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# The Effect of Successive Stages of the Melting Process on the Nucleation of Ductile Cast Iron

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## Abstract

The article discusses issues related to the melting of grey and ductile cast iron in terms of metallurgical quality. The derivative and thermal analysis (DTA) was used to assess this quality. The article presents the results of research carried out in industrial conditions and analysed by the Itaca system. In the paper, the effect of the furnace type, the charge materials and the inoculation process on the parameters characterising the cast iron being melted was analysed. The most important of these are the minimum eutectic temperature ($Te_{min}$), the liquidus temperature ($T_{liquidus}$) and the nucleation rate. The results of the research and calculations are shown in graphs and as dependencies. Some of DTA results were compared to the microstructure analysis results. The article shows that the derivative and thermal analysis is a very effective tool in the assessment of the metallurgical quality of cast iron. It is a very good addition to chemical analysis. Based on the results of the research, it was concluded that a very high correlation exists between the rate of nucleation (DTA) and the number of graphite nuclei (microstructure analysis). Furthermore, it was also found that an improvement in nucleation could be achieved by ensuring a high value of carbon equivalent (CE) and, above all, by conducting the primary and secondary inoculation processes, respectively.

**Key words:** Grey and ductile cast iron, Inoculation, DTA, Minimum eutectic temperature ($Te_{min}$), Nucleation

## 1. Introduction

In 2020, the article by D.M. Stefanescu "90 years of thermal analysis as a control tool in the melting of cast iron" was published where it was stated that the beginnings of recording the heating curve as a function of temperature appeared in 1877 and the first applications of thermal analysis (TA) for melting cast iron dated back to 1931 [1]. The initial applications included the rapid determination of a carbon equivalent. The development of electronics and computing techniques has provided conditions for the dynamic development of the method. In Poland, Prof. S. Jura can be regarded as a precursor of thermal analysis (TA) and derivative and thermal analysis (DTA). In 1980, two articles dealing with this issue were published in the Krzepnięcie Metali i Stopów (*Solidification of Metals and Alloys*) magazine [2-3]. The first covered the use of thermal and differential analysis for the determination of chemical composition parameters [2], whereas the latter concerned the differential analysis of grey cast iron solidification and crystallisation processes [3]. Prof. S. Jura and the researchers from the Department of Foundry of the Silesian University of Technology conducted research on iron and aluminium alloys using DTA for several years [4-6].

The achievements of Prof. S. Jura in this respect were summarised in the special edition of the Archives of Foundry Engineering entitled "Professor Stanisław Jura the creator of theory and the industrial uses of diagnostics and wear of metals and alloys" [7]. This subject matter was also being developed at the Lodz University of Technology by Prof. S. Pietrowski and his team. Their work resulted in a number of publications covering the theoretical foundations for the DTA method and its applications for testing various alloys [8-10]. Over the years, this method has been and continues to be used by all academic and research centres dealing with casting issues to describe the physicochemical state of the metals and alloys being melted. Of course, this research is conducted not only in Poland, but also all over the world [11-16].

The result of the scientific research, the development of electronics and the perception of a number of positive features of DTA by foundries, was the development of systems specifically dedicated to industrial applications. Currently, one of the most commonly used systems in foundries is ITACA [17], which was also used in tests described in the article. This system consists of a number of modules that allow the combination of the metallurgical quality assessment and the automation of melting and off-furnace treatment of cast iron (Itaca Meltdeck, Itaca Optidose, Itaca Wire, Itaca Stream). The compilation of the DTA system with the automation of various stages of the cast iron melting process was the subject of the implementation carried out by PGO Odlewnia Żeliwa Śrem (Iron Foundry in Śrem).The DTA systems used in Polish foundries which deserve particular attention include: ATAS [18], QUIK-LAB [19] and FERROLAB [20].

