisation along a given direction is characterized by a constant boundary shift between the liquid and solid phase and with migration of the crystallization front from inside the outside of the cast (counterpart to the heat transfer direction). Volume crystallization is characterised by a lack of a boundary between the liquid and solid phase and heat transport from the crystallized phase also through the liquid phase (negative temperature gradient). Such a crystallization process can occur in opposite to the casting direction, but can also occur in a whole casting volume [1-6].

Crystallisation with a give direction can occur by precisely defined conditions. One of them tells us that in the crystallization front there can not occurs a high value of concentration overcooling. Directional crystallisation occurs in case of a huge temperature decrease in the cast cross sections what can be achieved by appliance of metal moulds, which are characterized by a high ability for heat accumulation [1,18-21].

Volume crystallization occurs in sand moulds, which shows a very low ability for heat accumulation. The occurred crystallization has entire a volume character and lead to occurrence of equiaxial grained structure. This a the most often occurred crystallisation form in case of cast alloys [1,22-25].

Cooling rate Has a big influence on microstructure and properties of the cast zinc alloy. Appliance of high cooling rates and in consequence of high crystallization rates causes: avoidance of segregation (blocky, dendritic), significant phase dispersion (among others a decreasing distance between the plates in eutectics) [1,6,7].

Cooling rate has a huge influence on the dendritic segregation: low cooling causes microstructure homogenisation and dendrite disappearance, by a cooling rate which is typical for a given alloy type instead of a grained microstructure a dendritic one occurs; achieving of a defined temperature leads to achieving of maximal dendritic segregation. By a very high cooling rate a fine-grained microstructure occurs when difference In the chemical composition of the particular grains is present [1-3,26-28].

The reason for appliance of high crystallization rate is to achieve materials with enhanced properties, which can be realised by dendritic microstructure or eutectic refinement, decrease or disappearance of segregation, occurrence of phases with enhanced solubility of the compounds or new metastable phases, phase morphology change [1,6,7,29-31].

Properties of the elements worked out from cast alloys depends on the primary alloy microstructure, which depends on the crystallisation kinetics. The crystallisation kinetics is characterised by changes of following parameters: metal temperature, cooling rate, rate of hidden crystallisation heat generating, grain density, which is equivalent to the density of nuclei, and to the fraction solid of the crystallised metal, concentration of the compounds of the remaining melt, characteristic distances and parameters describing the shape and size of the structure compounds [1-5,32-34].

All these parameters are variables of the crystallisation time and geometric coordinates of the cast.

Crystallisation of the liquid metal proceeds from the liquid state - the liquidus line, the begin of the crystallization, with following eutectics and intermetallic phases crystallization until the solid state - the solidus line, according to the phase equilibrium diagram. For this reason, on the cooling curve there are some characteristic points (inflexion points) indicating the exothermic and endothermic reactions of the crystallising phases and eutectics [1-7,35,36].

It is difficult to determine unequivocally the crystallization temperature of the particular phases. The temperature determination makes it possible to interpret the first derivative of the cooling curve in function of time, that means the differential curve (ATD) known also as the derivative curve [1,7,8].

For improvement of the mechanical properties of the cast alloys, despite the heat treatment also modification of the alloy is applied, which causes change of the morphology and decrease of the inter-phase distance of the α+β eutectic, as well as microstructure refinement. For this reason different kinds of modification additive are used [1,6-10].

At present there are used strontium and antimony as modifier, because thy have a long-term performance. The effect of strontium is present also after several remelting processes of the alloy, what makes it possible to produce alloys, which are modified already in foundries. Rare earths metals are also e applied more and more for modification of cast alloys.

Zn alloys, particularly with high amount of al have an inclination for shrinkage occurrence, what can be decrease using alloying additives like: Sr, Ca, Li, Na, Be; where the occurrence of Ti, Zr, Sb, B and rare earths metals is in this case of low significance. Rare earths metals decrease the castability and mould filling, they causes also like Ti the ability for hot cracking [6-10].

Improvement of the carried out chemical modification and appliance of a proper cooling of the casts leads to improvement of the mechanical properties of the produced casts. Therefore it is very important to have the knowledge about the microstructure modification of the casts according to the cooling rate or chemical composition change by addition of modifiers to the liquid metal [1,37,38].

