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TEM microstructure investigations of aluminium alloys used for laser alloying

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ABSTRACT

Purpose: In this paper there are presented results of Transmission Electron Microscope investigation concerning the structure of the AlSi7Cu4 cast aluminium alloy using for alloying and remelting with the high power diode laser (HPDL). There are also presented the results of the thermo-derivative analysis performed using the UMSA (Universal Metallurgical Simulator and Analyser) device, allowing to determine the specific points of the solidifying alloy, what is helpful for phase determination occurred in this alloy. In this work especially the changes of the precipitation type, size and shape were determined.

Design/methodology/approach: The investigations were performed using electron microscopy for the microstructure and phases determination. By mind of the transmission electron microscopy, especially selected area diffraction method appliance it was possible to determine the phases occurred in the alloy in the as cast state. The morphology and size of the Mg2Si was also possible to determine as well the lattice parameters for this phase.

Findings: : The reason of this work was also to present the laser treatment technology, which will be used for further alloying and remelting with ceramic powders – especially carbides and oxides. Particularly the overview will be directed on the laser power to achieve good layer hardness for protection of this hot work tool steel from losing their work stability and to make the tool surface more resistant to action in external conditions. The structure of the surface laser tray changes in a way, that there are very high roughness of the surface zone and the flatness or geometry changes in an important manner, crucial for further investigation.

Research limitations/implications: The aluminium samples were examined metallographically using transmission electron microscope with different image techniques.

Practical implications: Developing of new technology with appliance of Al alloys, High Power Diode Laser and diverse ceramic powders will be possible to obtain, based in findings from this research project. Some other investigation should be performed in the future, but the knowledge found in this research concerning the proper process parameters for each type of alloy shows an interesting investigation direction.

Originality/value: The combination of metallographic investigation for cast aluminium alloys - including electron microscope investigation - and HPDL treatment parameters makes the investigation very attractive for automobile, aviation industry, and others where aluminium alloys plays an important role.

Keywords: Laser surface treatment; Aluminium cast alloys; Laser alloying; Thermo-derivative analysis

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1. Introduction

Laser surface allying using HPDL laser is well established method of surface properties improvement for many materials including tool steels, magnesium alloys, titanium alloys, aluminium alloys and many others. Laser surface remelting was successfully applied to restore corrosion resistance in sensitized and in cold-worked and sensitised stainless steel. Further improvement of surface layer in respect of wear resistance can be obtained by alloying of stainless steel with hard particles such as carbides to locally reinforce the surface of steel. The surface hardening can be achieved with different manners - the incorporation of hard particles of TiC, WC, VC, SiC, WC and carbon alloying in order to form carbides as well as alloying with Si_3N_4 nitrides or oxides like Al_2O_3, ZrO_2 and borides and their mixes or elements such as Zn resulting in the modification of their resistance to pitting or intergranular corrosion and stress corrosion cracking [1-5].

Aluminium alloys are widely used structural materials group and particularly interesting due to their wide application possibilities and low weight as well a very good application properties and huge range of investigations. Strong interest in these alloys gives rise to the research aimed at improving the functional properties of these materials. Laser alloying and remelting was performed using high-power diode laser HPDL - High Power Diode Laser. The advantages of such a treatment process may include very high-speed heating and cooling, the possibility of applying a vacuum and protective atmosphere and melting process and the ability to process arbitrarily small selected areas causing purposeful changes in the surface structure of the surface of the element and reduce the cost of materials. The disadvantages of this technology include the high cost of the laser and the large surface roughness of machined material [6-12].

Rapid solidification process plays a major role in recent developments in this field, since it promotes a general refinement of the microstructure, extension of solubility of critical alloying elements in the Alα terminal solid solution and the formation of metastable phases, including metallic glasses and quasisrystals. Since these microstructural modifications are often responsible for an increase in the wear and corrosion resistance an high temperature strength of aluminium alloys, RS is being seriously considered for the development of high performance alloys. Among RS techniques, laser surface alloying (LSA) is particularly efficient for producing surface with improved ear and corrosion resistance an aluminium alloys, since it combines the controlled modification both of the microstructure and of the chemical composition to get surface properties to the application requirements (Almide, 1995, Yongquing, 1997) [13-16].