## 2. Progression and methodology of the research

The article presents a section of the results of the research carried out in industrial conditions during the melting of grey and ductile cast iron. The research was performed part of a research and development project co-financed by the National Centre for Research and Development (NCBiR). This made it possible to conduct research in a very wide range of variabilities of many parameters affecting the process indicators. The melts were carried out in an electric double-crucible medium frequency induction furnace with a crucible capacity of 8000 kg. The primary inoculation was made using the Itaca Optidose system, which allows inoculant to be introduced into the stream while transferring metal from the furnace to the ladle. In spheroidisation, the Itaca Wire system (PE flexible wire) was used, whereas the secondary inoculation was also carried out with the Itaca Wire (PE) system and the Itaca XL Stream system, which allows an inoculant to be introduced with the assumed capacity into the metal stream when pouring it into the mould tank. In the experiments, the quality of cast iron melted in the campaign cupola was analysed too.

For each melt, the cooling and crystallisation curves (DTA) were recorded. Tests performed using the Itaca system. The system measures the temperature of the cooling cup(type K thermocoupl). Chemical analyses and metallographic tests were carried out, and for some of the melts the mechanical properties were tested. During the experiments, changes in the characteristic parameters of thermal analysis (nucleation rate, temperatures of $T_{liquidus}$, $Te_{min}$, $Te_{max}$, $T_{solidus}$, VPS ratio, REC (recalescence value) in the individual stages of the cast iron melting process were analysed [15]. Some of these results of research and calculations are provided in this article.

One of the most important parameters is the minimum temperature reached during eutectic solidification ($Te_{min}$). $Te_{min}$ is the most important indicator of cast iron nucleation. The higher this temperature, the better the nucleation level. Below 1135°C, nucleation is considered low and there is a high risk that primary carbides may occur in the casting. Between 1135°C and 1145°C, nucleation is considered optimal. Above 1145°C, nucleation is very good and there is no risk of the occurrence of primary carbides [15]. $T_{liquidus}$ temperature, which is the beginning of the crystallisation process, was also analysed in the research.

The next parameter included in the analysis is VPS, which is the angle formed by the cooling rate and whose value is strongly related to the formation of contraction cavities. In the initial cast iron used for the production of ductile cast iron and in the grey cast iron, this value should be less than 30. For ductile cast iron, it should be between 35 and 55 and it is linked to the magnesium content after the magnesium treatment process [15].

For the primary and secondary inoculation, inoculants with different chemical compositions were used. These were ferrosilicon-based inoculants (FeSi) with the addition of Ba, Sr and Zr, offered by various suppliers, metallurgical silicon carbide (SiC) and graphite carburiser (C). In the secondary inoculation process, inoculants with Bi and Ce were also introduced [15, 16].

## 3. Results of the research

As already mentioned, one of the most important parameters is $Te_{min}$ temperature, which refers to the tendency of the alloy being melted to form carbides and hard spots, and $T_{liquidus}$ temperature, which indicates the point in the iron-carbon equilibrium system the cast iron being melted is at. These temperatures can be recorded thanks to simple temperature transducers and systems. In addition, the ItacaX system provides the nucleation index which determines the metallurgical quality. It is associated with a number of temperatures and characteristic parameters ($T_{liquidus}$, $Te_{min}$, $Te_{max}$, $T_{solidus}$). The value of this index can range between 0 and 100. For the initial cast iron it should be 100. If the result is lower, the nucleation potential of cast iron is worse. It can be increased by carrying out the pre-inoculation (addition of inoculant – ferrosilicon (FeSi), silicon carbide (SiC) or synthetic graphite (C)), primary inoculation or secondary inoculation process.

The research carried out shows that this index is mostly affected by $Te_{min}$ temperature. Fig. 1 shows the change in the value of the nucleation rate as a function of $Te_{min}$ temperature, whereas Fig. 2 presents the effect of $Te_{min}$ temperature on the actual number of graphite precipitates per mm$^2$, obtained from the metallographic tests performed. Research samples of microstructure taken from cast samples. The results of the microstructure analysis are the confirmation of the obtained results. The most changing parameter describing the image of the cast iron microstructure in accordance with the PN-EN ISO 945-1: 2019-09 standard is in this case the determination of the number of precipitates. Fig. 3 shows the cast iron microstructure for $Te_{min}$ 1143ºC Fig. 4 shows the cast iron microstructure for $Te_{min}$ 1155ºC. The research carried out confirmed the very high relationship between $Te_{min}$ and the nucleation index (number of nuclei) obtained from DTA curves and the actual number of precipitates obtained from the metallographic tests. This means that the nucleation rate in systems where it is not calculated can be determined by forecasting based on $Te_{min}$ temperature.