As a result of the carried out investigations regarding the influence of rare earths metal addition is was found that deoxidation occurs during the melting process in the open air furnace, moreover also microstructure modification occurs as a result of addition of crystallisation nuclei injection and precipitation strengthening [7-11].

Rare earths metal addition make it possible to apply the produced material for elements working in elevated temperature, because it causes an improvement of the mechanical properties in the elevated temperature (more than 300°C) [1-6,39].

2. Materials and experimental procedure

For evaluation of the interdependence between the chemical composition and the structure of the ZnAl8Cu1 (PN-EN:1774) zinc cast alloy (Tables 1, 2), cooled with different cooling speed, following investigations were made [6-9]:

- Alloy structure using MEF4A optical microscope supplied by Leica together with the image analysis software as well electron scanning microscope using Zeiss Supra 25 device within high resolution mode. The samples for optical microscope investigations were etched using 10% HF solution.
- chemical composition of the investigated zinc alloy using qualitative and quantitative X-Ray analysis, as well EDS microanalysis,
- derivative thermo analysis using the UMSA thermo simulator,
- hardness measurements were performed using the Rockwell hardness tester supplied by Zwick ZHR 4150.

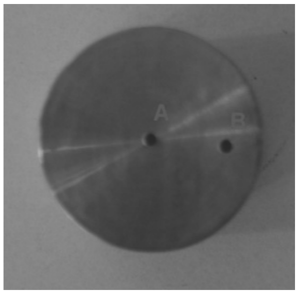
Investigation of lanthanum and cerium addition influence using the device for crystallisation process simulation UMSA was performed on cylindrical samples melted in graphite crucible (Fig. 1). On Fig. 1b there is presented the lay-out of the holes for thermocouples placement for temperature measurement during the heating and cooling process.

Table 1.
Chemical composition of ZnAl8Cu1 zinc alloy (PN-EN:1774)

Mass concentration of the elements, wt. %				
Al	Cu	Mg	Pb	Cd
3.2-8.8	0.9-1.3	0.02-0.03	max. 0.005	max. 0.005
Sn	Fe	Ni	Si	Zn
max. 0.002	max. 0.035	max. 0.001	max. 0.035	rest

a)

b)



c)

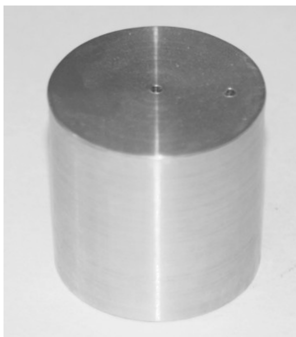


Fig. 1. Elements of the investigation configuration: a) graphite crucible, b) and c) sample

Table 2.
Chosen properties of the ZnAl8Cu1 alloy

Mass density	Tensile strength	Elongation	Hardness Brinell
6.7 kg/dcm^3	196 MPa	1%	65HB

For temperature measurement a chromel-alumel thermocouple of the K type was applied with a reaction time of 250 ms.

For the determination of the interdependence between the alloy crystallisation kinetics, chemical composition, micro-structure and mechanical properties of the cast zinc alloys rare earths metals were added, moreover cooling with different cooling rates was applied and following investigations were performed:

- thermo-derivative analysis of the investigated Zn alloys,
- macro- and microstructure of the alloys using light and scanning electron microscope with X-Ray microanalysis EDS,
- hardness of the modified Zn-Al alloys.

3. Description of achieved results

Preliminary investigations shows an advantageous influence of the cooling rate and modification of the Zn-Al alloys with lanthanum and cerium.

Addition of La and Ce into the ZL8 alloy causes modification of the microstructure (by mind of refinement of grains and subgrains, what is compatible with literature data), but it does not change significantly to derivative curve, because there are not any occurrence of new phases or eutectics found during the crystallisation process of the alloy (Figs. 2, 3).

As a result of the La and Ce modification occurs an increase of the ZL8 alloy overcooling, what is presented also on the fraction solid diagram (Fig. 4) calculated on the basis of the thermo-derivative analysis.

