

# Using of Thermal Analysis in the Industrial Practice – Consumption Reduction of Grain-Refinement Master Alloy and Optimization of Computer Simulation Results

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**The solidification process of a metal or alloy is accompanied by the evolution of heat the magnitude of which depends on the various phases that form during the solidification. Recorded temperature-time data can yield quantitative information about the alloy solidification process. Such a plot is called a cooling curve and the general name given to the technique is thermal analysis.**

**The cooling curve serves as a “finger print” of the solidification process and can be used to predict the structure of the test sample and consequently the actual casting. The aim of this paper is to show the ability of the thermal analysis technique in order to predict some of the key solidification parameters, which can be used to monitor and improve the quality of the casting. In addition, some of the results collected from the cooling curve can be used as an input data in existing software packages in order to improve their accuracy.**

**Key words:** thermal analysis, cooling curve analysis, grain-refinement, master alloy AlTi5B1, dendrite coherency point

## 1. Importance of molten mass quality

Aluminum casting plants are using significant amounts of primary, secondary and master alloys in order to produce automotive parts of high quality. The quality of cast products depends directly on the quality of molten metal from which the products are cast. Complete understanding of the melt quality is of vital importance for the control and production of the high quality cast parts [1]. Any defect added or created during the melting stage will be carried to the final microstructure, and certainly, affect the quality of cast products. Therefore it is apparent that the control of the quality of the cast products begins with the control of the quality of the melt.

The solidification process of a metal or alloy is accompanied by the evolution of heat of various phases that form during the solidification. Recorded temperature-time data can yield quantitative information about the alloy solidification process. Such a plot is called a cooling curve and the general name given to the technique is thermal analysis. The cooling curve serves as a “finger print” of the solidification process and can be used to predict the structure of the test sample and consequently the actual casting properties. This paper will briefly review application of thermal analysis in aluminum casting plants, showing its ability to predict some key solidification parameters, which, can be used to monitor the quality of cast products.

In the aluminum casting industry, the application of thermal analysis to study the development of the test sample structure was reported in early publications, for example Cibula [3]. In the early 1980's, this process control technology started to be regularly used in aluminum foundries. The thermal analysis test samples can be taken either by submerging a cylindrical (graphite ceramic or steel) cup into the melt or pouring the melt by ladle into test cup. One or two K-type thermo elements have been placed into the melt and measure the temperature during solidification of test sample. The outputs from the thermocouples were connected to a PC via data logger, where temperature/time data were recorded and later processed in various ways. At present aluminum casting plants are regularly using thermal analysis to control the efficiency of master alloys additions (grain refiner and modifier) into the aluminum melt. A state of the art thermal analysis has the potential to determine some solidification features such as characteristic solidification temperatures, fraction solid, dendrite coherency point and many others that can be used to fulfill the lack of data in presently used commercial simulation software packages.

## 2. Thermal Analysis Technique as a Melt Quality Control Tool

A comprehensive understanding of melt quality is of paramount importance for the control and prediction of actual casting characteristics. If process engineers can act in a proactive rather than a reactive manner with respect to melt and casting quality control, they can reduce cost downtime and scrap levels. Thermal analysis can provide such capabilities and therefore has important advantages over its post-process counterparts, which are often destructive in nature.

### 3. Assessment of Grain Size Using Thermal Analysis Technique

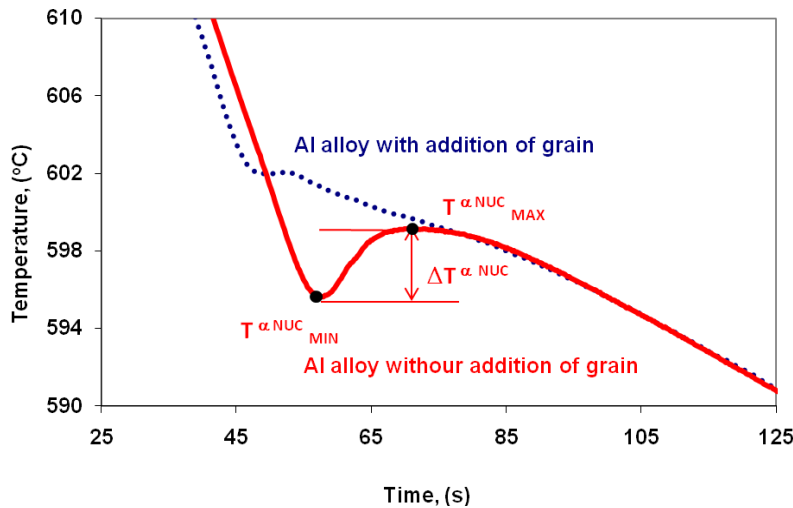
Aluminum alloys, like alloys of other light metals, will normally, without addition of grain refinement, form coarse, equiaxed and columnar crystals during solidification. The degree of coarseness or the length of the columnar crystals depends on the pouring temperature of the alloy, thermal gradients set up within the casting, and the presence of naturally occurring grain nuclei (i.e. imperfections such as high points on the mould surface, and insoluble particles) [2,3]. Grain refinement may be achieved through a number of different methods such as: increasing cooling rate, the dynamic techniques like vibration and stirring, constitutional undercooling and the addition of grain refiners.

