

## IMPACT OF MAJOR ALLOYING ELEMENTS ON THE SOLODIFICATION PARAMETERS OF CAST HYPOEUTECTIC AlSi6Cu (1–4 WT.%) AND AlSi8Cu(1–4 WT.%) ALLOYS

Mile B. Djurdjević<sup>1\*</sup>, Srećko Manasijević<sup>2</sup>

<sup>1</sup>Nemak Linz GmbH, Zeppelinstrasse 24, 4020 Linz, Österreich

<sup>2</sup>Lola Institute, Kneza Višeslava 70a, 11000 Belgrade, Serbia

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### Abstract

The present work displays the potential of cooling curve analysis to characterize the solidification path of cast hypoeutectic series of Al-Si6-Cu(1–4 wt.%) and Al-Si8-Cu(1–4 wt.%) alloys. The aim of this work was to examine how variation in chemical composition of silicon and copper may affect characteristic solidification temperatures, fraction solid, and thermal freezing range of investigated alloys. Eight different Al-Si-Cu alloys (Al-Si6-Cu1, Al-Si6-Cu2, Al-Si6-Cu3, Al-Si6-Cu4, Al-Si8-Cu1, Al-Si8-Cu2, Al-Si8-Cu3 and Al-Si8-Cu4) have been analyzed applying cooling curve analysis technique. Characteristic solidification temperatures have been determined using cooling curves or their corresponding first derivative curves along with  $\Delta T$  curves. Fraction solid curves determined from recorded cooling curves have been used to calculate terminal freezing range and estimate crack susceptibility coefficient for each alloy. Theoretical mode for prediction of the cracking susceptibility coefficient developed by Clyne and Davies has been considered in this work. In addition, a novel mathematical model for prediction of crack susceptibility coefficient based on data collected from cooling curve analysis has been proposed.

*Keywords: cooling curve analysis, aluminum hypoeutectic alloys, Terminal Freezing Range, Crack Susceptibility Coefficient*

### Introductions

The Al-Si-Cu alloys have been in widespread use in the automotive industry due to its good casting characteristics and outstanding mechanical properties [1]. These alloys have been characterized by presence of two Al-Si and Al-Si-Cu eutectics, which are primarily responsible for defining the microstructure and mechanical properties of these alloys [1–4]. Comprehensive understanding of solidification paths of

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\*Corresponding autor: Mile B. Djurdjević, Mile.Djurdjevic@nemak.com

these alloys is of paramount importance for metallurgical engineers. This knowledge enable the process, quality and simulation engineers as well as designers to ensure that the casting will achieve the desired properties for its intended application after corresponding melting, liquid metal processing, mould filling and heat treatment procedure. In order to ensure that cast components produced from those alloys have good mechanical properties their as-cast microstructures must be closely monitored [1–3].

Major alloying elements: silicon and copper are primarily responsible for defining the microstructure and mechanical properties of aluminum alloys. Silicon is added to improve castability and fluidity, as well as to reduce shrinkage and to give superior mechanical and physical properties (the more silicon an AlSiCu alloy contains, the lower is its thermal expansion coefficient). The copper is the second major alloying element in these alloys. It has great impact on the strength and hardness of Al–Si–Cu casting alloys in as cast and heat treated conditions. In additions, copper reduces the corrosion resistance of aluminum alloys and in certain alloys increases stress corrosion susceptibility. In order to ensure that cast components have good mechanical properties their as-cast microstructures must be closely monitored. Thermal analysis (TA) has such potentials and has been used for many years in aluminum casting plants as a quality control tool [1,5–9].

The TA (cooling curve) method is useful for commercial applications for a number of reasons: it is simple, inexpensive and provides consistent results. This technique is a good choice for drawing fundamental relationships between cooling curve characteristics, alloy composition and melt treatment. Beside characteristic solidification temperatures, TA is often used to calculate solid fraction distribution between liquidus and solidus temperatures [10,11].