Cast aluminium alloys cannot be work hardened, so they are used in either the as-cast or heat-treated conditions. Common heat treatments include homogenization, annealing, solution treatment, aging and stress relief. Typical mechanical properties for commonly used casting alloys range from 138-345 MPa ultimate tensile strength and 103-276 MPa yield strength with up to 20% elongation (Yongquing, 1997). Surface processing using laser treatment can offer a great flexibility such as a minimal process time, reaching few tents of seconds, a minimum thermal distortion and good metallurgical bonding between the feeded ceramic powder and the substrate. The major concern for laser treatment is to avoid defects, such as cracks, bubbles, depressions and pinholes in the coating. The elimination of the above defects depends much on the optimization of laser parameters. For the inspection of defects in the laser treated coatings, light microscopy and electron microscopy are often used, in this investigations the transmission electron microscope was used for investigations of the structure before laser alloying and before powder feeding into the molten metal. The motion of the laser beam over the sample generates a track (see Fig. 1). Convective flows, generated by a temperature gradient between the centre of the beam and the sides of the melted pool, ensure the incorporation of the powder and the homogeneity of this surface alloy. In particular, this study presents the microstructural characterisation of Al-Si aluminium alloys laser alloying. Effects of annealing temperature upon the microstructure and upon the micro-hardness are investigated. In addition to the elastic modulus, the plasticity index and the wear behaviour also are studied as a function of the Si and Cu ratio in the Al-Si alloys [17-21].

A correct selection of these conditions provides the required characteristics of geometric and mechanical properties of the resulting surface layer. With the increase of the laser beam power density, or decreasing the scanning speed increases the thickness of the resulting layer, for lower power density laser beam scan speed or greater, the depth of alloying and, consequently, the resulting surface layer thickness decreases. These values should be within the proper range, because for a high power density laser beam or a low-speed scanning, alloy material begins to sublimate, leaving small pits on the surface. If the laser beam power density is too low or too high scanning speed, alloyed layer structure may be heterogeneous. What matters is the appropriate choice of alloying material to the ground, due to melting and sublimation, which for a homogeneous mix should be included in a narrow range of values. In fact, the plasma melting and evaporation occurs the material. A characteristic feature of the laser alloying process is the presence of the boundary layer and the substrate melted a large temperature gradient, which in turn leads to rapid cooling and solidification of molten metal. Achieved under these conditions the cooling rate to reach 1011 K/s, while the solidification velocity often exceed 20 m/s, which in the case of some materials may cause quenching of thin layer of substrate material. Plasma affects the casting of two ways, on the one hand, it shields the lake from further absorption of energy from the laser beam, which inhibits sublimation, on the other hand, by its own pressure leads to mixing of the molten components. The laser beam will also rise to funnel-like depressions in the lake of molten metal, which is ionized gas, and liquid metal at the border - plasma is maintained constantly disturbed, unstable equilibrium. In order to regulate the impact of plasma on the lake of molten metal, various technological methods of activation or levelling. One method of limiting the influence of plasma on a lake of molten metal by blowing a cloud of plasma stream of inert gas. Introduced gas (e.g. argon) is often additionally heated, which prevents deterioration of the energy effect. While intensifying the effect of plasma is carried out using a plasma cloud blowing, but at the same time re-directing to the work area primarily reflected laser radiation by a system of flat mirrors or mirror dish [18-22].