Addition of grain refiners is at the present the most common method used in the aluminum casting industry. There are many elements and compounds that have grain-refining effects for aluminum alloys: (such as Ti, B, Zr, Cr, V, Mo, W and Nb carbides). The most potent of this group are Ti and B and carbides [3-10]. Thus, they are the most common grain refiners in use today in the form of AlTi and AlTiB master alloys.

The ability to closely control the grain size is of the major importance to solving casting problems on the foundry floor. Hypoeutectic alloys have a large proportion of primary aluminum grains in their microstructure. Applying grain refinements their size can be reduced giving the following benefits to the cast products:

- Improve feeding during solidification
- Reduces and more evenly distributed shrinkage porosity
- Better dispersion of second phases and impurities
- Reduced solution times for heat treatable alloys
- Improved surface finish
- Reduce hot tearing
- Better strength and fatigue life

Grain refinement that occurs during solidification can be a function of undercooling occurring during the liquidus arrest (see *Fig. 1*). The shape of the cooling curve at the beginning of the solidification process gives a good indication of the number of nuclei present in the melt. When there are a great number of nuclei, the curve exhibits little undercooling, (as illustrated in Figure 1 by the dotted line). When there are few nuclei, there is more undercooling, which is illustrated in the *Fig. 1* by the solid line.



*Fig. 1* A segment of the cooling curve showing the impact of grain refiners on undercooling

The relationship between  $T^{\alpha, \text{DEN}}_{\text{MIN}}$  and  $T^{\alpha, \text{DEN}}_{\text{MAX}}$ , graphically presented in *Fig.1* is mathematically expressed by equation (2). This equation can be used as a criterion for the estimating grain size [2,4,8].

$$\Delta T^{\alpha, \text{NUC}} = T^{\alpha, \text{NUC}}_{\text{MAX}} - T^{\alpha, \text{NUC}}_{\text{MIN}} \quad [^{\circ}\text{C}]$$

Where:

$T^{\alpha, \text{NUC}}_{\text{MAX}}$  - alpha Al dendrite growth temperature that is achieved as a result of the sudden release of the latent heat after the initial nucleation event.

$T^{\alpha, \text{NUC}}_{\text{MIN}}$  - alpha Al dendrite undercooling temperature is the temperature below the equilibrium transformation-temperature to which the Al melt cools before nucleation.

$\Delta T^{\alpha, \text{NUC}}$  - depression of the alpha dendrite growth temperature is the criteria for estimating grain size (smaller value of  $\Delta T^{\alpha, \text{NUC}}$  corresponds to finer grain structure).

### 3.1 Projekt at Nematik – Consumption Reduction of Grain-Refinement Master Alloy

Nematik is a worldwide leader in the production of cylinder heads via gravity semi-permanent mold casting of Al-Si alloys. In the plant Nematik Slovakia, the main goal of the project was to optimize - reduce the amount of the master alloy (AlTi5B1) addition into the aluminum melt under the condition not influence the casting scrap or mechanical properties, optionally to improve the quality of cast products. This paper is only the part of global project that Nematik run among other casting plants in Europe in order to optimize the amount of master alloys additions into Al melts [11]. The whole project has been scheduled for 5 months and has been run in following 4 phases:

#### Phase No. 1: **Present state**

- Quantity of master alloys added into melts per year and cost
- Design the experiments
- Define measure parameters
- Review the problems recorded during the last month
- Present status of Thermal analysis application at casting plant
- Scrap level analysis as well as other relevant documents critically analyzed

#### Phase No. 2: **Optimisation under laboratory conditions**

- Experiments under laboratory conditions (Thermal and Metallographic analysis)
- Experiments under production conditions
- Full proof of cast parts (100%) using Roentgen and Metallographic analysis