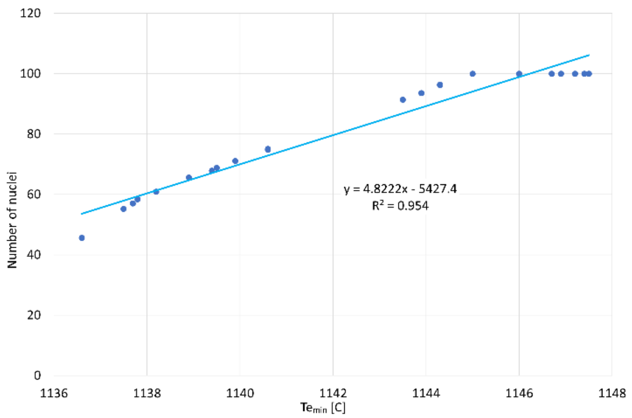


Fig. 1. Number of nuclei as a function of $Te_{min}$ temperature (values derived from DTA curves)

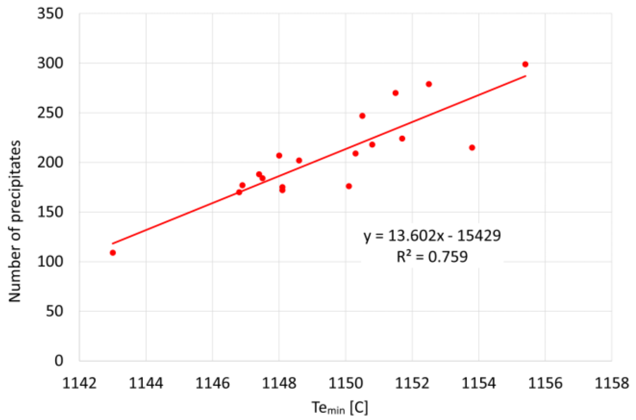


Fig. 2. Number of graphite precipitates in ductile cast iron, obtained in metallographic tests as a function of $Te_{min}$ temperature

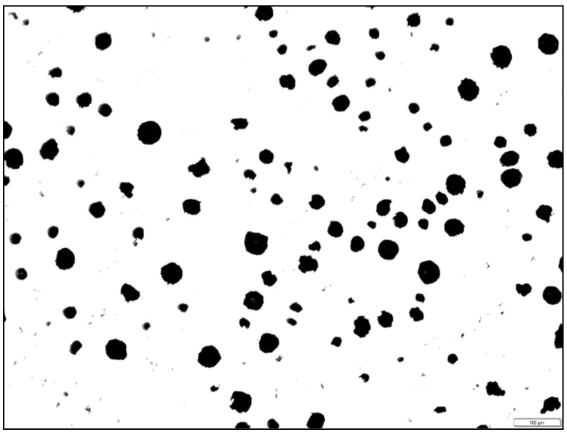

Fig. 3. Image of the cast iron microstructure, 109 graphite precipitates per mm², $Te_{min}$ 1143ºC

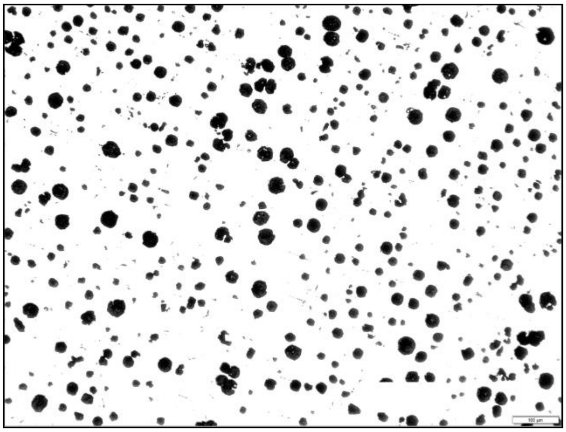

Fig. 4. Image of the cast iron microstructure, 299 graphite precipitates per mm², $Te_{min}$ 1155ºC

