THERMAL ANALYSIS OF INOCULATED DUCTILE IRONS

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Modificarea grafitizantă (inocularea) în formă (aliaje Ca-FeSi şi Sr-FeSi) are un efect puternic asupra solidificării fontelor tratate cu magneziu. În procesul de solidificare, cei mai reprezentativi parametrii ai analizei termice au fost îmbunătăţiţi prin inoculare, precum temperaturi eutectice ridicate, subrăciri scăzute şi valori ridicate ale temperaturii la sfârşitul solidificării. A fost considerată eficienţa relativă a modificatorilor grafitizanţi, prin intermediul căreia a fost găsit faptul că sistemele Ca,Ba-FeSi şi Ca,RE,S,O-FeSi sunt foarte performante. Aliajul Ca,RE-FeSi ce include şi S şi O poate fi o soluţie pentru producerea pieselor turnate din fontă cu grafit nodular cu pereţi subţiri, deoarece procesul de solidificare este influenţat benefic pe întreaga sa durată.

Late in-mould inoculation (Ca-FeSi and Sr-FeSi alloy systems) appears to have a very strong effect on subsequent solidification of Mg-treated irons. As for solidification pattern, the most representative parameters of thermal analysis were improved by inoculation, such as higher eutectic temperatures, lower undercooling and higher temperature at the end of solidification. The inoculant's relative performance was considered which further confirm that Ca,Ba-FeSi inoculant and Ca,RE,S,O-FeSi inoculant give a very high performance. S and O bearing Ca,RE-FeSi alloy appears to be a solution for thin wall ductile iron castings production, as solidification pattern is beneficially influenced on entire eutectic solidification range.

Keywords: Ductile iron, thermal analysis, inoculation, relative performance

1. Introduction

Ductile iron remains a material of choice in automobile industry owning to its unparallel favorable features: it can be produced by casting in large amount at low cost, excellent castability, superior damping capacity and good combination of strength, ductility and toughness has made this iron to experience rapid growth rate. Inoculation which is a means of controlling the structure and properties of cast irons by increasing the number of nucleation sites available for the growth of graphite nodules is an important part in the treatment enacted to obtain the desired result. Inoculation treatment is very important because it improves the

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homogeneity of the cast iron structure, it helps to eliminate the formation of carbides into thin parts or salient angles and promote the formation of graphite during eutectic solidification, direct the solidification towards the stable diagram, with graphite precipitation, refine the structure (higher cells count, finer grains in the microstructure) etc.

In recent times thermal analysis became an important tool to evaluate the solidification behavior of cast iron. Using thermal analysis becomes imperative as a result of rising demand of industries to precisely use the needed materials which will ultimately save cost and materials. Secondly, the quest for new technology that will completely remove manual method of predicting metallurgical properties has been the driven force. Many researchers have concluded that the shape of a cooling curve measured by a thermocouple mounted in thermal analysis sample cup reflect the solidification process of melt iron in the sample cup.

It can be summarized that a lot of factors influence the solidification process, such as normal chemical composition, nodulisation treatment and inoculation which control the shape of the cooling curve. In-depth understanding of the solidification pattern will help to notice some specific points on the cooling curve, where chemical composition and metallurgical structure of the cast irons may be determined as long as the solidifying process is monitored accurately and also to determine the microstructure and mechanical properties of castings. Any little variation in iron melt will result in changing the shape of the cooling curve [1-5].

In the present work, the use of thermal analysis parameters were employed to evaluate the solidification pattern in cooling curve as well as cooling rate during various phases of solidification. Some inoculants were used as case study and the determination of their relative performance clearly give the true picture of each alloy [6].

2. Experimental procedure

The charge consists in a synthetic base cast iron - made without any pig iron. It was melted in an acid lining coreless induction furnace with 100 kg, 2400 Hz frequency, 100 Kw. It was superheated to 1550^{o}C and maintained for 8 minutes. The melt was further superheated to 1561-1563^{o}C and thereafter tap into the Tundish Cover nodulising ladle (2.5wt.% FeSiMgRE alloy).

In-mould inoculation (0.5wt% inoculant) was used as the inoculants were added into non-tellurium standard Quik-Cup^{TM}. The time of mould (cup) filling was 3-4 sec, while the total time of iron melts processing (tapping, Mg-treatment, desslaging, samples pouring) was 3.0-3.5 min.