Table 1.
Properties of the ceramic powders using for alloying

Properties	WC	VC	TiC	SiC	Al_2O_3
Density, kg/m^3	15.69	5.36	4.25	3.44	3.97
Hardness, HV	3400	2850	1550	1600	2300
Melting temperature, °C	2870	2830	3140	1900	2047

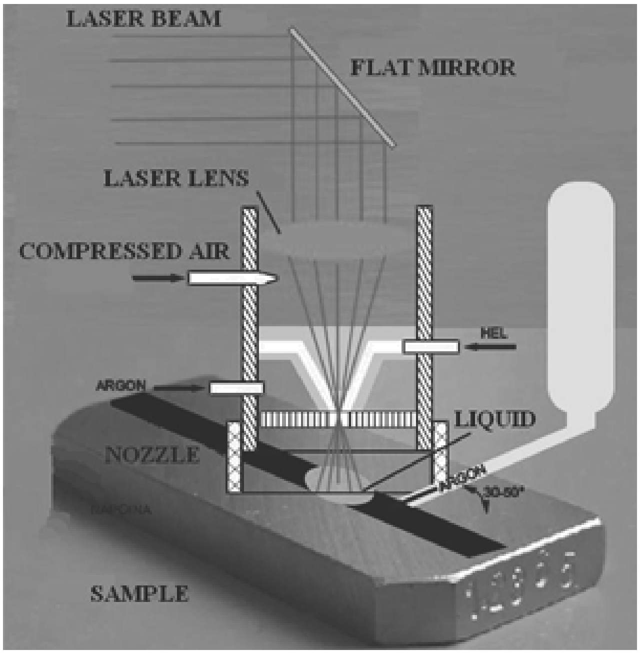

Fig. 1. Scheme of the laser alloying process performed on the investigated AlSi7Cu4 alloy

This paper presents the results of structural investigations performed on transmission electron microscope of the $AlSi_7Cu_4$ aluminium alloy before alloying with ceramic powders including titanium carbide, vanadium carbide, tungsten carbide, silicon carbide and aluminium oxide. The main objective of this investigations was also conducted to determine the effect of heat treatment conditions on the structure of the substrate material, corresponding to the matrix and the morphology of the phases present in the aluminium alloys investigated. Alloying and laser remelting will be carried out using modern high-power laser HPDL (High Power Laser Diode). Based on the obtained results structural changes were found in particular by structure and phase fragmentation, as well changes in hardness compared to as cast aluminium. In the next research step there will be compared the influence of laser power, as well as ceramic powders used to improve the properties of the resulting composite material, in particular on the hardness of the surface layer. The aim of the present work is also to highlight the advantage of using TEM imaging in the optimization of laser parameters for laser alloying of aluminium alloy substrates [20-26].

Laser treatment is presented based on remelting of $AlSi_7Cu_4$ cast aluminium alloy. The basic laser treatment parameters is the practical aim of this work, as well as improvement of hardness. Special attention was devoted to monitoring of the surface layer morphology of the investigated material and on the laser tray quality - the roughness, flatness, width and porosity.

2. Experimental procedure

2.1. Substrate material

The aluminium alloy - $AlSi_7Cu_4$ was used for investigations with the chemical compositions presented in Table 2. The investigated aluminium alloy was supplied in form of moulds, without any heat treatment carried out.

Table 2.
Chemical composition of the investigated Al alloy

Chemical composition of the investigated alloys, in mass %	
Elements	$AlSi_7Cu_4$
Si	7.45
Fe	0.17
Cu	3.6
Mn	0.25
Mg	0.28
Zn	0.05
Ti	0.13
Al	87.97
Rest	0.1

2.2. Heat treatment

Heat treating is a crucial step in the aluminium manufacturing process to achieve optimal properties. The heat treatment of aluminium alloys requires precise control of the time-temperature profile, which was ensured by use of the K-typ thermocouples, as well tight temperature uniformity. The applied heat treatment consists of processes including solution heat treatment and ageing with parameters such as times, temperatures presented in Table 3 and quenching agents in form of water and air at room temperature.

Table 3.
Heat treatment parameters applied for the investigated aluminium alloys

Alloy	Solution heat treatment		Ageing	
	Time [min]	Temperature, [°C]	Time [min]	Temperature, [°C]
$AlSi_7Cu_4$	600	505	720	170

The goal of aging is to cause precipitation dispersion of the alloy solute to occur. The degree of stable equilibrium achieved for a given grade is a function of both time and temperature. In order to achieve this, the microstructure must recover from an unstable or “metastable” condition produced by solution treating and quenching or by cold working. Whereas the purpose of solution heat treatment is the dissolution of the maximum amount of soluble elements from the alloy into solid solution. The process consists of heating and holding the alloy at a temperature sufficiently high and for a long enough period of time to achieve a nearly homogenous solid solution in which all phases have dissolved.