Table. 3.
Description of the characteristic points on the cooling curie from Figs. 2, 3

Point on the graph	Description
I	T_{DN} nucleation temperature
II	T temperature of the beginning of the crystal growth (α phase dendrites)
III	Present in liquid metal dendrites of the α phase are coherent, and the second derivative of the cooling curie Has the value zero
IV	Stable growth of the dendritic α phase
V	Nucleation of the β+α eutectic
VI	Stable growth of the β+α. Eutectic. This process occurs in constant temperature, so there exists an thermal equilibrium of the crystallised phases. In this point, the derivative of the cooling curve achieves the value zero for the second time
VII	T_{Sol}- end of the eutectic crystallisation, the alloy is completely crystallised

Crystallisation sequence of phases and eutectics marked on Figs. 2 and 3, for both the modified zinc alloy and after modification presented in Table 3.

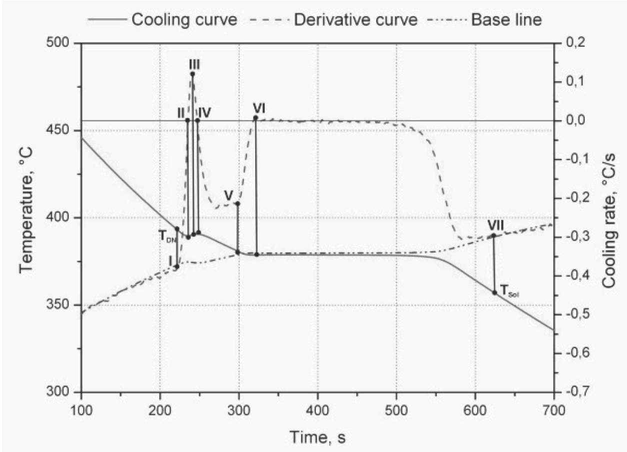


Fig. 2. Cooling curve, crystallisation curve and calorimetric analysis of the ATD diagram for the ZL8 alloy, cooling rate 0.09°C/s

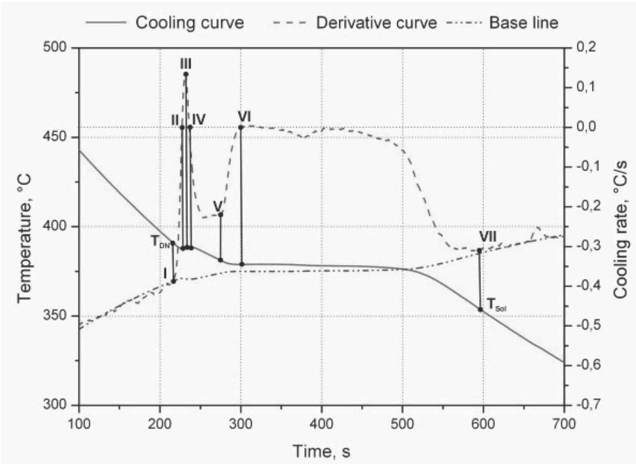


Fig. 3. Cooling curve, crystallisation curve and calorimetric analysis of the ATD diagram for the ZL8 alloy, modified with 0.5% (mass) La and 0.5% mass Ce cooling rate 0.15°C/s

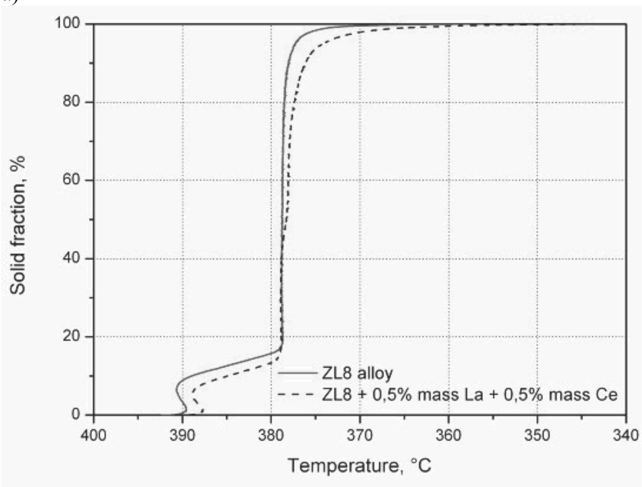
As a result of alloy modification, there occurs microstructure refinement of the cast ZL8 alloy, what is presented on Fig. 6.

There is also visible the change of Al dendrite morphology, which have extended secondary arms before modification (Figs. 5 and 6), whereas after modification there occurs an limited amount of dendrite secondary arms, and on its place comes into existence globular shaped precipitations (Figs. 7, 8).