The Al-Si6-Cu4 and Al-Si8-Cu3 alloys are among the mostly used alloys for production of such intricate parts like cylinder heads and motor blocks for automotive industry. One of the problem that can appear during production of those parts is hot tearing (cracks). From the literature is well known that the terminal freezing range (TFR) has significant impact on the hot tearing formation [12,13]. The TFR represents the non-equilibrium partial freezing range near termination of solidification. Among many factors that have impact on hot tearing such as freezing range, fraction of eutectic phases, grain size, segregation and other, chemical composition is the most influencing factor. In general as the freezing range increases the hot cracking susceptibility also increases. In the literature there are several theoretical models [14] for the calculation of the hot crack susceptibility. The most commonly used is the cracking susceptibility coefficient (CSC) model proposed by Clyne and Davies for shape casting [15]. The CSC model correlates the susceptibility-composition relationship based on the consideration of the time during which processes related to crack production may take place and the structure is most vulnerable to cracking (critical time interval during solidification). According to equation (1) the CSC is defined as follows:

$$CSC = t_V/t_R \quad (1)$$

Where:

- $t_V$  is the vulnerable time period and is calculated as the time difference between mass fraction of liquid 10% and mass fraction of liquid 1%.

- $t_R$  is the time available for stress relief processes and is calculated as the time difference between mass fraction of liquid 60% and mass fraction of liquid 10%.

The aim of this work is to examine how variation in chemical composition of Silicon and Copper may affect characteristic solidification temperatures of Al–Si–Cu alloys as well as their fraction solid, and thermal freezing range.

## Experimental procedure

### *Melting and Thermal Analysis Procedures*

Eight different Al–Si–Cu alloys with the chemical compositions as presented in Table 1 are synthetically produced. The content of the major alloying elements varied between 5.78–8.14 wt.% Si and 1.07–4.31 wt.% Cu, while the contents of other alloying elements try to be as low as possible. Their chemical compositions have been determined using optical emission spectroscopy (OES).

*Table 1. Actual chemical composition, (in wt. %) of synthetic Al–Si–Cu alloys*

Alloy	Zn	Mn	Mg	Fe	Cu	Si
6/1	0.01	0.01	0.14	0.07	1.07	5.91
6/2	0.01	0.01	0.15	0.11	1.83	5.90
6/3	0.01	0.01	0.15	0.06	3.03	5.82
6/4	0.01	0.01	0.13	0.07	3.96	5.78
8/1	0.003	0.0029	0.0006	0.136	1.09	8.03
8/2	0.0025	0.0028	0.0007	0.123	1.93	8.14
8/3	0.0031	0.0032	0.0009	0.141	2.96	8.03
8/4	0.003	0.0031	0.0008	0.138	4.31	7.84

The alloys were melted in an electric resistance furnace capacity 10 kg. No grain refining and modifier agents were added into the melt. Samples with masses of approximately 250 g were poured into thermal analysis steel test cups (high of the cap was 60 mm, while bottom and top diameters were 45 and 55 mm respectively). Two calibrated K type thermocouples were inserted into the melt and temperatures between 700–400 °C were recorded. The data for TA was collected using National Instrument data acquisition system linked to a personal computer. The tip of the thermocouple was kept always at the constant high, 15 millimeters from the bottom of the crucible. The cooling conditions were kept constant during all experiments and were approximately 0.15 °C/s. The cooling rate has been calculated as the ratio of the temperature difference between liquidus and solidus temperature to the total solidification time between these two temperatures. Each TA trial was repeated two times. Consequently, a total of sixteen cooling curves were gathered during experiments.

The TFR and the CSC were calculated from the fraction solid curves. The Newtonian method has been applied to calculate base lines for the further calculation of the fraction solid distribution curves.

## Results and discussion

### Characteristic solidification temperatures

All characteristic solidification temperatures have been determined using cooling curves, their corresponding first derivatives or  $\Delta T$  curves, as it is illustrated on Figure 1.