The heat treatment presented on Fig. 2 was carried out in the electric resistance furnace U117, with a heating rate of 80°C/s for the ageing process and 300°C/s for the solution heat treatment process with two holds at 300°C and 450°C performer for 15 minutes. Cooling of the samples after heat treatment was performed in air for the ageing process and in water for the solution heat treatment process. The solution heat treatment temperature was 505°C for 10 hours, and then ageing was performed at 175°C for 12 hours.

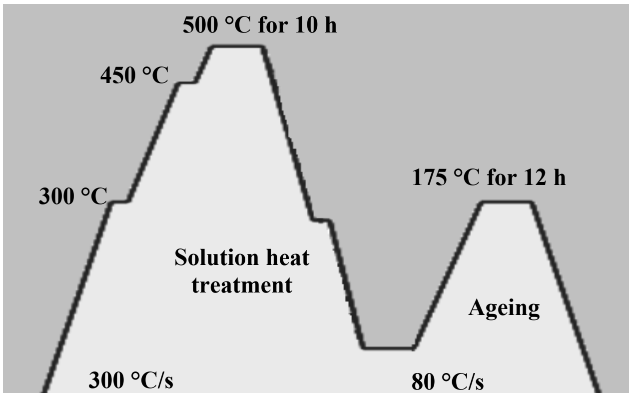

Fig. 2. Heat treatment parameters used for the investigated aluminium cast alloys

2.3. Remelting and alloying process

For remelting it was using the high power diode laser HPDL Rofin DL 020. The used laser is a device with high power, used in materials science, including for welding. The laser equipment used included such as: rotary table and moving in the XY plane, the nozzle of the powder feeder to the enrichment or welding, shielding gas nozzle, laser head, power and cooling system, and the computer system controlling the operation and location of the laser the working table.

Remelting was performed in argon, in order to protect the substrate from oxidation. The sample was subjected to laser fusing the protective gas blowing the cover of the two nozzles, one directed axially to the laser-treated sample and the other directed perpendicular to the weld area. Flow rate of shielding gas (Argon 5.0) was 10 l/min. Nozzle distance from the sample was set in the range of ca. 20 mm. On one surface of the rectangular samples was performed by one track by fusing laser at different laser power and at a laser scan rat of 0.25 m/s.

2.4. Thermo-derivative analysis

Investigation of the influence heat treatment conditions were performed using the UMSA device for simulation of crystallization processes on samples melted in a cylindrical graphite crucible. The UMSA device used for investigations is designed to overcome the existing problem of laboratory and industrial equipment on the present market. This platform combines computer controlled melting and heat treatment devices with a quench equipment, as well the device for thermal analysis and testing equipment for in-situ investigations of test sample crystallization characteristics. The UMSA device is used mostly in the following areas:

- development of new materials and processes as well in quality control process,
- analysis of phase nucleation, crystallization and growth, phase transformations during melting, solidification,
- heat treatment performed under assumed conditions,
- physical simulation of metal casting technologies including melting, chemical and thermal treatment, solidification and heat treatment operations with operations like solution heat treatment, aging and quenching,
- analysis of structure changes of the test sample material according to quenching at determined temperatures.

2.5. Sample preparation

The examinations of thin foils microstructure and phase identification were carried out on the JEOL 3010 transmission electron microscope (TEM), at the accelerating voltage of 300 kV using selected area diffraction method (SAD) for phase investigations. This microscope has a "cold finger" device which permits the observation of the sample at liquid nitrogen temperature (73 K) and reduces the contamination process of the sample. The diffraction patterns from the TEM were solved using a special computer program "Eldyf" software supplied by the Institute of Material Science o the University of Silesia.

