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Thermal-Derivative Analysis and Precipitation Hardening of the Hypoeutectic Al-Si-Cu Alloys

P.E. Smolarczyk *, M. Krupiński
Silesian University of Technology, Institute of Engineering Materials and Biomaterials
Konarskiego 18a, 44-100 Gliwice, Poland
* Corresponding author. E-mail: paulina.smolarczyk@polsl.pl

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Abstract

The research focused on the influence of the solution temperature on the structure of precipitation hardening multi-component hypoeutectic aluminium alloys. The AlSi8Cu3 and AlSi6Cu4 alloys were used in the study and were subjected to a thermal-derivative analysis. The chemical composition and crystallization of phases and eutectics shift the characteristic points and the corresponding temperatures to other values, which affect to, for instance, the solution temperature. The alloys were supersaturated at 475°C (according to the determined temperature (T_{Sol}) and 505°C for 1.5 hours. Aging was performed at 180°C for 5 hours. The Rockwell hardness measurement, metallographic analysis of alloys by means of light microscopy as well as chemical and phase analysis using scanning electron microscopy and X-ray crystallography were carried out on alloys. The use of computer image analysis enabled the determination of the amount of the current Al_2Cu phase in the alloys before and after heat treatment.

Keywords: Thrermal-derivative analysis, Heat treatment, Hardness, Metallographic analysis, Al-Si-Cu alloys

1. Introduction

Depending on the chemical content and aging parameters, the individual process is determined for the alloy. This means that the sequence and number of phases may vary [1].

The process of supersaturation, according to the literature, should take place at a temperature below the temperature of the crystallization end (T_{sol}.) of the alloy. Raising the said temperature may cause partial or complete dissolution of multi component eutectics. In this case, instead of precipitation hardening, it is be possible to observe solution hardening [2].

During the aluminum alloys precipitation hardening a homogeneous solution is obtained. After applying the appropriate cooling rate, the solution is entering the state of metastability, which leads to the separation of alloy components. During aging, phase characterized by the dispersion, is released from the supersaturated solid solution [3].

Inside the aluminum alloys, hard and brittle intermetallic phases are formed. In the tested multi-component Al-Si-Cu alloys, the characteristic phases are: aluminum (α), silicon (β), aluminum with copper (Al_2Cu), magnesium with silicon (Mg_2Si) and the phase containing iron and manganese (Al_5SiFe, $Al_8FeMg_3Si_6$ or $Al_{15}(Fe, Mn)_3Si_2$ [4].

The strengthening of the solution in aluminum alloys is influenced by the amount and type of elements that are dissolved inside of it. A choice of the solution temperature above the T_{sol} temperature will cause partial or complete dissolution of some components. In the case of multicomponent aluminum alloys Al-Si-Cu (Fig. 1) [5], it leads to a dissolution of greater amount of copper than it results from the phase equilibrium system of the two-component aluminum alloy Al-Cu (Fig. 2) [6].

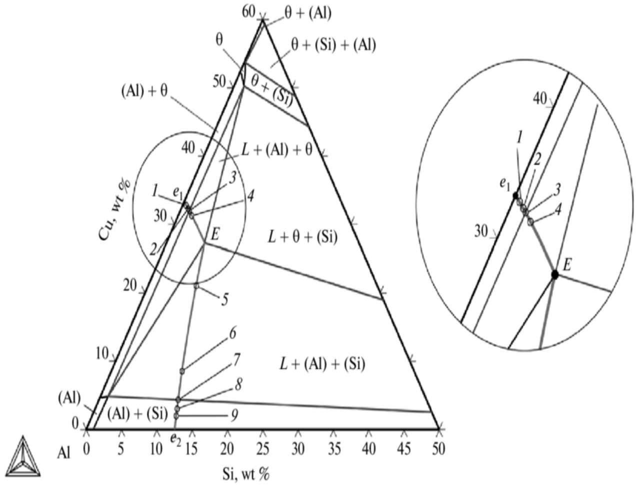

Fig. 1. Isothermal cross section of the Al–Si–Cu phase diagram at 525°C with superimposed eutectic transformation polytherms L + (Al) + (Si) and L + (Al) + θ [5]