#### Phase No. 3: **Optimisation under production conditions**

- Optimisation of master alloys addition under production conditions
- Full proof of cast parts (100%) using Roentgen and Metallographic analysis

#### Phase No. 4: **Applied optimized parameters in the daily production**

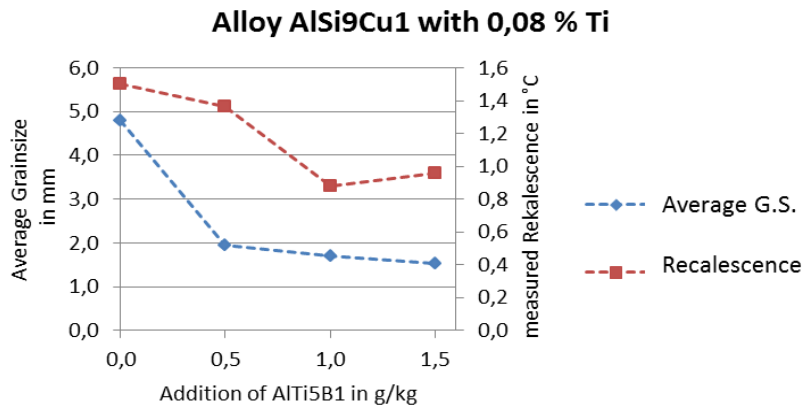
- Incorporation new knowledge into serial productions

During the whole project, two aluminum alloys (AlSi10Mg and AlSi9Cu1 – A359 and A354) have been used for optimisation of the grain refinement additions. Both alloys have been used to produce the cylinder heads for gas and diesel engines. The addition of master alloys into these alloys was not previously standardized. Depending on operators, various amounts of master alloys have been added into melts. In additions, two thermal analyses equipment, which have been installed at the foundry floor have not been properly used due to lack of knowledge and experience of operators. The consumption of grain refiners (AlTi5B1 master alloys) per year was extremely high. Nematik Ziar casting plant produces around 1 million cylinder heads per year. Around 26000Tonne aluminum melt need to be grain refined and for that purpose the casting plant is spending around 100 000 € per year. This project should give us answers on two questions:

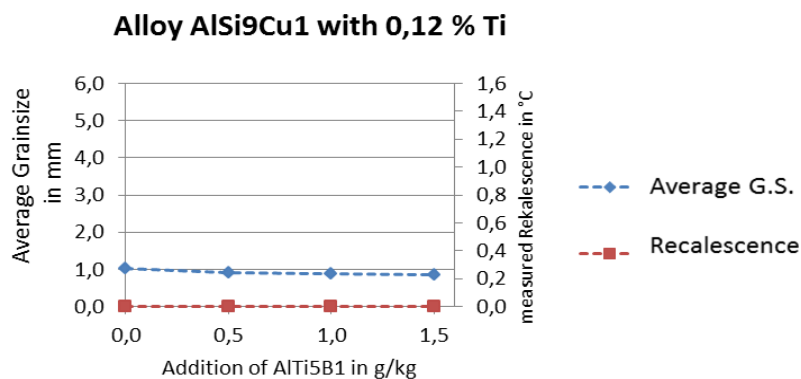
- What is the optimal amount of master alloys that need to be used in order to be able to produce sound product?
- Is there any cost saving potential by optimizing master alloy addition into aluminum melts?

### 3.2 Results of optimization

As can be seen from *Fig. 2* and *3* the residual amount of Ti play significant impact on the grain refinement. The presence of 0.12 wt.% Ti in AlSi9Cu1 alloy as a residual amount does not required addition of master alloy. In the case that residual amount of Ti is 0.08 wt.% it is necessary to add master alloys in order to reduce the grain size (G.S) in solidified alloy. Even though that alloy with 0.12 wt.% Ti does not required addition of Ti under laboratory conditions in the production some amount of grain refiner has been added (2 rods/bars) as a safety precautions.