TEM specimens were prepared by cutting thin plates from the material. The specimens were ground down to foils with a maximum thickness of 80 μm before 3 mm diameter discs were punched from the specimens. The disks were further thinned by ion milling method with the Precision Ion Polishing System (PIPS™), used the ion milling device model 691 supplied by Gatan until one or more holes appeared. The ion milling was done with argon ions, accelerated by a voltage of 15 kV, energy and angle are presented in Table 4.

Table 4.
Ion milling parameters using for polishing

Angle [°]	Energy [KeV]	Time [min]
6	3.8	180
3	3.2	15

2.6. Investigation methodology

As a result, the proper selection of feather conditions can be achieved on the surface, a single composite matrix consisting of (Al alloy). In determining the conditions of the process should take into account several important factors, including the following: the beam energy, absorption differences between the cast of aluminium alloys, laser scan rate.

The micrographs of the micro- and macrostructure investigation was performed using the light microscope Leica MEF4A supplied by Zeiss in a magnification range of 50-500x. The micrographs of the microstructures were made by means of the KS 300 program using the digital camera equipped with a special image software.

Microstructure investigation and phase identification was performed using transmission electron microscopy (TEM) JEOL JEM 3010 TEM with the bright and dark field image technique and SAD diffraction method. The diffraction pattern calculation was performed using the "Eldyf" software supplied by the Institute of Material Science of the University of Silesia.

The hardness was measured with Rockwell hardness tester with a chosen load of 60 kGf in the HRF scale, according to the PN-EN ISO 6507-1 standard. A minimum of 3 indentations was made on each of the tested samples.

3. Results and discussion

The material base alloys reveals a similar $AlSi_7Cu_4$ solid solution with hardness values of ca. 70 HRF for the as cast alloy. After the heat treatment applied to hardness increase to ca 90 HRF (Table 6), caused mainly the occurrence of the Al_2Cu eutectics, as well the iron and manganese containing phases(s) visible on the crystallisation curve and the performed calorimetric analysis of the ATD diagram - especially in point 5 on Fig. 9. On Fig. 10 so called fraction solid diagram, there is presented the percentage measure of how much of a sample is solid within the semi-solid region. It is 0% at the liquidus temperature and 100% at the solidus temperature.

This data can be used for determination of the volume fraction of phases formed diring the solidification process.

TEM investigation results are presented on Figures 3-8. The analysis of thin foils after the process of ageing has validated the fact that the structure of the aluminium cast alloy consists of the solid solution α - Al (matrix) and intermetallic secondary phases in the form of bulk precipitations.

The presence of the cubic Al_2Cu phase was confirmed by selected area diffraction patterns (Fig. 8).

This phase was distinguished as a precipitates of: roughly spherical particles types, of approximate size 0.2-0.4 μm in diameter, in literature there is also mentioned the Mg-Si particles, of size 2 mm, adjacent to the Al-Cu particles.

Moreover, the examinations of the thin aluminium cast alloy foils after the ageing process confirm the existence of a high density of crystal structure defects identified as a series of straight and parallel dislocations resembling a network (Figs. 3 to 4). The casting and next heat treatment process has caused also the precipitation of evenly distributed dispersive α Si phase in the needle form that has in the major performed investigations a preferred crystallographic orientation in the matrix.

The tested material $AlSi_7Cu_4$ cast aluminium alloys in as cast state and heat treated base material, applied for laser alloying have a microstructure consisting of matrix in form of Al-Si solid solution with eutectic (Si) and primary hard phases (Table 5).