One of the factors affecting the metallurgical quality is the type of the metallurgical furnace used [15]. With the coke-fired campaign cupola furnace and DTA apparatus, the cooling and crystallisation curves for the cast iron taken from the cupola spout (22 measurements) were analysed. Contrary to the general expectations that cast iron from the cupola furnace demonstrates the best tendency to nucleation, the obtained values of $Te_{min}$ temperature were relatively low. The reason is the low C and Si contents and consequently the low carbon equivalent (CE) value. The examples of chemical compositions and the values of carbon equivalent and $Te_{min}$ and $T_{liquidus}$ temperatures are given in Table 1, whereas the cooling curves are shown in Fig. 5.

Table 1.
Chemical composition of cast iron melted in cupola furnace, wt%

| CE | C | Si | P | S | $Te_{min}$ | $T_{liquidus}$ |
|---|---|---|---|---|---|---|
| 3.7 | 3.21 | 1.37 | 0.093 | 0.077 | 1126.1 | 1211 |
| 3.8 | 3.24 | 1.64 | 0.102 | 0.080 | 1128.2 | 1200 |
| 3.9 | 3.27 | 1.80 | 0.098 | 0.080 | 1129.9 | 1208 |
| 4.0 | 3.31 | 1.97 | 0.112 | 0.088 | 1132.7 | 1180 |
| 4.2 | 3.40 | 2.30 | 0.106 | 0.086 | 1136.2 | 1165 |
| 4.3 | 3.50 | 2.21 | 0.116 | 0.092 | 1135.4 | 1171 |

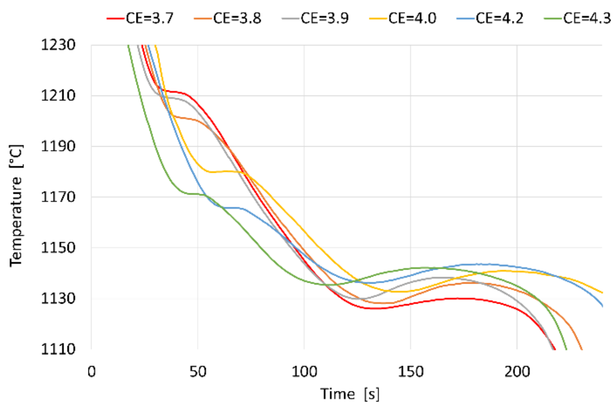


Fig. 5. Cooling curves for variable value of carbon equivalent (CE)

Fig. 6 shows the relationship of the minimum eutectic temperature (for all melts) as a function of carbon equivalent (CE), whereas Fig. 7 shows the relationship of $T_{liquidus}$ temperature as a function of carbon equivalent (CE)

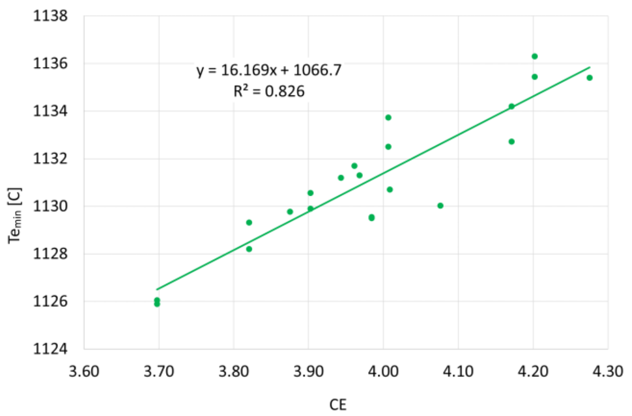


Fig. 6. Minimum eutectic temperature($Te_{min}$) as a function of carbon equivalent (CE)

The research mainly included the melting of cast iron from a solid charge in a medium frequency electric induction furnace. Cast iron melting experiments were performed for different melting power settings in terms of melting time.