Table 1 shows the spectrometric chemical analysis of final, hyper-eutectic (CE = 4.49%) ductile iron while Table 2 includes the chemical composition of the experimental alloys, used as Mg-treatment alloy and inoculants. The solidification

Table 1

Spectrometric chemical analysis of final ductile iron (wt.%)

C	**Si**	**Mn**	**P**	**S**	**Mg**	**Mo**	**Ni**	**Al**	**Co**
3.56	2.78	0.470	0.0202	0.0084	0.0505	0.0042	0.0234	0.0201	0.0047
Cu	**Nb**	**Ti**	**V**	**W**	**Pb**	**Sn**	**Cr**	**Zr**	**Fe**
0.0397	0.00053	0.0052	0.0017	0.0026	0.00061	0.0039	0.0384	0.0034	92.9

Table 2

Chemical composition of the experimental alloys (wt.%)

Role	**Alloy Type**	**Si**	**Ca**	**Al**	**Ba**	**Zr**	**Sr**	**RE****	**Mg**	**Fe**
Nodu-liser	Mg-FeSi	46	1.2	0.61	-	-	-	0.96	6.05	bal.
Inocu-lants	Ca-FeSi	76	0.75	1.25	-	-	-	-	-	bal.
	Ca,Ba-FeSi	76	1	1	1	-	-	-	-	bal.
	Ca,Zr-FeSi	76	2.3	1.25	-	1.6	-	-	-	bal.
	Ca,RE,S,O-FeSi^x	73	1	1	-	-	-	1	-	bal.
	Sr-FeSi	76	0.1	0.5	-	-	0.8	-	-	bal.
	Sr,Zr-FeSi	76	0.1	0.5	-	1.25	0.8			bal.

^{x} Up to 1.0 wt. % S and O; **RE – Rare Earth

process was investigated by Quik-cup^{TM} cooling curve analysis (Fig. 1) having a modulus of approximately 0.75 cm (equivalent to 30 mm diameter bar). The cooling curves and their first derivatives were recorded, both for un-inoculated and inoculated irons (Fig. 2).

3. Results and discussion

The cooling curve itself as well as its first derivative and related temperatures can be used to predict the characteristics of the iron. The optimal cooling curve varies for different castings (due to their configuration) and various types of mould material (due to mould stability). A cooling curve is often easier to interpret if the first derivative is plotted. It represents the cooling rate and it is useful in detecting various events during solidification.

If the curve value at a given point is negative it means that the basic curve slopes downwards (Fig. 1). The horizontal line in the middle is where the cooling rate is zero. When dT/dτ=0 it means that heat generated inside the sample just balances the heat [1,2].