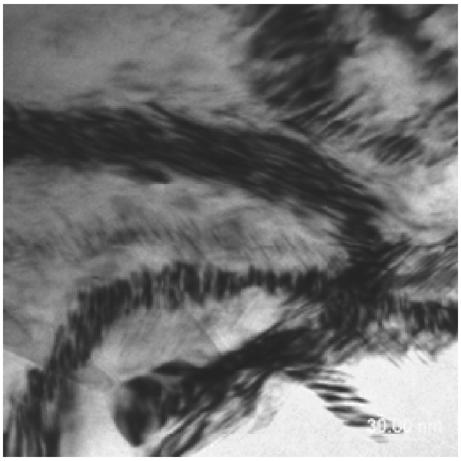

Fig. 3. Structure of the Alα phase, bright field

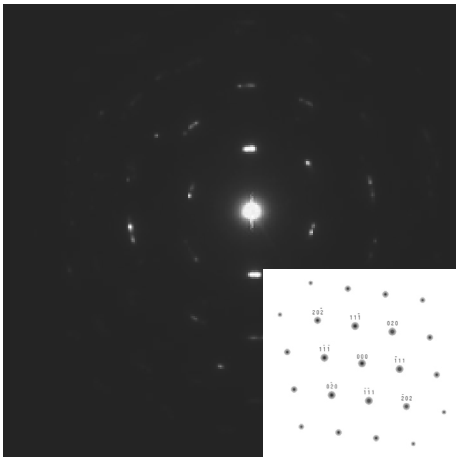

Fig. 4. Diffraction pattern of the Al α phase in Fig. 2 zone axis, [-10-1]

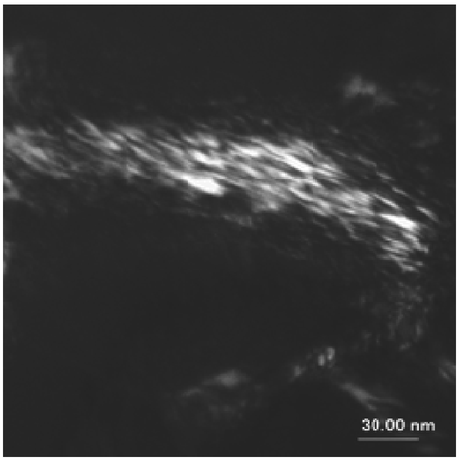

Fig. 5. Structure of the Alα phase, dark field

With following phase transition points on Fig. 9:

I. point of beginning of the α phase dendrites nucleation,
II. point, where the nuclei reach the critical value and where the α phase dendrite growth is continued. From this point increases the hidden heat amount of the α phase dendrites crystallization process,
III. point, where the α phase dendrites occurred in the liquid alloy becomes coherent,
IV. point, where a stable growth of the α phase dendrites is achieved,
V. point, where the precipitations of the Fe and Mn phase crystallises.
VI. point, where the nucleation of the +β eutectics begin,
VII. point, where the created nuclei of the β phase (of the α+β eutectics) reach the critical value and they grow together with the α phase creating the α+β eutectics,
VIII. point, where a stable growth of the α+β eutectics occurs. This process goes on in a constant temperature, so there exist an equilibrium of heat between the crystallised phases,
IX. point, where the crystallisation of the three-components eutectics begin,
X. point, where the crystallisation of the Cu, Mg rich precipitation ends and the crystallisation of multifunctional eutectic begins,
XI. point, where the crystallisation of the multifunctional eutectic ends and the alloy is entire crystallised.

Table 5.
Phase occurred in the investigated material

Alloy	Eutectic hard phases	Primary Hard phases
$AlSi_7Cu_4$	Si, Al_2Cu, Mg_2Si	Si

Table 6.
Hardness of the investigated aluminium alloys

Hardness of the investigated aluminium alloys, HRF		
alloy	As cast state	After heat treatment
$AlSi_7Cu_4$	67	91

4. Conclusions

The analysis of the thin foils after the ageing process has confirmed that the structure of the aluminium cast alloy consists of the solid solution α - Mg (matrix) with embedded secondary phase Al_2Cu evenly distributed in the structure. Furthermore, the examinations of thin foils of aluminium cast alloys after ageing confirm the appearance of a high density of defects of the crystal structure in the material (Figs. 3-8). It can be also found that:

- The measured hardness increases compared to the as cast state of ca. 35%.
- As the most often occurred phase, despite to Si needle, the Al_2Cu precipitations were found using transmission electron microscope.

Thermo-derivative analysis makes it possible to found thermal points, where another type of phases crystallises - point 5 on Fig. 8, probably Fe and Mn containing phases.

