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An Assessment of the Derivative Thermal Analysis of Grey Cast Iron

F. Binczyk *

[a] Chair of Metal Alloys and Composites Engineering, Silesian University of Technology,
Krasińskiego Str. 8, 40-019 Katowice, Poland

* e-mail: franciszek.binczyk@polsl.pl

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Abstract

The study presents a method used for assessment of the consistency of results obtained by measurements and calculated by computer from a graph of Derivative Thermal Analysis (DTA) to determine the chemical composition and selected mechanical properties. For an assessment of the degree of consistency obtained between the examined parameters, the analysis of regression and Pearson's correlation based on a linear model were used. The subject of assessment were 72 melts made in a period from October till December 2006. The best consistency was achieved for carbon and silicon content and for the carbon equivalent liquidus CEL - over 50% consistency of the obtained results. In the case of hardness HB and tensile strength R_m only 6 and 13,8 % of the results obtained by measurement and DTA rendered the same values. Therefore, it has been claimed that in such cases the data obtained from DTA curve analysis cannot serve as a tool valid in forecasting of the above mentioned properties. Since the analysed parameters have been reported to lack the required consistency, some doubts may raise also the evaluation of recalescence, the value of which is used as a starting point in determination of the inoculant level. Consequently, some changes were proposed in the method used so far to evaluate the effectiveness of the cast iron inoculation process.

Keywords: DTA diagram, Chemical composition, Hardness, Tensile strength, Correlation diagram, Inoculation

1. Introduction

Testing of mechanical properties on specimens taken from the ready castings can only confirm the situation already existing. What really matters is to have the possibility of making assessment (forecasting) at the stage when the metal is melted, still before moulds are poured. This possibility is offered by the Derivative Thermal Analysis (DTA) [1-3]. Basing on the theoretical analysis of phase equilibrium diagrams and on the practical measurements taken under industrial conditions, some relationships were established between the liquidus/solidus point and solidification time in function of the chemical composition and mechanical properties of the examined alloys [4-6]. An advantage quite unique of this method is the short time in which the data necessary for evaluation of the tested material can be obtained. Still in the course of production cycle, in a time not longer than a few minutes, one can learn if the examined material is or is not consistent with the specification. Unfortunately, not all of the phenomena that take place during alloy crystallisation and solidification can be recorded on the cooling curve. The determination of the temperature of the beginning and end of solidification is often troublesome and ambiguous. Applying in such cases the graph of temperature derivative with respect to time enables more accurate assessment of the processes which take place during solidification, and which on the derivative curve are shown as local maxima and minima and as a derivative crossing the zero value. To these points on the temperature diagram correspond the respective points of phase transformations.

By application of a suitable computer program, it is possible to determine in a relatively short time on a DTA diagram the characteristic points which will correspond to the chemical composition and properties of the examined alloy.

2. Materials and methods of investigation

The investigations were conducted at one of grey cast iron foundry. The subject of the investigations was grey cast iron containing: from 3,25 to 3,45% C, 1,9 to 2,0% Si, from 0,5 to 0,7% Mn, 0,15 to 0,25 % Cr, 0,15 to 0,25 % Cu, up to 0,08% S, and up to 0,1% P. Melts were conducted in induction furnace of mains frequency.

For control of the cast iron melting process, the results of the derivative thermal analysis DTA made on a QuiK-Lab-E apparatus and the results of a spectrometric analysis of the cast iron chemical composition were used. The analysis was conducted in QuiK-Lab type standard cups with tellurium. The content of carbon (and silicon) in cast iron depends strongly on the solidification temperature of the cementite eutectic which is formed under metastable conditions [6]. A suitable computer program displays the measured content of carbon and the value of carbon equivalent CE, both being recorded in the cast history sheet.

It is also necessary to check the value of undercooling DT, or of the - so called - recalescence (DTA analysis in QuiK-Lab cup without tellurium), which will give us information on the volume of inoculant addded to cast iron on tapping [7]. As an inoculant, FeSi75 were used in an amount of 6 kg per ladle, when DT<12, or 8 kg per ladle, when DT>12 (the results are given in the table in Fig. 2).

Simultaneously a sample was cast (the, so called, "coin" test) for spectrometric analysis of the chemical composition.

The same tests were carried out on a mould pouring stand. If DT<12, to the stream of cast iron during pouring a loose inoculant was fed by special feeder in an amount of 0,20 % per 80 kg of molten cast iron. On the other hand, when DT>12, the cast iron was additionally treated with an inoculant in the form of rods (elastic wire).

3. The results of investigations

Examples of the results of thermal analysis made for the cast iron to evaluate its chemical composition and the value of recalescence ΔT are shown in Figures 1 and 2.