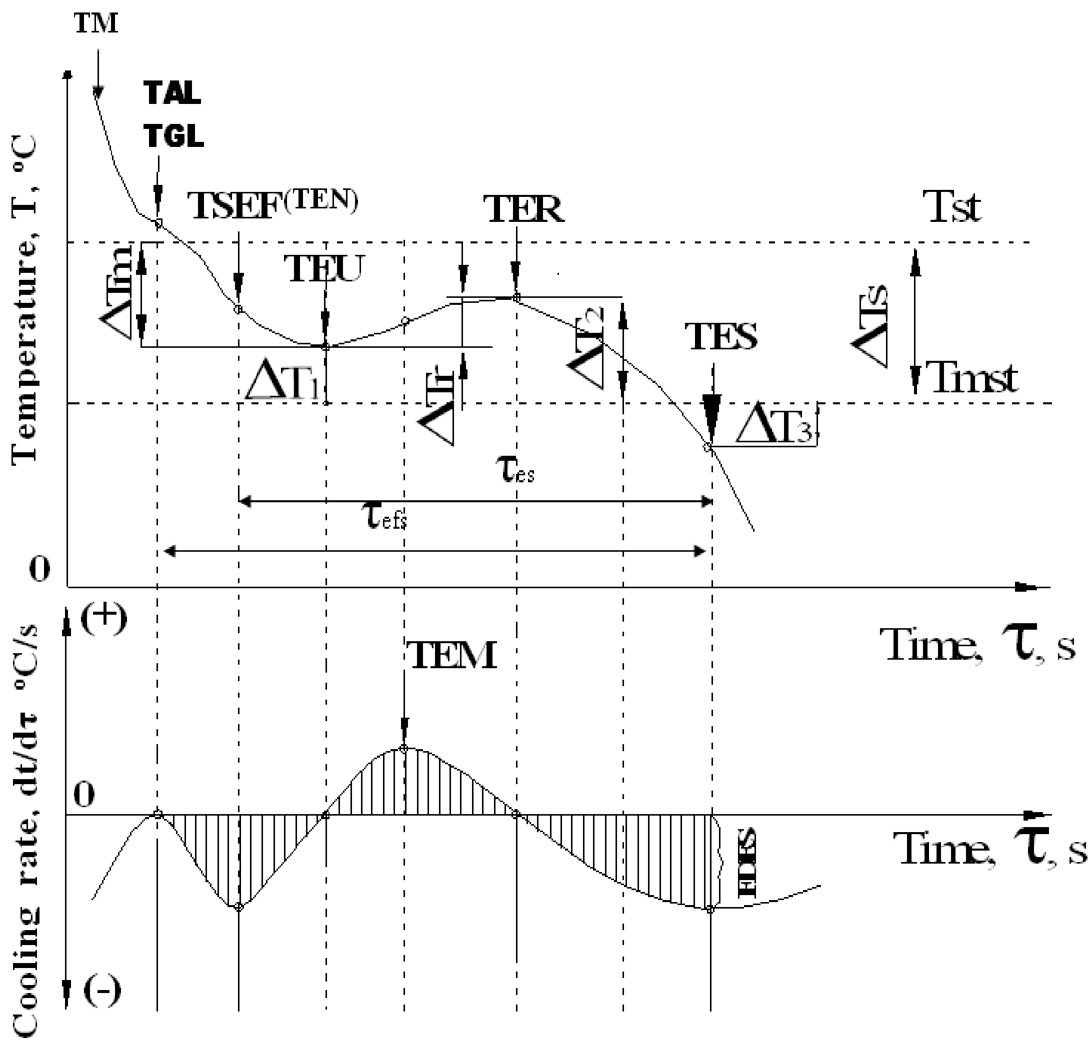


Fig. 1. Typical cooling curve and its first derivative of cast iron

TM - maximum temperature of the poured melt, °C; **TAL -** temperature of austenitic liquidus, °C; **TGL** - temperature of graphitic liquidus, °C; **TSEF (TEN)** - temperature of the start of eutectic freezing (nucleation), °C; **TEU** - temperature of eutectic undercooling, °C; **TER** - temperature of the graphitic recalescence, °C; **TES** - temperature of the end of solidification (end of solidus), °C; **TEM** -

maximum recalescence rate, ^{o}C/sec; **Tst** – graphitic eutectic equilibrium temperature, ^{o}C; **Tmst** – carbidic eutectic equilibrium temperature, ^{o}C; **ΔTs** – range of equilibrium eutectic temperature (ΔTs = Tst - Tmst), ^{o}C; **ΔTm** – maximum degree of undercooling (ΔTm = Tst - TEU), ^{o}C; **ΔTr** – recalecence degree (ΔTr = TER - TEU), ^{o}C; $\boldsymbol{\tau_{es}}$ – duration of eutectic solidification, sec; $\boldsymbol{\tau_{efs}}$ – duration of total solidification, sec; **FDES -** minimum value of the first derivative of cooling curve at the end of eutectic solidification, ^{o}C; $\Delta \mathbf{T_1}$ = TEU - Tmst; $\Delta \mathbf{T_2}$ = TER **-** Tmst; $\Delta \mathbf{T_3}$ = TES - Tmst.