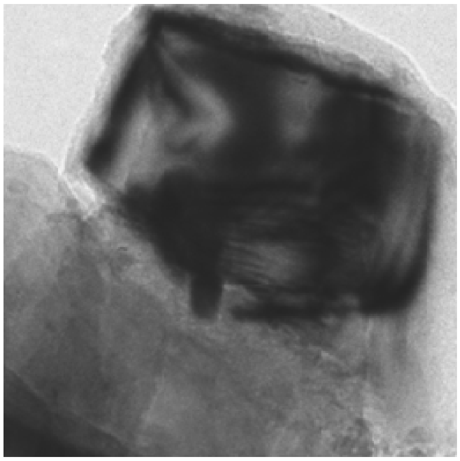

Fig. 6. Al_2Cu phase in the Al matrix, bright field

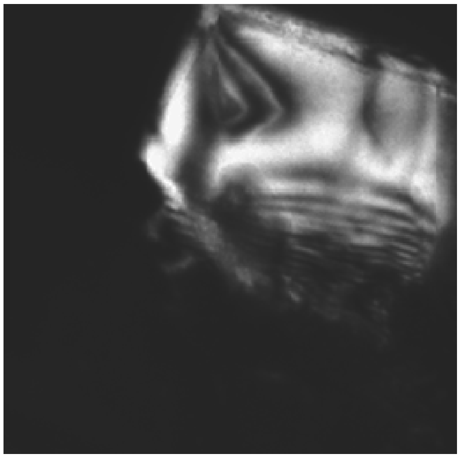

Fig. 7. Al_2Cu phase in the Al matrix, dark field

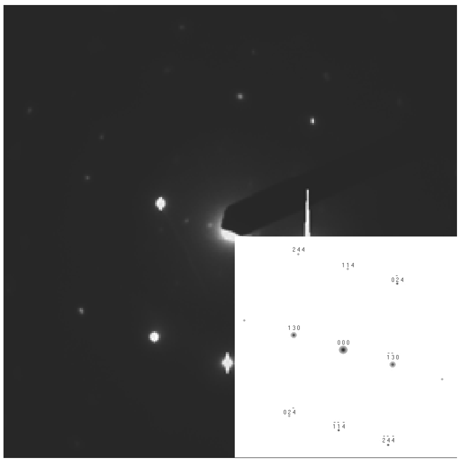

Fig. 8. Diffraction pattern of the Al_2Cu phase in Fig. 5, zone axis [-621]