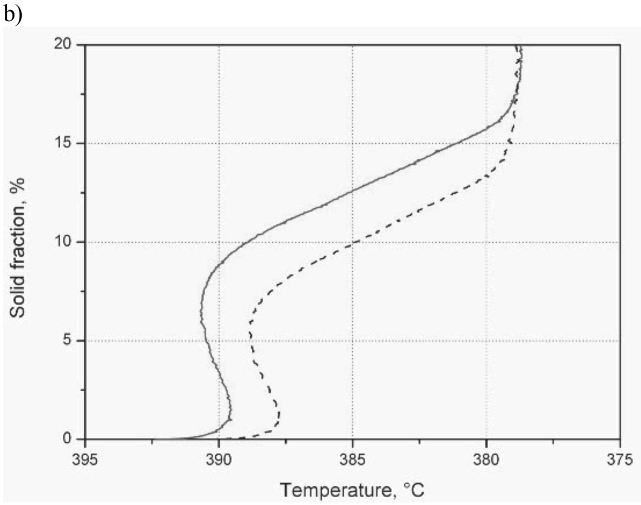
Modification of the investigated ZL8 alloy causes hardness increase. On Fig. 9 there is presented the hardness change for the non-modified as well as modified, freely cooled alloy.

The average hardness value calculated on the basis of particular investigation results, is characterized also by a lower standard deviation value for the ZL8 alloy modified with rare earths metals.

a)



b)



c)

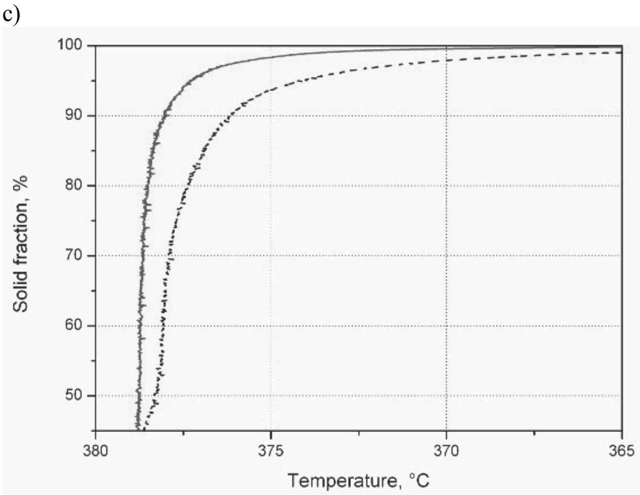


Fig. 4. Fraction solid diagram of the ZL8 alloy: a) entire diagram, b) and c) magnified part of the diagram from Fig. 4a

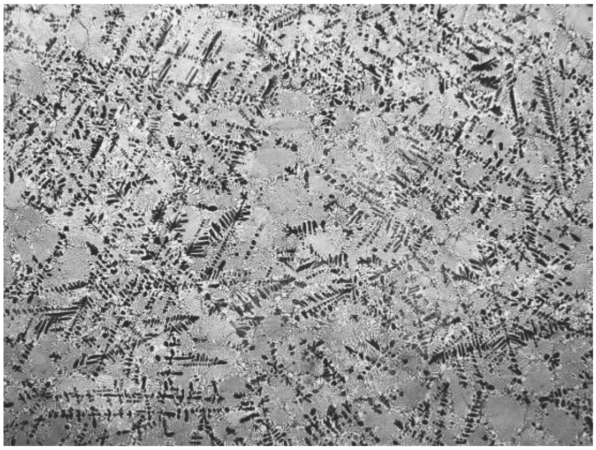


Fig. 5. Microstructure of the non-modified ZL8 cast alloy, etched in 10% HF, mag. 25x

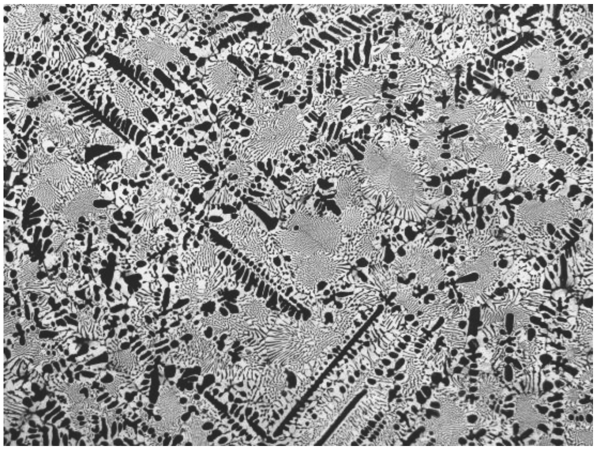


Fig. 6. Microstructure of the non-modified ZL8 cast alloy, etched in 10% HF, mag. 100x