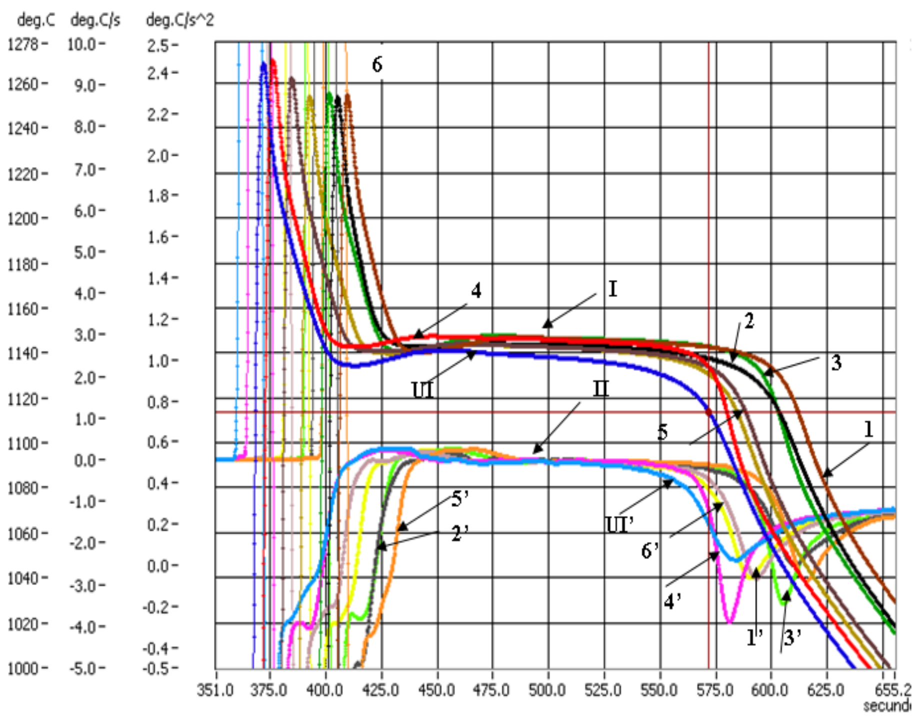


Fig. 2. General view of the cooling curves [I] (UI, 1, 2, 3, 4, 5, 6) and their first derivatives [II] (UI', 1', 2', 3', 4', 5', 6'); UI - Uninoculated iron, 1 - Ca-FeSi, 2 - Ca,Ba-FeSi, 3 - Ca,Zr-FeSi, 4 - Ca,RE,S,O-FeSi, 5 - Sr-FeSi, 6 - SrZr-FeSi

Because the minor element levels were carefully controlled by the metallic charge purity, the graphite eutectic equilibrium temperature (Tst) and carbide eutectic equilibrium (white) temperature (Tmst) (Table 3) were mainly considered a function of the silicon content [1]:

$$\text{Tst} = 1153^{o}\text{C} + 6.7\ (\%\text{Si}) \quad (1)$$

$$\text{Tmst} = 1147^{o}\text{C} - 12\ (\%\text{Si}) \quad (2)$$

During solidification, a considerable amount of heat is evolved by the formation of austenite in hypo - eutectic iron and graphite in hyper - eutectic iron. The amount of heat evolved was documented by measuring the temperature change in a standard sample, thereby obtaining an accurate measure of the melt's carbon equivalent. Thermal analysis is very useful in detecting various events of cast irons during solidification [7-15].

Table 3

Eutectic temperatures and eutectic range

Param.	Signification	Comments
Tst	Stable (graphite) eutectic equilibrium temperature	*Theoretical temperature for C to precipitate as graphite; *It should be as high as possible.
Tmst	Metastable (white) eutectic equilibrium temperature	*Temperature when C is chemically combined with iron (Fe_3C); *It should be as low as possible.
ΔTs	Range of equilibrium eutectic temperature [ΔTs = Tst - Tmst]	*ΔTs should be as large as possible; *Favourable elements: Si, Ni, Cu, Co, Al.

Just after pouring the melt the cooling begins. When the liquidus temperature referred to as austenitic (TAL) or graphitic (TGL) liquidus is reached, the cooling curve shows a quasi-horizontal plateau which corresponds to zero point on the first derivative. This zero point means that the heat losses at that time exactly balance the released heat in the sample (Table 4).

The length of the horizontal plateau is a function of the time it takes for the graphite to grow from the walls of the cup to the center where the thermocouple is located. Then, there is contraction of the melt at both the liquidus state and during the crystallization of the primary graphite. However, the decrease of temperature continues until eutectic freezing starts at TSEF which is the minimum point on the first derivative and this continues until the lowest eutectic temperature TEU. At that time the eutectic reaction where simultaneously austenite and graphite are precipitated has just started.

The lowest eutectic temperature (TEU) – which is reached when the heat generated from recalescence specific heat and latent heat just balances the heat losses – is shown as a zero-point on the first derivative curve. At this zero-point, the eutectic reaction occurs, and the recalescence energy causes the temperature to increase to the temperature of graphite recalescence (TER). A second zero-point on the first derivative curve then occurs. The difference between (TER) and (TEU) is called recalescence (ΔTr).

Usually, the degree of eutectic undercooling (ΔTm) is defined as the difference between the eutectic temperature in the stable system (Tst) and the lowest produced eutectic temperature (TEU). Other parameters were also introduced [8 - 15], which consider the produced lower (TEU) and higher (TER) eutectic temperature and the calculated metastable (white) eutectic temperature (Tmst).