In Table 1 (Fig. 1) the results of spectrometric analysis of the carbon and silicon content and of the computed value Sc are compared with the same data read out from the DTA diagram.

Table 1. Results of chemical composition and mechanical properties

Method	Parameter					
	% C	% Si	Sc	CE	HB	RM, MPa
Measur.	3,33	1,96	0,9123	3,93	226	286
ATD	3,39	1,90	0,9255	4,04	210	252

The results presented in Table 1 indicate quite significant differences in the values measured (true) and read out from the DTA diagram. To determine the degree of correlation between the parameters given in Table 1, 72 melts made in the period from October till December 2006 were examined.

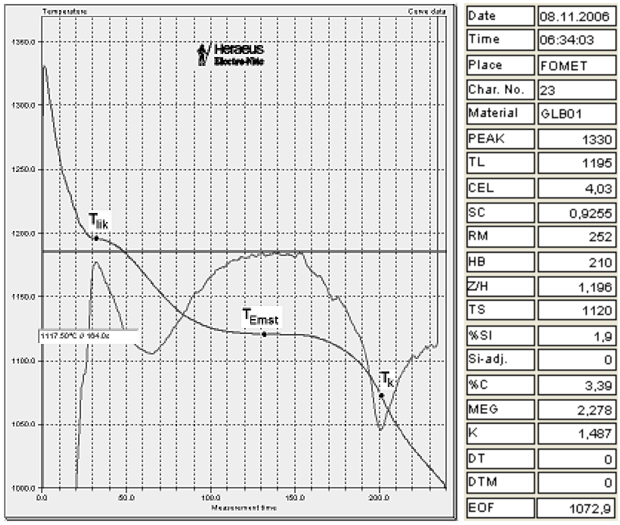

Fig. 1. DTA diagram for C and Si content estimation

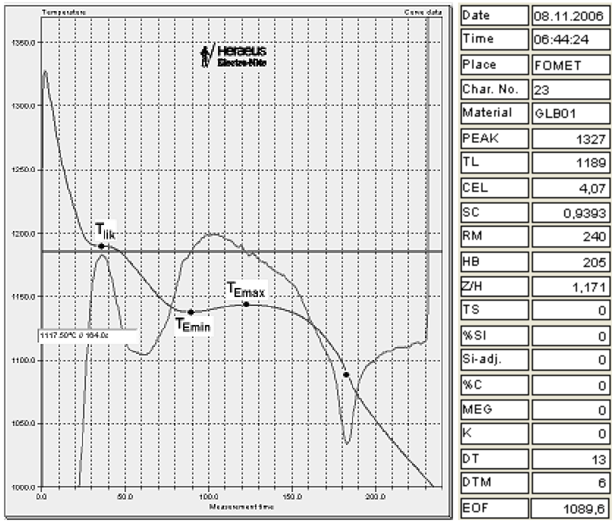

Fig. 2. DTA diagram for recalescence ΔT estimation

Below an example is given of the computation cycle to establish a correlation between the carbon content as determined by spectrometric analysis and read out from the DTA diagram.

At the very beginning, an initial correlation diagram was plotted, and a “cloud of points” was obtained. When the shape of this “cloud of points” differs substantially from a circle, the continuation of computations is necessary [8].

For an assessment of mutual correlations that are supposed to exist between the carbon content determined by spectrometry and the value calculated by computer from the DTA diagram, the analysis of regression and Pearson’s correlation based on a linear model were used. In estimation of the statistical significance, the level of $p < 0,05$ was adopted.

The results of an estimation of the parameters of a monofactorial function of regression are shown below [9]:

R= 0,7080 R^2= 0,5012
F(1,69)=69,350 p<0,00000 est. stand. error 0,02791

	BETA	stand. err. BETA	B	B	t(69)	insulation level p
Free term			0,867	0,294	2,947	0,0044
C_{ATD}	0,708	0,085	0,730	0,088	8,328	0,0000

Final diagram with the correlation equation and plotted area of the significance level is shown in Figure 3.

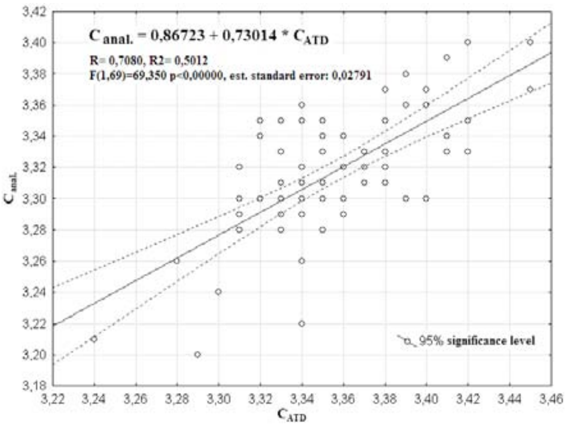

Fig. 3. Correlation diagram for C content

The same correlation was computed for the silicon content, carbon equivalent liquidus CEL, hardness HB and tensile strength R_m. The results are shown in Figures 4 to 7.