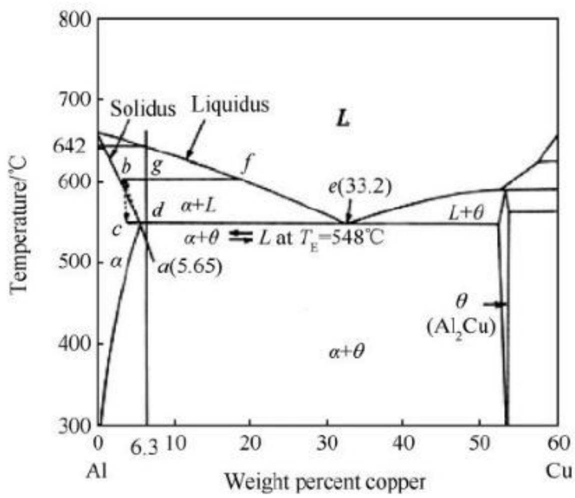

Fig. 2. Part of phase diagram of Al-Cu alloy [6]

2. Materials and experiments procedure

For analysis of an effect of the solution temperature on the structure, multi-component Al-Si-Cu alloys were researched with the chemical composition shown in Table 1, chemical analysis was carried out on ARL 3460 Emission Spectrometer Thermo Fisher Scientific .

During the experiment, the UMSA MT5 device was used to determine the start temperature ($T_{liq.}$) and end temperature ($T_{sol.}$) of the crystallization of the tested alloys. The samples were heated to a temperature of 750 °C, kept in it for 180 seconds and then cooled, cooling rate was 0,15°C/s . The samples had the shape of a cylinder with following dimensions: height h = 40 mm, diameter d = 40 mm. The K type thermocouple was located in the sample axis at the height of h_t = 20 mm. The aluminum alloys were heated using an induction coil.

Table 1.
The chemical composition of tested alloys.

Element	Mass concentration of elements, [%]	
	AlSi6Cu4 alloy	AlSi8Cu3 alloy
Si	5,82	8,32
Fe	0,43	0,33
Cu	4,30	3,05
Mn	0,48	0,46
Mg	0,28	0,24
Cr	0,04	-
Ni	0,02	0,02
Zn	0,64	0,20
Pb	0,02	0,01
Sn	0,01	0,01
Ti	0,15	0,15
Other	<0,35	0,25

As part of the research, the Rockwell hardness measurement was carried out at a load of 60 kG using a ball-shaped indenter in accordance with the PN-EN ISO 6508-1: 2016-10 standard on the Zwick ZHR 4150 TK device.

The metallographic analysis was performed using light microscopy on the Axio Observer of ZEISS device. The alloys were etched into two reagents - 48% HBF4 (digestion time 90 s) and 20% NaOH (digestion time 120 s). Observations of the performed defects were carried out in a bright field and in a polarized light.

The scanning microscope ZEISS SUPRA 35 was used to study the microstructure using the secondary electron method. The EDS technique was used for quantitative and qualitative analysis in micro-areas.

The alloys were subjected to X-ray structural examination using the Panalytical MPD X'Pert Pro device with a cobalt cathode. The reason for it was to obtain a diffraction pattern, allowing identification of a structural components and the alignment of the Al matrix.

The images obtained during metallographic studies were subjected to a computer image analysis using the Image-Pro 10 program. As a result, the changes in the amount of strengthening phase Al_2Cu, were determined in both alloys, after saturation at the different temperatures.

3. Results

As a result of the thermal-derivative analysis of the obtained crystallization graphs of the alloys tested (Figure 3,4), the temperature of the beginning and the end of the crystallization was read.

In the studied alloys, multi-component $E_{(Al + Si + Cu + Mg)}$ begins to solidify at point II and ends at point III, which is also the end point of alloy crystallization. For the AlSi6Cu4 alloy, the beginning temperature for the multicomponent eutectic α+Al_2Cu+β is 504 °C and the ending is 488 °C, while for the AlSi8Cu3 alloy the temperature of the beginning of the crystallization is 504°C and the ending is 490 °C.