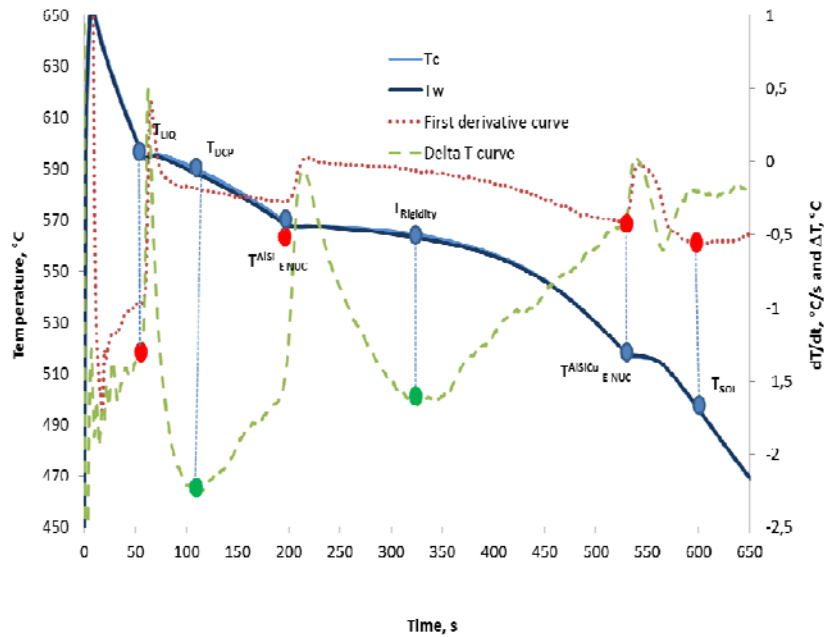


Fig. 1. Determination of characteristic solidification temperatures using cooling curve analysis

The liquidus, both eutectic temperatures (Al–Si and Al–Si–Cu) as well as solidus temperatures have been determined from the first derivative curves. The dendrite coherency points (DCP) and Rigidity temperatures have been determined from the  $\Delta T$  curves, using two thermocouples method proposed by Bäckerd [1]. In this method one thermocouple is located at the center ( $T_c$ ) of a test crucible, while the other at a nearby inner wall ( $T_w$ ). The DCP and Rigidity temperature are determined by identifying the first and second local minimums respectively on the  $\Delta T$  versus time curve ( $\Delta T = T_w - T_c$ ) [1,16]. The characteristic solidification temperatures determined using cooling curve analysis for all investigated alloys have been summarized in Table 2.

In order to eliminate the impact of the cooling rate on the depression of characteristic solidification temperatures during all experiments the cooling rate was kept constant ( $\sim 9$  °C/min). From the Table 2 is clear that any increase in the content of silicon and copper depresses the characteristic solidification temperatures, some of them significantly. The obtained results are in agreement with binary Al–Si, Al–Cu and ternary Al–Si–Cu phase diagrams from which is obvious that higher concentrations of silicon and copper lead to lower liquidus temperatures of corresponding (Al–Si, Al–Cu and Al–Si–Cu) binary and ternary systems.

Table 2. Characteristic solidification temperatures of Al–Si–Cu alloys determined using cooling curve analysis

Temperature	Cu, wt.%		Si, wt.%
	8	6	
T <sub>LIQ</sub>	600.7	622.4	1
T <sub>DCP</sub>	598.7	616.1	
T <sub>E</sub> <sup>Al-Si</sup>	575.0	573.7	
T <sub>Rigidity</sub>	571.4	573.1	
T <sub>E</sub> <sup>Al-Si-Cu</sup>	517.4	518.7	
T <sub>SOL</sub>	501.8	501.7	
T <sub>LIQ</sub>	601.7	618.9	
T <sub>DCP</sub>	597.9	615.0	
T <sub>E</sub> <sup>Al-Si</sup>	572.2	570.5	
T <sub>Rigidity</sub>	569.0	569.8	
T <sub>E</sub> <sup>Al-Si-Cu</sup>	517.2	518.3	
T <sub>SOL</sub>	499.39	502.2	
T <sub>LIQ</sub>	600.5	614.8	3
T <sub>DCP</sub>	591.0	611.7	
T <sub>E</sub> <sup>Al-Si</sup>	569.3	569.3	
T <sub>Rigidity</sub>	566.1	566.5	
T <sub>E</sub> <sup>Al-Si-Cu</sup>	517.7	522.0	
T <sub>SOL</sub>	494.5	502.7	
T <sub>LIQ</sub>	596.8	610.3	
T <sub>DCP</sub>	587.8	608.3	
T <sub>E</sub> <sup>Al-Si</sup>	565.9	566.9	
T <sub>Rigidity</sub>	563.2	563.3	
T <sub>E</sub> <sup>Al-Si-Cu</sup>	516.3	522.9	
T <sub>SOL</sub>	495.7	501.8	