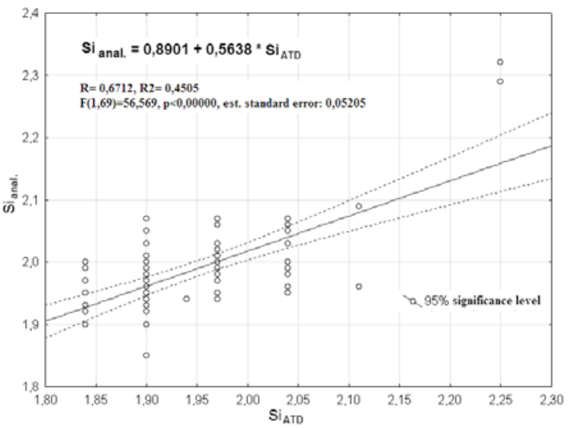

Fig. 4. Correlation diagram for Si content

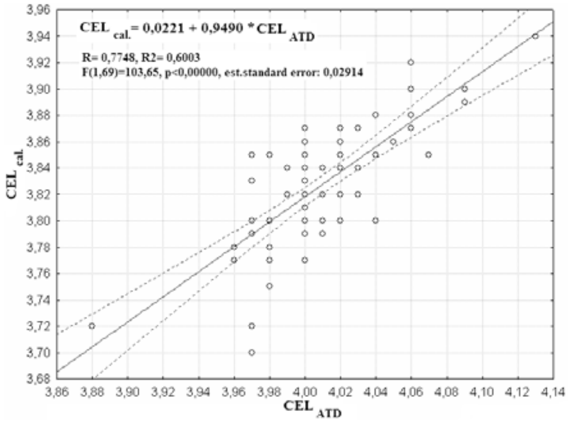

Fig. 5. Correlation diagram for CEL value

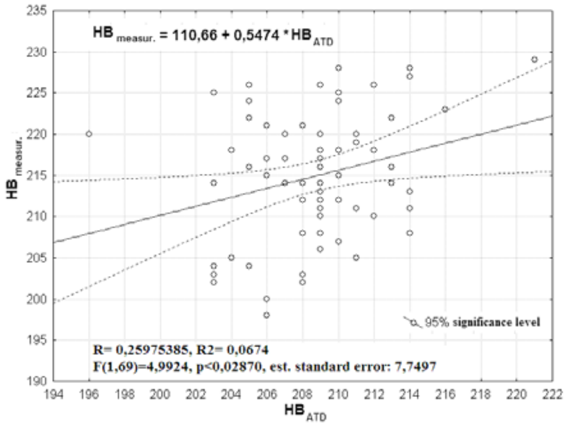

Fig. 6. Correlation diagram for HB hardness

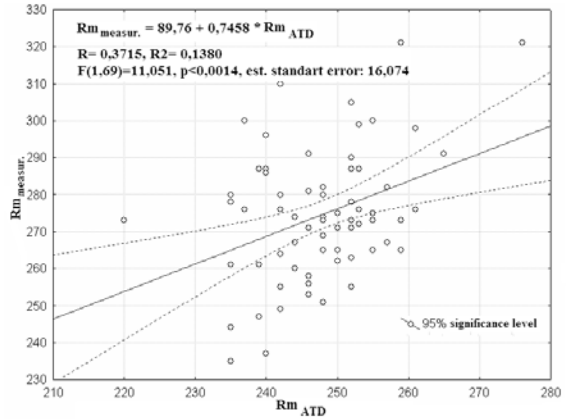

Fig. 7. Correlation diagram for tensile strength R_m

4. Analysis of the results

From the results of the computations it follows that the degree of correlation obtained between the examined parameters is very diversified. The smallest divergences in the results are obtained for the chemical composition, i.e. the content of carbon and silicon, and hence also for the carbon equivalent liquidus CEL. The value of the correlation coefficient R, and specially of the parameter R^2, is sufficient for correct reasoning about the consistency of results obtained in industrial practice.