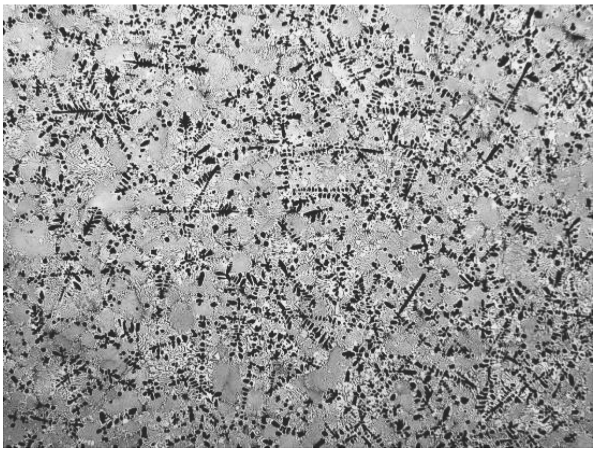


Fig. 7. Microstructure of the ZL8 cast alloy, modified with 0.5% (mass) La and 0.5% (mass) Ce etched in 10% HF, mag. 25x

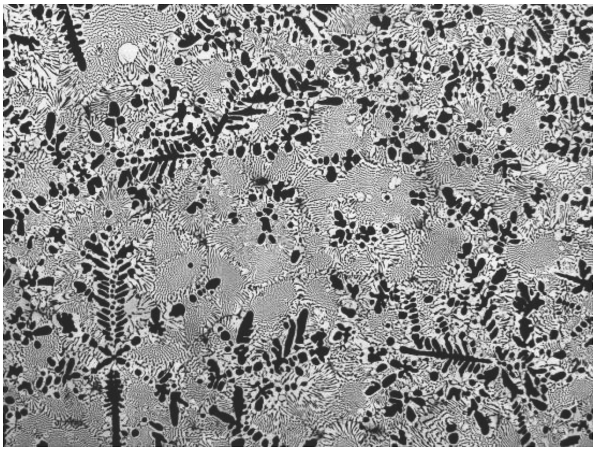


Fig. 8. Microstructure of the ZL8 cast alloy, modified with 0.5% (mass) La and 0.5% (mass) Ce etched in 10% HF, mag. 100x

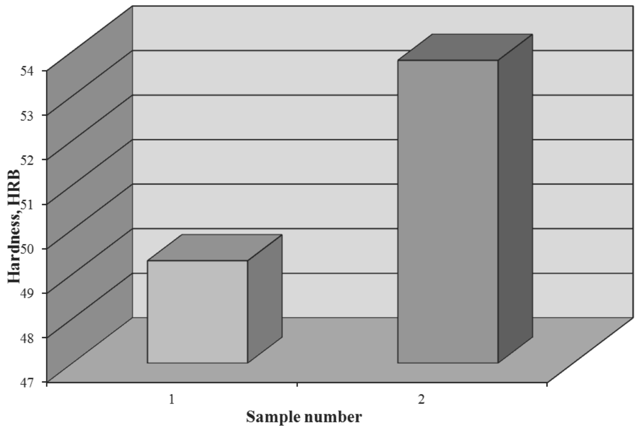


Fig. 9. Hardness measurement results of the ZL8 alloy: 1 - non-modified, 2 - modified with La and Ce

4. Conclusions

The investigated alloys were free cooled on air, with a chosen cooling rate in a range of time between T_{DN} and T_{Sol}. Simultaneously the addend amount of modifiers does not influence significantly the increase of individual production costs, what is undoubtedly an advantage.

Es a result of the performed investigations it was fund that: there occurs morphology changes of the α phase dendrites as well microstructure refinement, alloy modification causes a shift of the characteristic points of the phases and eutectics crystallisation as well solidus/liquidus points, and in case of La and Ce increase of the alloy overcooling, microstructure changes as result of chemical composition change causes hardness increase for freely, cooled samples compared to samples without alloying additives.

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