ΔT_1 = TEU - Tmst parameter illustrates the position of irons at the start of eutectic reaction, while ΔT_2 = TER - Tmst at the end of this stage. If $\Delta T_1 < 0$ chill and white iron will appear.

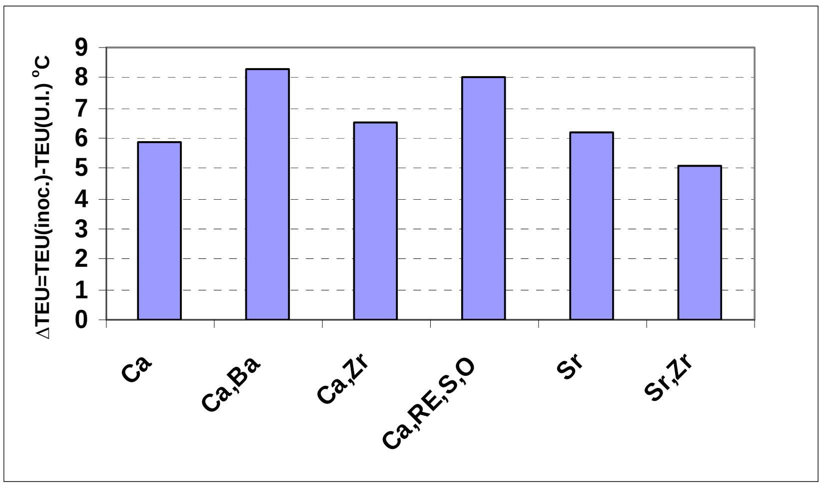
Table 4

Eutectic solidification parameters

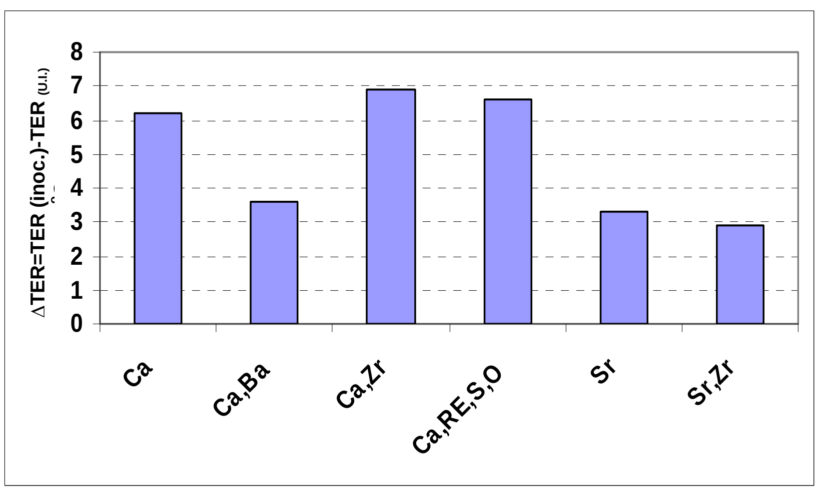
Param.	Signification	Comments
TAL TGL	Liquid temperature commences solid precipitation	*First arrest temperature (no recalescence has occurred); *The first derivative is zero. *TAL - austenitic and TGL - graphitic liquidus.
TSEF	Temperature of the start of eutectic freezing (nucleation)	*Derivative has a minimum, between TAL/TGL and TEU; *It should not be too deep.
TEU	Lowest eutectic temperature	*The minimum point from which the temperature is increasing; *The first derivative is zero; *Inoculation increases TEU.
TER	Highest eutectic temperature	*The maximum eutectic temperature after the increase in temperature; *The first derivative is zero; *High cooling rates may not achieve this temperature
ΔTm	Conventional eutectic undercooling degree [ΔTm = Tst – TEU]	*Comparing to graphite eutectic temperature (Tst); *The maximum eutectic undercooling; *A high undercooling means: -Free carbides (chill) if $\Delta Tm > \Delta Ts$ *Higher the ΔTm of base iron, the higher the need for inoculation; *Inoculation reduces eutectic undercooling.
ΔT_1	Undercooling comparing to Tmst [ΔT_1 = TEU – Tmst]	*Beginning of eutectic reaction; *Carbides (chill), if $\Delta T_1 < 0$ [TEU < Tmst]; *Inoculation increases ΔT_1 parameter.
ΔT_2	Undercooling comparing to Tmst [ΔT_2 = TER – Tmst]	*End of eutectic reaction, no white iron if $\Delta T_2 > 0$; *Inoculation increases ΔT_2, at lower power comparing to ΔT_1 parameter.
ΔT_r	Recalescence Degree [ΔTr = TER – TEU]	*It reflects the amount of austenite and graphite that are precipitated during the first part of eutectic freezing; *Too high recalescence might be harmful, in soft moulds; *Ideal values depend in the type of mould and the casting modulus: ΔTr = 2…5^{o}C, as a guideline;
TEM	Maximum recalescence rate	*Maximum value of the first derivative between TEU and TER