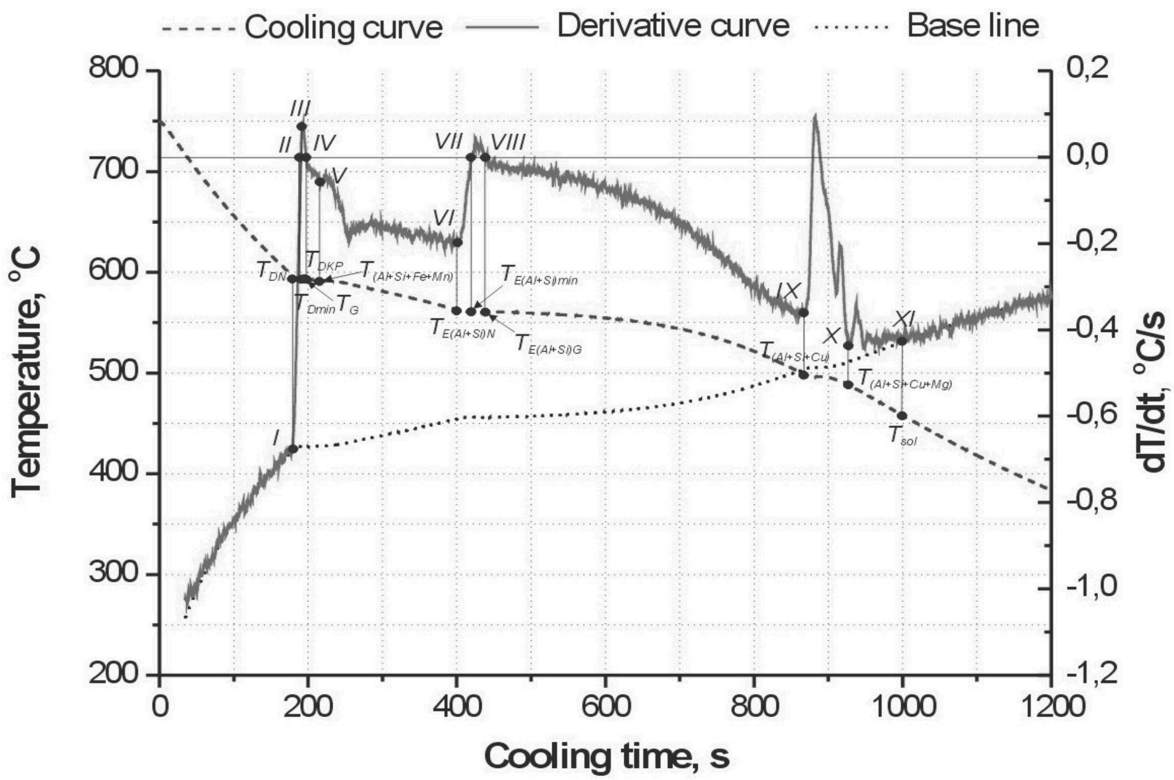

Fig. 9. Cooling and crystallisation curve and the calorimetric analysis of the ATD diagram for the AC $AlSi_7Cu_4$ alloy, cooling rate 0.2°C/s

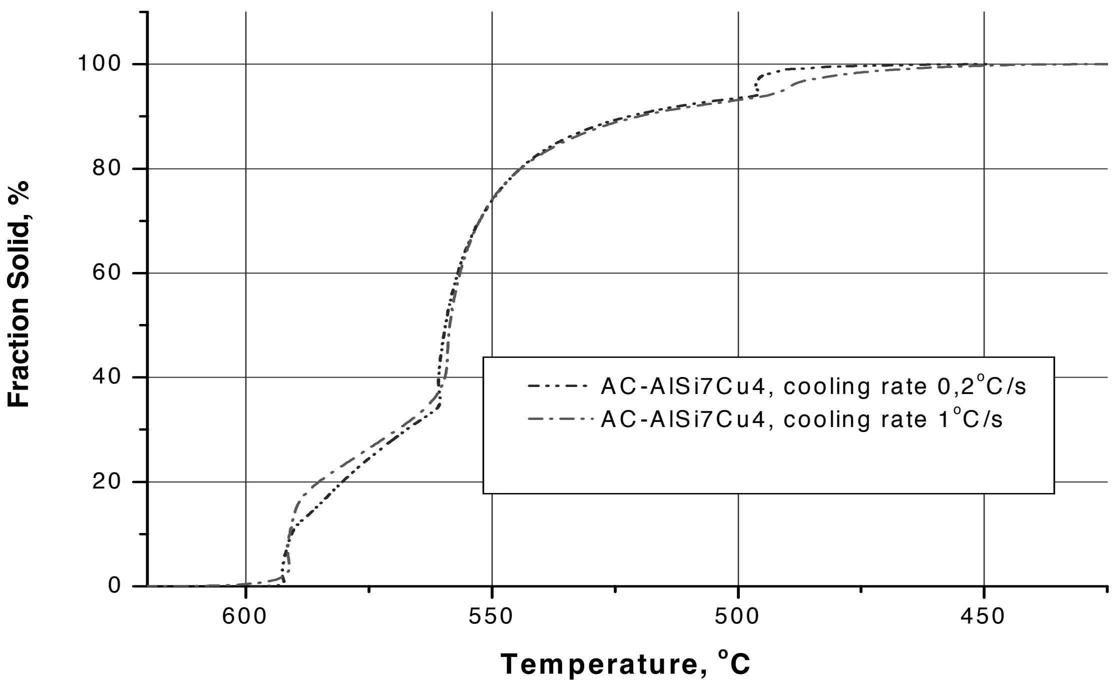

Fig. 10. Fraction solid of the $AlSi_7Cu_4$ alloy

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