#### Cooling curves analysis\_Liquidus Temperature

The liquidus temperature specifies the maximal temperature at which the crystal can coexist with the melt in thermodynamic equilibrium. Above the liquidus temperature does not exist any crystal and the melt is liquid and homogeneous. This temperature is very important in casting industry because it defines the preheating temperature of the melt (difference between the liquidus temperature and initial/pouring temperature).

Table 2 shows characteristic solidification temperatures of all investigated alloys that have been determined using cooling curve analysis. According to those results increase in the Silicon content for one weight percent depress the liquidus temperature for 7.7 °C by the constant content of copper. The obtained results are in agreement with binary Al-Si phase diagram, where increase of silicon content from 6–8 wt.% decrease the liquidus temperature for 13.28 °C (the temperature drops almost linearly from 623.3–609.9 °C) what is approximately decrease for 6.6 °C per 1 wt.% of silicon. The higher decrease of liquidus temperature from experimental results is related to the additional impact of copper content. The calculation run in Pandat has proved this

assumptions, that addition of 1 wt.% of Cu into Al-Si6 and Al-Si8 alloys has additionally decreased the corresponding liquidus temperatures for  $\sim 1.2$  °C.

#### *Cooling curves analysis\_Dendrite Coherency Temperature*

During the solidification of any aluminium hypoeutectic alloys dendritic network of primary  $\alpha$  - aluminium crystals will be developed. In the early stage of solidification dendritic crystals are separate and move freely in the melt. However, as the melt cools, the dendrite tips of the growing crystals begin to impinge upon one another until a coherent dendritic network is formed [17]. The temperature at which this occurs is called dendrite coherency temperature (DCT) and is very important feature of the solidification process [17–22]. This temperature marked the moment when the “mass” feeding transferred to interdendritic feeding. [1,17–22]. Casting defects such as macrosegregation, shrinkage porosity and hot tearing begin to develop after the DCT [17–22].

In this work the DCP has been determined for each set of experiments using traditional two thermocouples technique developed by Bäckerud [1,17]. The solidification conditions, chemical compositions of alloy and addition of grain refiners are major factors that have significant impact on the dendrite coherency temperature. Independent from applied measured techniques, it has been verified that faster cooling rate and increase in solute concentration postponed the coherency point to the lower temperature [1,17–22].

The influence of silicon and copper on the dendrite coherency temperature is visible in Table 2. The higher silicon and copper contents progressively reduce the dendrite coherency temperature. Impact of silicon is more significant (decrease DCT for  $\sim 8.7$  °C per 1 wt % of Si) than that of copper (decrease DCT for  $\sim 3.0$  °C per 1 wt % of Cu).

These results are not unexpected. It is well known that the sizes of the dendrites are influenced by the levels of alloying elements present in the melt (of course the main influence is the solidification rate). During the primary solidification of the aluminium alloys the alloying elements are not evenly distributed between solid and liquid phases. Excess amount of solute displaced away from the solidification interface into the melt results in an increase in volume of solute located between already formed dendrite arms. This supersaturation (or related constitutional undercooling) represents the driving force for the growth of the dendrites. The space between  $\alpha$ -aluminium dendrite arms must increase to accommodate an increasing amount of solute elements. Clearly then a higher concentration of alloying elements will reduce the growth of dendrites and postpone their contact (coherency) to lower temperature. It is also expected that the elements having a higher solubility in the aluminium melt are less effective in reducing the size of the secondary dendrite arm spacing (SDAS). Therefore, the effect of the same content of copper is significantly less than that of the same amount of silicon.