In these experiments, un-inoculated irons mainly presented white iron solidification ($\Delta T_1 < 0$), while inoculation moved the ductile irons to positive values for ΔT_1 in all cases.

Un-inoculated iron is characterized by the lowest TEU and TER temperatures [6, 7]. Although inoculation increases both of these temperatures, the amount of the increase is dependent on the potency of inoculants (Fig. 3).



(a)



(b)

Fig. 3. Undercooling (a) and recalescence (b) difference between inoculated and un-inoculated ductile irons for representative inoculants application

The difference between the TEU temperature of inoculated and un-inoculated iron was at 5 - 10^{o}C range for the experimental conditions. A higher TEU value indicates that eutectic undercooling is at a temperature farther from the (white) carbide eutectic and the metal is therefore more resistant to chill than with a lower TEU value. Each inoculated iron was subtracted from un-inoculated iron as shown in Fig. 3a. It is visibly shown that Ca,Ba-FeSi inoculated iron and Ca,RE,S,O-FeSi inoculated iron showed a high efficiency more than others, while Ca-bearing inoculants are generally more efficient comparing to Sr-bearing inoculants.

The highest eutectic temperature (TER) is attained as a result of increase in temperature because of the release of inherent heat called latent heat. The recalescence degree (ΔTr = TER – TEU) reflects the amounts of austenite and graphite that precipitate during the first part of eutectic freezing. Too high recalescence might be harmful especially in soft moulds, as the volume expansion is high and might increase the size of the mould cavity.

While the ideal recalescence depends on the type of mould material and on the modulus of the castings, a range of no more than 5^{o}C is preferred. Lower level of recalescence will depict high efficiency of inoculants and the risk for micro shrinkage and porosity will be reduced.

On the other hand if the recalescence degree is high, it may indicate undesirable, low nodule count, early graphite expansion that increases the risk for wall expansion effects and primary shrinkage. As far as limited capacity to increase recalescence is concerned, Sr- bearing inoculants appear to be better for ductile iron production in comparison with Ca-bearing inoculants (Fig. 3b).

The final freezing of the casting (Table 5), especially of the eutectic cell boundaries, can happen up to 100^{o}C later, given the metastable (white) eutectic temperature (Tmst), which is not the real point of final solidification. This difference is important to the soundness and properties of the castings.

Table 5

Parameters at the end of solidification

Param.	**Signification**	**Comments**
TES	Temperature of the end of solidification (solidus)	*All metal has solidified; *Lowest value of the negative peak on the first derivative; *Lower (TES), higher sensitiveness to contraction defects and inverse chill incidence.
ΔT_3	Undercooling at the end of solidification [ΔT_3 = TES – Tmst]	*Usually at negative values, as TES < Tmst; *Intercellular carbides, inverse chill and micro-shrinkage occurrence especially if $\Delta T_3 > 20^{o}$C (more negative); *Inoculation normally decreases ΔT_3 and the incidence of contraction defects and intercellular carbides.

FDES	The depth of the first derivative at solidus	*The depth of the negative peak; *It should be less than -3.0 (i.e. deeper) for ductile irons (high amount of graphite at the end of solidification); *Inoculation normally has a positive influence.

After most of the heat of solidification has been used up, the low melting phases in the iron are still liquid. If these phase concentrations are high, the end of freezing temperature will be lower. The difference between the solidus (eutectic freezing) temperature and the end of freezing temperature is then an indicator of the concentration of these low melting point phases.

The difference between the temperature of the end of solidification (TES), which corresponds to the lowest point on the first derivative of the cooling curve (FDES), and the metastable (white) eutectic temperature (Tmst), ΔT_3 = TES - Tmst, is an important parameter for characterizing the behavior of the end of solidification. In the experimental conditions, this parameter had only negative values, but in a very large range distribution.