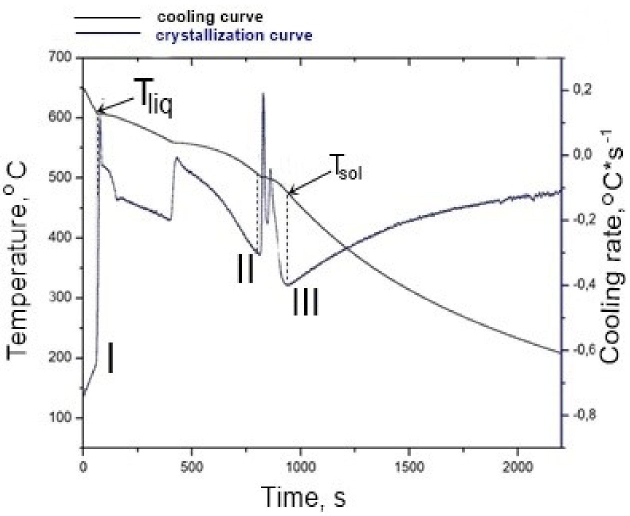

Fig. 3. Cooling curve and derivative curve for AlSi6Cu4 alloy

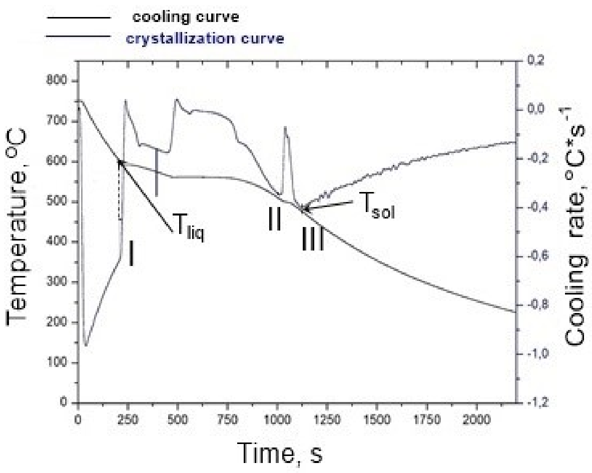

Fig. 4. Cooling curve and derivative curve for AlSi8Cu3 alloy

Table 2 shows the temperature of the beginning and the ending of crystallization for the tested alloys, read from the graphs generated during the thermal-derivative analysis.

Table 2.
The temperature of the beginning (T_{liq}) and the ending of crystallization (T_{sol}) of the alloys tested

	Temperature of the beginning of crystallization, $T_{liq.}$ [°C]	Temperature of the ending of crystallization, $T_{sol.}$ [°C]
AlSi6Cu4 alloy	612	488
AlSi8Cu3 alloy	604	490

The average hardness results obtained during Rockwell measurements are shown in Table 3.

Table 3.
The results of Rockwell hardness in the HRF scale

	AlSi6Cu4 alloy		AlSi8Cu3 alloy	
	Cooled at 0.15 ° C / s.	σ	Cooled at 0.15 ° C / s.	σ
DTA	76,85	1,06	80,19	5,64
Solution temp. 475°C	76,41	3,35	42,69	8,67
Solution temp. 475°C and artificial ageing	87,61	6,79	85,19	5,92
Solution temp. 505°C	93,19	3,05	88,06	2,07
Solution temp. 505°C and artificial ageing	85,9	9,1	95,28	1,33

In both alloys, after solutioning at 505°C, an increase of the hardness of the AlSi6Cu4 alloy by approximately 20% and for the AlSi8Cu3 alloy by approximately 9%, was noticed. The obtained results indicate that at 475°C, precipitation hardening occurred for both alloys, while at 505 ° C (above $T_{sol.}$), an increase in hardness without aging is visible, which indicates the solution hardening of the alloy.

Performing X-ray structural analysis allowed to identify the existing Al, Si and Al_2Cu phases in the tested alloys. In addition, a change in the parameter of the distance between atoms in alloys was observed (Table 4).

Table 4.
The value of the distance of the network parameter depending on the state of the alloys

	AlSi6Cu4 [Å]	AlSi8Cu3 [Å]
DTA	2,33605	2,33853
Solution 475°C	2,33625	2,33820
Solution 505°C	2,33730	2,34134

The metallographic analysis of AlSi6Cu4 alloy (Fig.3) indicates a morphology - β phase (point #1), Al_2Cu phase (point #2) and $Al_{15}(Fe_3Mn)_2$ phase (point #3) [9-11,13].

Figures 5-10 obtained as a result of light microscopy were subjected to computer image analysis, which allowed to determine changes in the amount of Al_2Cu phase in both alloys (Table 5, 6) after saturation at 475°C and 505°C. The AlSi6Cu4 alloy shows fragmentation after supersaturation at both temperatures, while the lowest value of the average phase separation of Al_2Cu in the alloy was noticed after supersaturation at 505°C (tab. 5). The AlSi8Cu3 alloy also shows the microstructure fragmentation after the solution process. The lowest percentage content of the Al_2Cu phase was noted for the AlSi8Cu3 alloy after supersaturation at 505°C (tab. 6), indicating the maximum dissolution of copper in the α-phase.