#### *Cooling curves analysis\_Al-Si Eutectic Temperature*

In order to control the quality of aluminium melts (f.e., level of modification) it is necessary to detect the eutectic temperature ( $T_E^{Al-Si}$ ) of the aluminum alloys with the highest possible degree of accuracy. As can be seen from Table 2, silicon has not significant impact on this temperature (1 wt.% of silicon decrease this temperature for  $\sim 0.5$  °C), while increase of the copper content from 1wt.% to 4 wt. % by the constant silicon content, dropped this temperature for  $\sim 2.6$  °C. From the available literature

[3,23,24] it is well known that some minor alloying elements such as Sr, Na, Sb and other have significant impact on this temperature. Small additions of those elements (even in ppm range) significantly depress this temperature up to approximately 12 °C, altering drastically corresponding Al–Si eutectic morphology from coarse to fine structure and finally improving mechanical properties of Al–Si–Cu alloys. Therefore, this temperature need to be constantly monitored and it has been very often used in aluminium casting plants as a quality control parameters.

#### *Cooling curves analysis\_Rigidity Temperature*

The Rigidity temperature is determined as a second  $\Delta T$  minimum identify in the region of primary precipitation of Al–Si eutectic. This temperature, according to the literature [1,17], indicated the moment when the interdendritic feeding transfer to burst feeding. Recently, it has been develop a procedure to detect this temperature from the cooling curve [16]. Unfortunately, in the available literature there is a lack of data regarding the value of Rigidity temperature for cast Al–Si–Cu alloys. Bäckérud is the only one researcher that methodically tries to determine this solidification property of automotive cast Al–Si–Cu alloys applying rheological measurement [17]. In his comprehensive studies [1,17] he analyzed the impact of chemistry, cooling rates and liquid metal processing on the solidification behavior of wrought and cast aluminum alloys using together cooling curve and rheological techniques. The Rigidity temperature for all investigated alloys through all his studies [17] has been determined only applying rheological measurement technique, while DCT have been determined with two thermocouples technique. Recently, it has been upgraded existing TA technique to be able to detect the Rigidity Point/Temperature using two thermocouples. In that work [16], it has been shown reasonable agreement regarding determination of Rigidity temperature, independent from the applied measured techniques (for Al-Si5-Cu1 alloy Rigidity temperature was 571.5 °C and 572.8 °C respectively determined using rheological and thermal analysis techniques with two thermo elements). From the Table 2 it is clear that silicon has no any significant impact on this temperature, while increase in copper content for 1 wt.% decrease this temperature for ~3 °C per 1 wt.% Cu.

#### *Cooling curves analysis\_Al–Si–Cu Eutectic Temperature*

The Al-Si-Cu eutectic temperature is the second eutectic temperature that characterized the solidification paths of AlSiCu alloys. From Table 2 it is obvious that increase of the silicon (from 6 wt.% to 8 wt. %) and copper (from 1 wt. % to 4 wt.%) contents have some effect on the precipitation temperature of copper rich phase(s). These experimental results (Table 2) indicate that the copper enriched phases precipitate at different temperatures depending on the amount of copper present in the particular Al-Si6-Cu(1–4) and Al-Si8-Cu(1–4) alloys. The nucleation temperature of the copper enriched phases as well as their solidus temperatures can be accurately read from the first derivatives of the cooling curves and used to define the maximum temperatures that the castings can be exposed to during the conventional solution treatment process. From the available literature is well known that also some other elements (Sr, Na, Pb and Sn) have significant impact on the precipitation temperatures of copper rich phases [3,25,26] but their impact have been not taken under consideration in this work.

*Cooling curves analysis \_ Solidus Temperature*

The solidus temperature identifies the temperature at which the last portion of the liquid has been transformed into a solid. Below this temperature the given alloy is stable in solid phase. The results presented in Table 2 indicated that various silicon and copper contents in investigated alloys have impact on solidus temperature caused by segregation of silicon and copper in the last portion of the melt, immediately before it entirely solidify.