**Fig. 2** Influence of 0÷1,5 g grain refinement addition per kg AlSi9Cu1 alloy on grain size [mm] and measured recalescence [°C]; the basic content of titanium in the alloy is 0,08 weight %



**Fig. 3** Influence of 0÷1,5 g grain refinement addition per kg AlSi9Cu1 alloy on grain size [mm] and measured recalescence [°C]; the basic content of titanium in the alloy is 0,12 weight %

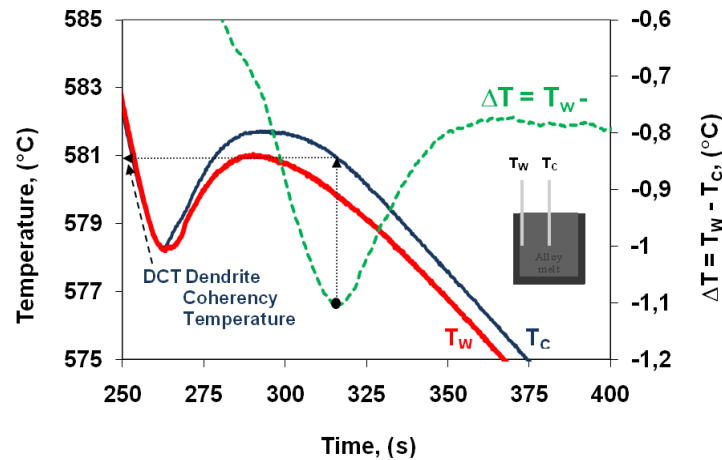
At the end of these experiments we were astonished with obtained results. It has been recognized that there is a potential for approximately 70% cost saving by reduction of master alloys additions into investigated alloys. In additions, the mechanical, metallurgical and physical properties of cast parts did not change. Even though, these properties have been even better or at least the same as before reduction of grain refiner additions. In particular case, by AlSi10Mg alloy, the grain refinement additions for one holding furnace have been reduced from 1200g (6 rods) to only 200 g (1 rod). In other words, it is possible to save around 12000€ per holding furnace in one year. By AlSi9Cu1 alloy this cost saving for one year was a little bit higher, around 16000€ per holding furnace. Before optimization, the addition of master alloy in this case was 16 bars (1600g) and after experiments it has been reduced to 200g (1bar). As a results of optimization the target amount of Ti in both alloys has been increased from 0.1 wt.% on the 0.12 wt.%.

#### 4. Assessment of the Dendrite Coherency Temperature (DCT) Using Thermal Analysis Technique

In the early stages of equiaxed dendritic solidification, the dendritic crystals are separate and can move freely throughout the liquid metal. The entire solidifying metal shrinks lowering the surface of the melt. This simultaneous movement of the slurry of solidified dendrites and the remaining liquid is termed “mass feeding”. At later stages of solidification, the developing dendrites begin to impinge upon one another and the dendritic network becomes coherent. At this point the system behaves less like a liquid or slurry and starts to behave more like a solid. It is at this point where properties such as shear-strength and thermal conductivity have transition points and where “mass feeding” cease. This transformation point is called the dendrite coherency point [2].

When dendrites become coherent a number of processes start to take place simultaneously in the semi-solid region. As cooling continues, the dendritic skeleton is subjected to strain from thermal contraction. Hot tears may form if the strain is sufficient and if insufficient liquid metal is available to fill the inter-dendritic regions. In addition, macrosegregation results if solidification processes occur after a great deal of inter-dendritic feeding has taken place.

The DCT can be defined by the two thermocouples thermal analysis technique and by comparing the temperature measured at the center of the sample versus that measured near the wall. The “point” of maximum temperature difference is the dendrite coherency point (DCP), *Fig. 4* [2].



*Fig. 4*  $T_c$  and  $T_w$  cooling curves vs. the time and temperature difference ( $\Delta T = T_w - T_c$ ) curve with identified DCT being the minimum of  $\Delta T$  according to the Bäckerud method

In the late 1980's Bäckerud [2] developed a method that utilizes two thermocouples. One thermocouple is located at the centre of a crucible, and the other one at nearby inner wall. The DCT is determined by identifying the point of minimum temperature difference at the  $\Delta T$  curve.

The interest in simulation applied in the aluminium casting industry has extended significantly in the last few decades. Three main reasons are responsible for that:

- the necessity to improve productivity and quality of cast products
- to speed up the design process and
- to investigate the influence of different process parameters without the need for expensive experimental trials.

The accuracy of casting simulation depends in great deal on the quality of the available physical and thermo physical material properties provided by the software's database. Available databases presently used by commercial simulation software packages for the casting industry usually come with material properties for only a few selected standard alloys. In the case of more sophisticated alloys with differing chemical compositions, refinement and/or modification treatments, thermal analysis can be a very useful tool in order to collect the missing parameters or more accurate thermo physical data for investigated alloys. Providing more realistic data for exact alloy compositions and actual casting conditions (different cooling rates) thermal analysis can considerably improve the accuracy of simulation process. In additions, some of the provided data such as f.e. dendrite coherency point, can't be find in existing commercial software database packages.