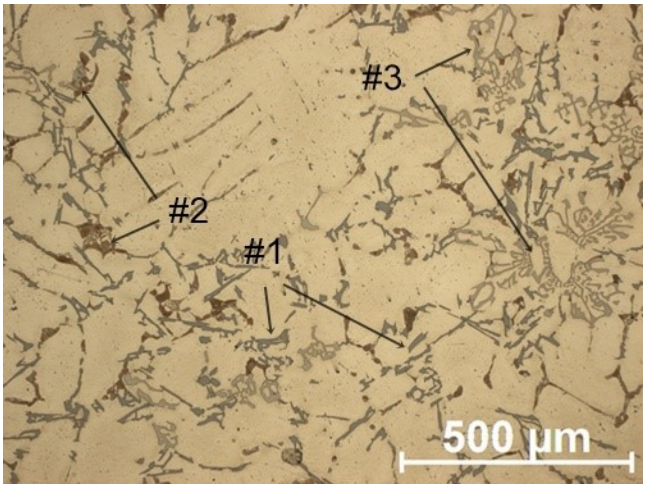

Fig. 5. AlSi6Cu4 alloy after thermal-deriavate analysis.
#1 - β phase , #2 – Al_2Cu , #3 - $Al_{15}(Fe_3Mn)_2$ phase

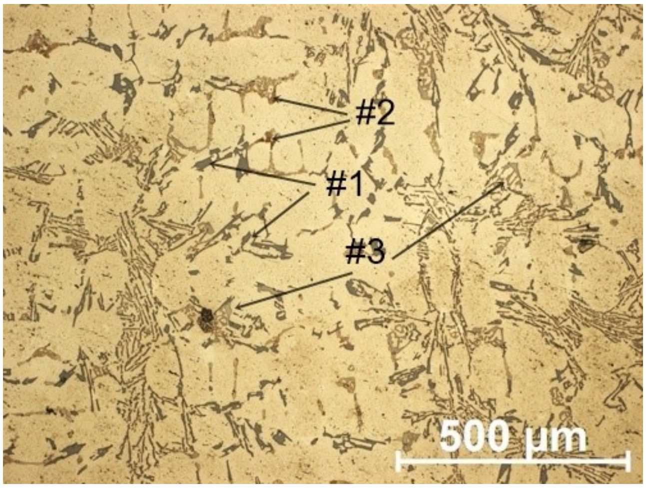

Fig. 6. AlSi6Cu4 alloy after solution in 475°C

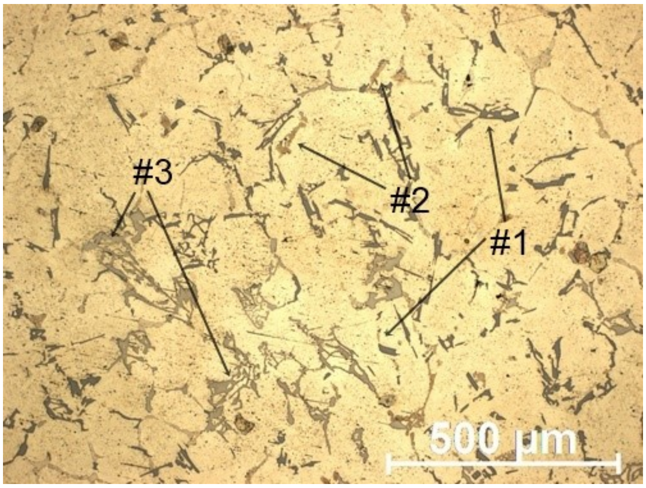

Fig. 7. AlSi6Cu4 alloy after solution in 505°C

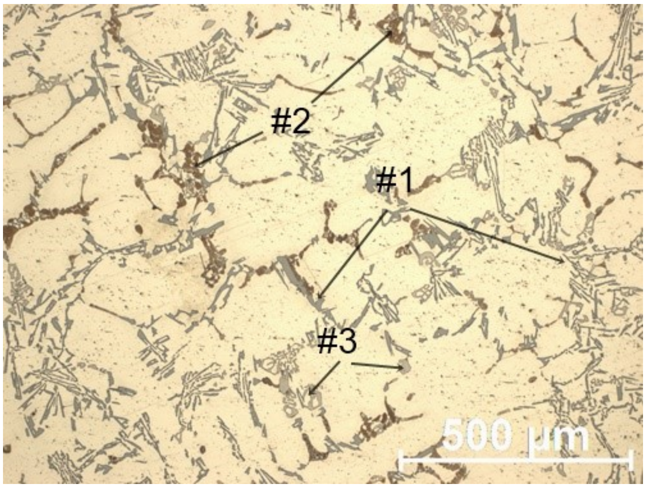

Fig. 8. AlSi8Cu3 alloy after thermal-deriavate analysis.
#1 - β phase , #2 – Al_2Cu , #3 - $Al_{15}(Fe_3Mn)_2$ phase