The relative performance of inoculants to control representative thermal analysis parameters was calculated to determine the efficiency of the experimental alloys [15]. The relative performance (RPi) of inoculants –i- is estimated as:

$$RPi = \frac{\sum_k (X_{ik} - CL_k)}{S_k} \tag{3}$$

where X_{ik} is measured value of property –k- using inoculants –i-;

CL_k is average value for property set –k-;

S_k is standard deviation from the set.

Thermal analysis performance is averaged and used as one parameter –*k*-. Average performance has level 0%. This tool was used to determine and distinct the close performance of the alloys in all the analysis carried out in this work. High and low performance values were mainly used, because some parameter's high values are not beneficial while some are very helpful. High value of the following parameters (TEU, TES, ΔT_1, and ΔT_3) means high relative performance and low value of TER, ΔTr, ΔT_2 and ΔTm equally means high relative performance.

The results showed that some inoculants performed better than the other alloys bearing the same base inoculating element (Ca or Sr). In the same system group, one inoculant is more efficient than the other. Inoculants have different positions for different thermal analysis parameters reference.

Iron inoculated with Ca-bearing FeSi alloys showed generally best performance in these tests as eutectic undercooling control. Increase in lowest eutectic temperature (TEU) which denote that the inoculant has high efficiency was observed in Ca,Ba-FeSi and Ca,RE,S,O-FeSi inoculated irons as they gave relative performance of 156% and 130%, respectively (Fig. 4). The highest eutectic temperature (TER) was less affected by Sr-bearing FeSi alloys, comparing to Ca-bearing alloys (excepting Ca,Ba-FeSi). The lower recalescence is, the higher the efficiency of inoculants. Temperature at the end of solidification

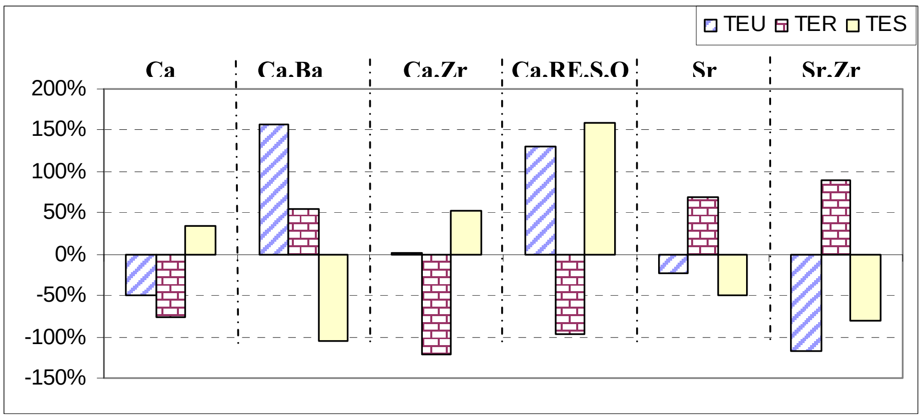


Fig. 4. Relative performance of inoculants in representative temperatures on the cooling curves composition

(TES) was also considered. Ca-FeSi, Ca,Zr-FeSi and Ca,RE,S,O-FeSi inoculants have relative performance of 35%, 53% and 159% respectively. The latter will have the highest efficiency as it has highest relative performance.

The eutectic solidification of inoculated ductile irons were compared, referring to four major parameters of thermal analysis (Fig. 5); maximum degree of undercooling (ΔTm=Tst-TEU); undercooling temperature compared to metastable at the beginning of eutectic reaction (ΔT_1 = TEU - Tmst); undercooling at the end of solidification (ΔT_3 = TES - Tmst); eutectic recalescence degree (ΔTr = TER – TEU).

It is visibly showed (Fig 5) that all the inoculants demonstrate high efficiency with Ca-bearing FeSi alloys appeared to have more beneficial effects. The inoculant’s relative performance was considered which further confirm that Ca,Ba-FeSi inoculants and Ca,RE,S,O-FeSi inoculants give a very high performance while other inoculants showed un-conclusive result. The average of the major parameters was also considered which clearly gives the true pictures of all the inoculants, Ca,Ba-FeSi alloy and Ca,RE,S,O-FeSi alloy gave the best performance.

Generally, the intent of effective cast iron inoculation is to initiate solidification at the highest possible temperature in the eutectic solidification temperature range. Inoculation promotes the stable austenite – graphite solidification and minimizes any austenite – carbide solidification.