Dendrite coherency temperature has not been often used in aluminum foundry daily activities. But this temperature is a very important parameter for simulation, because it defines the temperature at which mass feeding start to be restricted. Further feeding is possible by melt flow through interdendritic channels initiated by gravity force (so called interdendritic feeding) [12,13]. Applying this parameter the simulation engineers are capable more realistic to predict the occurrence of defect during solidification of cast parts.

## 5. Conclusions

Thermal analysis can be used to evaluate the following processing and materials parameters: grain size, dendrite coherency temperature, dendrite arm spacing, the level of Al-Si eutectic modification, fraction solid as well as the characteristic temperatures of various metallurgical reactions between liquidus and solidus temperatures. With the use of modern data acquisition systems and computer processing thermal analysis this becomes a powerful tool for casting process control. Thermal analysis can provide process engineers with the ability to act in a proactive manner in respect to melt and casting quality.

In this particular case, experiments were carried out to observe the effect of master alloy AlTi5B1 on the microstructure of the aluminium-silicon alloy. It was found a way to reduction of master alloy in more cases. The cooling curve analysis,  $\Delta T^{\alpha, NUC}$  parameter, has shown that already by lower addition of master alloy (0.5g per 1 kg melt) the grain refinement is achieved.

Beside that the thermal analysis technique is capable to provide available software with a new data (f.e. DCT) that can drastically improve its accuracy. This temperature marked the moment when the “mass” feeding transferred to interdendritic feeding. Casting defects such as macrosegregation, shrinkage porosity and hot tearing begin to develop after the DCT. Therefore, a thorough understanding of the solidification behavior at the DCT and the factors that influence it are crucial for the engineering of a new alloys and the development of related manufacturing processes.

## References

- [1] Tillová, E., Chalupová, M., Hurtalová, L., Ďuríníková, E.: *Quality control of microstructure in recycled Al-Si cast alloys*. Manufacturing Technology, 11, No. 12 - 2011, p. 70 – 76.
- [2] Bäckerud, L., Chai, G., Tamminen, J.: *Solidification Characteristics of Aluminum alloys*. Volume 2. AFS-SKANALUMINIUM, 1986, p. 95-105.
- [3] Cibula, A.: *The Mechanism of Grain Refinement of Sand Castings in Aluminum Alloys*. J. Ins. Metals vol. 76, p. 312.
- [4] Apelian, D., Sigworth, G. K., Wahler, K. R.: *Assessment of Grain Refinement and Modification of Al-Si Foundry Alloys by Thermal Analysis*. AFS Transaction, 1984, 161, p. 297-307.
- [5] Murty, B. S., Kori, S. A. and Chakraborty M.: *Grain Refinement of Aluminum and its Alloys by Heterogeneous Nucleation and Alloying*. International Materials Reviews, Vol. 47, No. 1, p. 3-29, 2002.
- [6] Kashyap, K. T. and Chandrashekar, T.: *Effects and Mechanisms of Grain Refinement in Aluminum Alloys*. Bull. Mater. Sci. Vol. 24, No. 4, August 2001, p. 345-353.
- [7] Johnsson, M.: *Grain Refinement of Aluminum Studied by Use of a Thermal Analytical Technique*. Thermochemica Acta 256, 1995, p. 107-121.
- [8] Easton, M. A. and StJohns, D. H.: *A Model of Grain Refinement Incorporating Alloy Constitution and Potency of Heterogeneous Nucleant Particles*. Acta Materialia, 49, 2001, p. 1867-1878.
- [9] Pasciak, K. J. and Sigworth, G. K.: *Role of Alloy Composition in Grain Refining Aluminum 319 Alloy*. AFS Transactions, p. 329-338.
- [10] Sigworth, G. K. and Guzowski, M. M.: *Grain Refining of Hypo-Eutectic Al-Si Alloys*. AFS Transactions, Vol. 93, 1985, p. 907-912.
- [11] Dirnberger, F.: *Einfluss der Begleitelemente auf die Wirkung von Kornfeinung und Veredelung in der Legierung G-AlSi8Cu3*. Diploma work, Fachhochschule Oberösterreich, Wels, Austria, November 2009.
- [12] Campbell, J.: *Feeding Mechanisms in Castings*. AFS Cast Metal Research Journal, 1969, p. 1-8.
- [13] Chai, G.: *Dendrite Coherency During Equiaxed Solidification in Aluminum Alloys*. Chemical Communications. No. 1, Stockholm University, Stockholm, Sweden 1994.