*Table 3. Characteristic fraction solid values of AlSiCu alloys determined using cooling curve analysis*

TA results			
$f_s$	wt.% Si		Cu, wt.%
	8	6	
$f_{S_{LIQ}}$	0	0	
$f_{S_{DCP}}$	13.85	16.17	
$f_{S_E^{Al-Si}}$	33.64	51.08	1
$f_{S_{Rigidity}}$	67.85	75.52	
$f_{S_E^{Al-Si-Cu}}$	98.40	99.08	
$f_{S_{SOL}}$	100	100	
$f_{S_{LIQ}}$	0	0	
$f_{S_{DCP}}$	14.10	13.10	
$f_{S_E^{Al-Si}}$	34.67	51.82	2
$f_{S_{Rigidity}}$	68.15	72.60	
$f_{S_E^{Al-Si-Cu}}$	96.92	97.75	
$f_{S_{SOL}}$	100	100	
$f_{S_{LIQ}}$	0	0	
$f_{S_{DCP}}$	15.09	13.34	
$f_{S_E^{Al-Si}}$	31.71	51.32	3
$f_{S_{Rigidity}}$	58.51	65.78	
$f_{S_E^{Al-Si-Cu}}$	94.35	95.66	
$f_{S_{SOL}}$	100	100	
$f_{S_{LIQ}}$	0	0	
$f_{S_{DCP}}$	13.95	13.24	
$f_{S_E^{Al-Si}}$	30.67	48.94	4
$f_{S_{Rigidity}}$	53.16	62.50	
$f_{S_E^{Al-Si-Cu}}$	91.21	92.76	
$f_{S_{SOL}}$	100	100	



### Fraction solid analysis

Beside characteristic solidification temperatures, the thermal analysis is often used to calculate solid fraction distribution between  $T_{LIQ}$  and  $T_{SOL}$  temperatures. A critical requirement for the solid fraction calculation applying cooling curve analysis is determination of, what is called a base line [1,10,11]. The base line is in principle the first derivative of the cooling curve measured by the thermocouple(s), inserted in the alloy test sample, assuming that the metal doesn't undergo any phase transformation during the solidification process. In other words the base line overlaps the first derivative of the cooling curve in single phase parts of the sample cooling process, for temperatures higher than  $T_{LIQ}$  and for temperatures lower than  $T_{SOL}$ . In the literature are two known methods, Newtonian [10, 11,27] and Fourier [10,27], that have been successfully used to calculate solid fraction distribution using cooling curve analysis. In this work only Newtonian method has been applied to calculate the base line using cooling curve analysis. Table 3 summarized the values of fraction solid at each characteristic solidification temperatures calculated using recorded cooling curves. By increased contents of silicon and copper in aluminum melts the fraction solid values at characteristic solidification temperatures increased. It looks that impact of silicon is more significant than that of copper. All those values have been used to calculate the TFR and the CSC for investigated alloys.

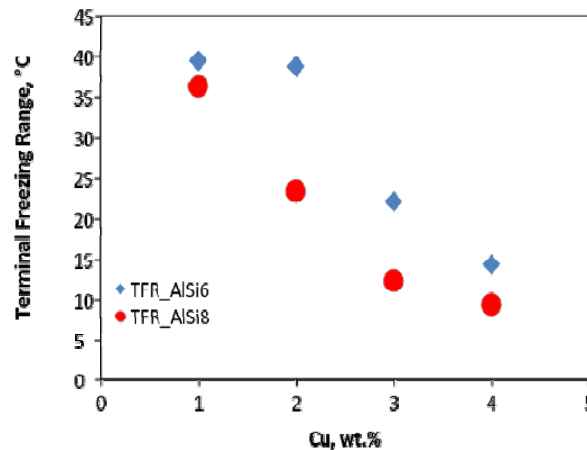


Fig. 2. The impact of Cu and Si on the TFR ( $TFR = f_{S,95\%} - f_{S,99.5\%}$ ) by AlSiCu alloys, determined from the recorded cooling curves.

Analyzing results from Figure 2 it could be concluded that increases in 1 wt.% of Cu decrease TFR for  $\sim 8.2$  °C, while the increases in 1wt. % of Si reduce the TFR for  $\sim 1.75$  °C. From Figure 2 it is also evident that copper contents have larger impact on the hot tearing formation than silicon contents. The lower TFR has been achieved at the AlSi8Cu4 alloy and the higher at the Al-Si6-Cu1 alloy. The drop in the TFR is a slightly higher at the AlSi8Cu(1-4) alloys ( $6.727$  °C per 1 wt.%Cu) in compare to Al-Si6-Cu(1-4) alloys ( $6.275$  °C per 1 wt.% Cu). Experimental evaluation of hot tearing tendency by aluminum alloys is very complex. Cooling curve analysis has potential these criterions to quantify and should be more often used.