For carbon - the value of R^2 is 0,50, for silicon - 0,45, for CEL - 0,60. This means that at least one half of the results obtained for these parameters is consistent, no matter whether calculated from actual measurements or read out from the DTA diagram. In the case of hardness HB and tensile strength R_m, the value of R^2 amounts to 0,06 and 0,138, respectively. This means that only 6 and 13,8 % of the results evaluated by measurements and the DTA analysis are consistent. Therefore, in this case, it has been claimed that the data obtained from DTA diagram cannot be used as a tool in forecasting of these properties. The determination of the value of DT and DTM is often difficult because of frequent "failures" of the thermocouple placed in a quartz tube inside the probe. If this happens, sudden "leaps" appear on the derivative curve. The determination of the values of DT and DTM is in such cases impossible, and the results computed for these parameters by a computer program are not true. In view of the reported lack of correlation between the examined parameters, some doubts must also raise the evaluation of recalescence, the value of which is a basis for determination of the amount of inoculant added to cast iron.

The computer program gives two values of the recalescence:

- DT - difference between the equilibrium eutectic temperature under stable conditions (generally assumed to be at a level of about 1150^{o}C) and the minimum temperature of eutectic solidification (recorded by the measuring apparatus),
- DTM – difference between the maximum and minimum temperature of eutectic solidification (recorded by the measuring apparatus).

The DTM parameter seems to be more reliable in determination of the recalescence under industrial conditions. The authors verified this parameter when used to assess the amount of inoculant added to cast iron, adopting as a reference point the size of the chill zone obtained in a wedge chill test.

The determination of the chill zone and the results of the Derivative Thermal Analysis (DTA) have proved that the evaluation of cast iron inoculation effectiveness based on the value of recalescence is not fully satisfactory. The value of the recalescence DT of the cast iron after it had been handled to a stand for mould pouring, was still the same or higher even than the value obtained in furnace before the inoculation (the results of measurements). Fading of the inoculation effect was due to the time elapsing until the next test was made on a stand for mould pouring. The results of the wedge chill test generally indicated a satisfactory effectiveness of the cast iron inoculation. The largest zone of chill on the wedge fracture gave the cast iron held in induction furnace, i.e. still before inoculation. This zone has shrunk considerably in the wedge specimens after inoculation on a stand for mould pouring, to finally assume the smallest size after an in-mould inoculation. For this test the cast iron was taken from a pouring basin.

In view of the above, the authors have proposed the following changes in the process organisation scheme:

- the operation of inoculation should be carried out when the cast iron is poured from a transportable ladle on the stand for mould pouring; the effectiveness of inoculation should be checked by application of the Derivative Thermal Analysis DTA and wedge chill test,
- the DTA analysis should be made immediately after the cast iron has been poured into a mould, using for this purpose the cast iron taken from a pouring basin. Due to this it should be possible to make quick adjustments in the amount of the inoculant introduced to the cast iron stream.

5. Conclusions

Summing up, it should be stated that the application of thermal analysis in control of the technological process of melting and pouring the grey cast iron for brake discs does not stand up to the expectations. The obtained results lack the required consistency as regards the chemical composition (the content of carbon and silicon) and are not acceptable as regards the mechanical properties. The computer program should be subjected to verification to adjust it better to the cast iron grades melted by the foundry, differing quite considerably in the chemical composition, specially in carbon content. To improve both the effectiveness of the inoculation process as well as its control it is necessary to introduce some definite changes to the technological process of iron casting and to conduct periodical thermal analysis DTA on the stand for mould pouring.

References

[1] S. Jura, Z. Jura : Calorimetric curve and heat source in thermic and derivation analysis of cast iron solidification, Solidification of Metals and Alloys, No 16, Gliwice, (1992), 126-139 (in Polish).

[2] S. Pietrowski, B. Pisarek: Estimation of chromium cast iron structure by DTA method, Solidification of Metals and Alloys, No. 16, Gliwice, (1992), 179-190, (in Polish).

[3] M. Stawarz , J. Szajnar: Evaluation of ductile cast iron quality with DTA method, Archives of Foundry, vol. 3, No 10, Katowice, (2003), 199-206, (in Polish).

[4] Pietrowski S, Władysiak R.: Assessment of cast steel crystallization with DTA method, Archives of Foundry, vol.3, No 8, Katowice, (2003), 293-300, (in Polish).

[5] F. Binczyk: Evaluation of AK11 silumin refining and modification by simultaneous DTA and DTA thermical analysis method, Solidification of Metals and Alloys, No 32, Katowice (1997), 170-177. (in Polish).

[6] C. Popdrzucki: Cast iron-structure and properties, ZG STOP, Kraków, (1991), (in Polish).

[7] E. Fraś, Crystallization of metals, WNT, Warsaw, 2003 (in Polish).

[8] M. Maliński, J. Szymszal, Współczesna statystyka matematyczna w medycynie w arkuszach kalkulacyjnych. Wyd. Śl.A.M., Katowice (1999), (in Polish).

[9] T. Gerber: Statystyczne sterowanie procesami – Doskonalenie jakości z pakietem STATISTICA, Wyd. StatSoft, Kraków (2000), (in Polish).