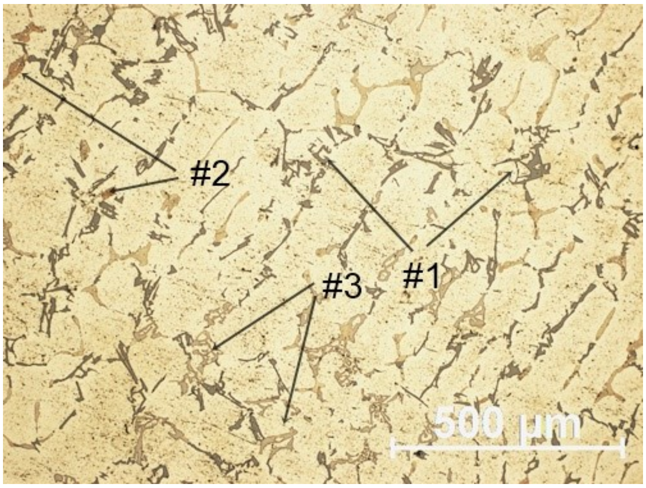

Fig. 9. AlSi8Cu3 alloy after solution in 475°C

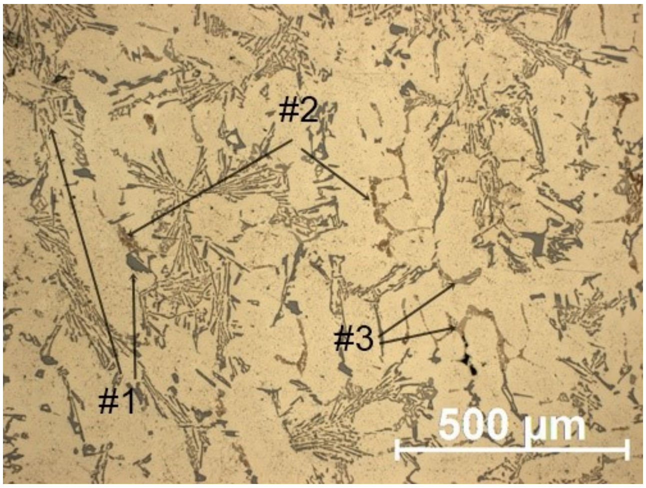

Fig. 10. AlSi6Cu4 alloy after solution in 505°C

Table 5.
Results of computer image analysis for the AlSi6Cu4 alloy

AlSi6Cu4 alloy			
	Average phase size with Cu (μm^2)	Amount of elements	Percentage of phase on the picture
DTA	217,61	6583	6,12
Solution 475°C	92,16	2145	5,46
Solution 505°C	57,88	7090	3,61

Table 3.
Results of computer image analysis for the AlSi8Cu3 alloy

AlSi8Cu3 alloy			
	Average phase size with Cu (μm^2)	Amount of elements	Percentage of phase on the picture
DTA	104,61	4084	5,23
Solution 475°C	82,14	10646	4,97
Solution 505°C	60,13	14345	2,25

4. Conclusions

An appropriate choice of the temperature of the solution in the heat treatment process is based on the data obtained during the thermal-derivative analysis (DTA). It cannot be ignored, because for multi-component systems, the phase coagulation temperature and eutectic are shifted. Each of elements added to material cause change of derivate curve. For Al-Cu alloys $T_{sol.}$ is 548°C, for AlSi8Cu3 490°C and for AlSi6Cu4 488°C. DTA is so important because it shows change characteristic points during modification alloy. But more importantly, thanks to DTA is allowed to define change average phase size with Cu after solutioning in 475°C and 505°C (tab. 5,6).

The performed research allows to formulate the following conclusions: Cu concentration in the tested range does not affect the value of solution temperature, increase in mass concentration of copper in the alloy from 3 to 4.3% by mass does not affect the amount of copper dissolved in the solution, after saturation at a specific temperature and time. Saturation at 505°C (above $T_{Sol.}$) proves that the hardness increases as a result of solution strengthening (tab.3).

In multicomponent systems, the heat treatment temperature can be optimized as a result of the thermal-derivative analysis.

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