In ductile irons, a substantially greater number of eutectic cells are necessary, in order to achieve effective control of solidification. Each graphite spheroid is the result of a separate and distinct nucleation event, as is evidenced

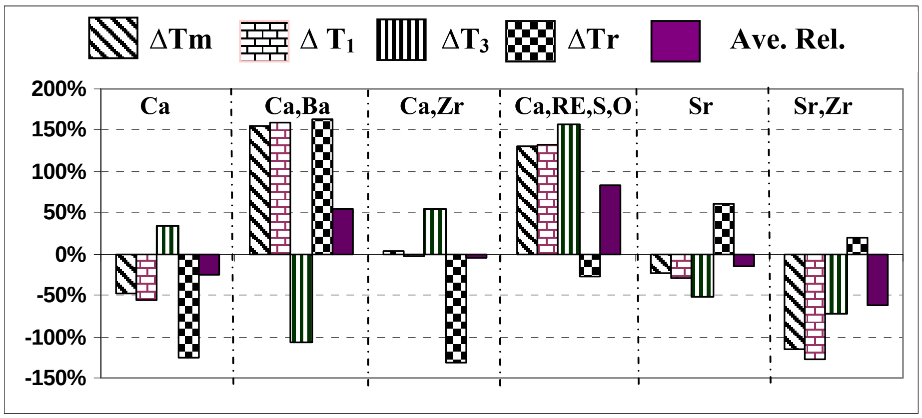


Fig. 5. Relative performance of inoculants as beneficial effects on the representative thermal analysis parameters

by the presence of a substrate particle at the growth center of each spheroid. Since the spheroidal graphite eutectic growth occurs by carbon diffusion through the austenite shell, increased shell thickness will result in increasingly slower solidification rate, with final solidification occurring at lower temperatures [16].

MgS–CaS–CeS compounds are generally formed as the primary microinclusions in the Mg treated iron melt as nucleation sites for the subsequent complex Mg silicates formation, such as $MgO.SiO_2$ and $2MgO.SiO_2$. However, these silicates present a large planar disregistry with graphite and will not act as efficient nucleation sites for graphite.

Subsequent inoculation with FeSi containing Al and X (Ca, Ba, Sr) alters many of these inclusions by forming hexagonal silicate phases of $XO–SiO_2$ or $XO–Al_2O_3–2SiO_2$ on the surface of the previously formed Mg silicates. These new silicates are more favourable sites for graphite nucleation, at lower eutectic undercooling, since they form coherent/semicoherent low energy interfaces between the nucleant and graphite [17].

The complex alloy, as Ca,RE,S,O-FeSi system, appears to be the most efficient inoculant in ductile iron production. This alloy contributed not only in efficient inoculating elements (Ca, Ce, La etc), but also in sulphur and oxygen, beneficial in the formation of sulphides and oxides. It is able to control the cooling process and to promote a lower undercooling at the beginning and the end of eutectic solidification, to limit free carbides (chill) formation and contraction defects occurrence, respectively. This inoculant is recommended especially for thin wall castings production and/or for castings very sensitive to macro-shrinkage and micro-shrinkage formation [12].

4. Conclusions

- A synthetic base cast iron-made without any pig iron - undergoes solidification that is characterized by high eutectic undercooling degree after Mg-treatment.
- Late in-mould inoculation appears to have a very strong effect on subsequent solidification of Mg-treated irons.
- Ca-series and Sr-series bearing inoculants are materials that should be considered in ductile iron production, but the former proved to be better, especially for eutectic undercooling control.
- As for solidification pattern, the most representative parameters of thermal analysis were improved by inoculation, such as higher eutectic temperatures and lower undercoolings, higher temperature at the end of solidification and larger maximum rate of eutectic freezing at this temperature.
- The use of relative performance is a tested tool to distinguish alloys of losed efficiency. On the average, Ca,Ba-FeSi inoculant and Ca,RE,S,O-FeSi inoculant efficiency was clearly differenciated with 54% and 84%, respectively.
- The distinction in alloys efficiency are better enhance with the use of relative performance setting the special alloy Ca,RE,S,O-FeSi inoculant as the best as it gave the most positive influence.
- S and O bearing Ca,RE-FeSi alloy appears to be a solution for thin wall ductile iron castings production, as solidification pattern is beneficially influenced on entire eutectic solidification range.

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