### Crack Susceptibility Coefficient

Potential application of the fraction solid values at DCP and Rigidity temperature to predict the CSC. Among many mathematical models that have been used to predict crack susceptibility coefficient as a function of chemical composition, the model proposed by Clyne and Davies is mostly applied for shape casting. The equation (1) analytically expresses this relationship. The Figure 3 illustrate the impact of major alloying elements, silicon and copper on the CSC for Al-Si6-Cu(1-4) and Al-Si8-Cu(1-4) alloys calculated applying equation (1).

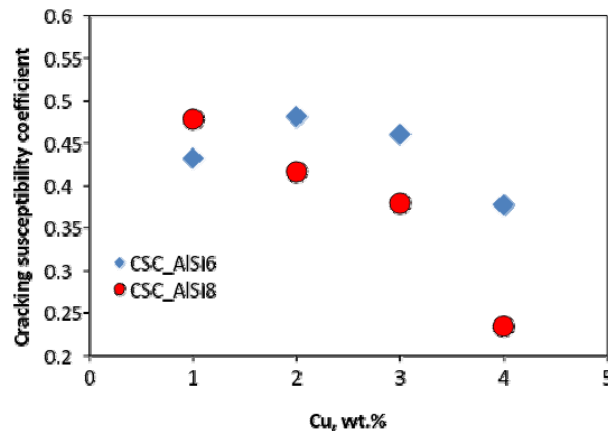


Fig. 3. The impact of Cu and Si on the CSC (calculated applying Clyne and Davies model) by Al-Si-Cu alloys, determined from the recorded cooling curves.

In the available literature there are some others mathematical models that can be used to predict the impact of chemistry on the development of the hot tearing [14,18-21]. Among them, Katgerman [19] also developed a modified hot tear criteria defined by equation (2):

$$CSC = (t_{0.99} - t_{cr}) / (t_{cr} - t_{coh}) \quad (2)$$

These two time periods ( $t_{0.99} - t_{cr}$  and  $t_{cr} - t_{coh}$ ) are bordering at the critical point ( $t_{cr}$ ) where, the system transits from a regime where liquid feeding is adequate to a regime where liquid feeding is inadequate. Katgerman also makes the point clear that the time period for stress relief starts when dendrite coherency is attained, since the latter is by definition the point where the stress sustained by the solid phase becomes different from those in the liquid phase.

The novel approach proposed by author assumed that the Katgerman criteria ( $t_{cr}$ ) can be modified by introducing  $t_{Rigidity}$  instead  $t_{cr}$  parameter in the above mentioned CSC expression. The new expression can be written as follows by equation (3):

$$CSC = (t_{0.99} - t_{Rigidity}) / (t_{Rigidity} - t_{coh}) \quad (3)$$

According to Figure 1, Rigidity temperature is the border temperature between interdendritic and burst feeding. This temperature exactly describes the transition from the region where feeding is relatively carried out with some difficulty to a region where liquid feeding is almost impossible without extra forces. This assumption is going to be

analyzed in a series of experiments on cast Al–Si–Cu alloy with varying content of silicon and copper.

## **Conclusions**

A comprehensive understanding of the solidification process is of paramount importance for the control and prediction of actual casting characteristics. This work has shown that thermal analysis is a valuable tool widely used in aluminium casting plants that can collect numerous parameters (characteristic solidification temperatures, fraction solid distribution, and TFR) beneficial for better understanding the solidification path of Al–Si–Cu alloys.

In addition, the data collected using cooling curve analysis should be applied in existing simulation software in order to improve accuracy of simulation. Naturally, the industrial interest is to be able to make simulation and optimization based on more realistic database in order to more reliably predict the quality of very complex cast parts. The TA has such opportunity, and has to be used more often in providing simulation engineers with all necessary data for simulation in synergy with commercial software packages.

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