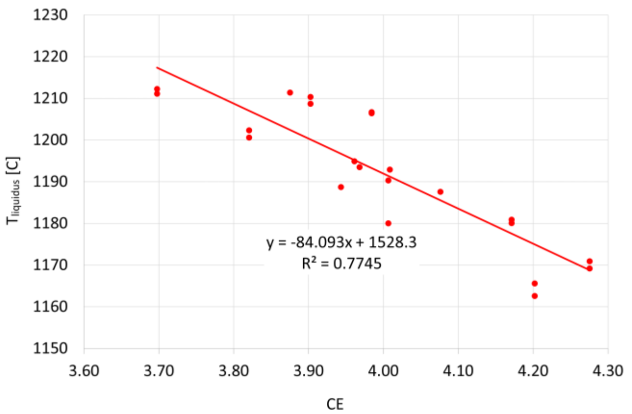


Fig. 7. $T_{liquidus}$ temperature as a function of carbon equivalent (CE)

The purpose of these tests was also to determine the limits of the melting time for which the initial cast iron is nucleated at above 80% and the minimum eutectic temperature is above 1140°C. The results of the research and calculations are given in Fig. 8 and 9.

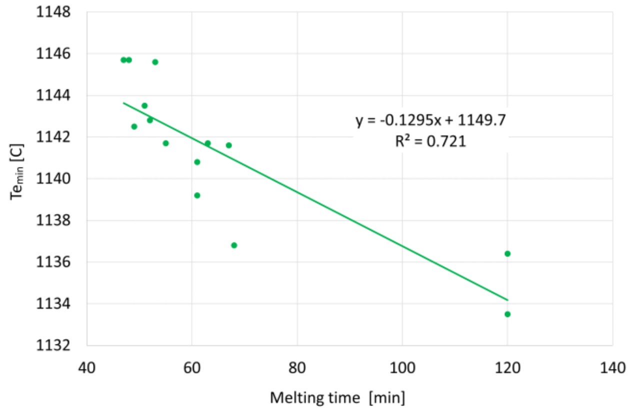


Fig. 8. Effect of melting time on the minimum eutectic temperature ($Te_{min}$)

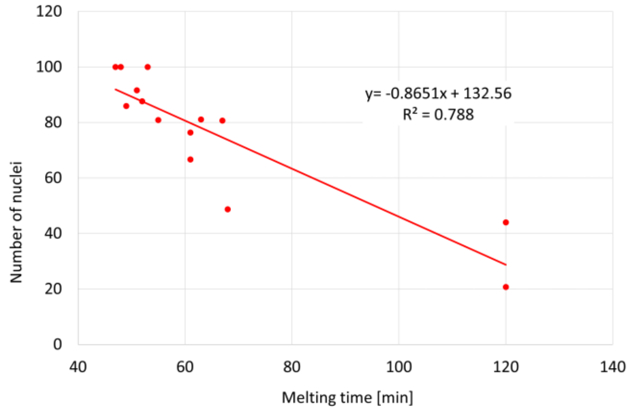


Fig. 9. Effect of melting time on the number of nuclei

The results of the research showed that prolonging the melting time resulted in a decrease in $Te_{min}$ temperature and the nucleation rate. In order to ensure the assumed values of these parameters, the melting time should not be longer than 60 minutes. Increasing the melting time up to 120 minutes will result in the reduction in the nucleation rate even to 20.

The next stage of the cast iron melting process is the primary inoculation, most often carried out while transferring metal from the furnace to the ladle. The own research confirmed that the most effective method was to introduce the inoculant into the stream of poured cast iron. As part of the research, the selected inoculants were tested by introducing them into the metal stream at an amount of 0.1% using the automated dispensers of the Itaca Optidose system. Synthetic graphite (Graph), silicon carbide (SiC), FeSiBa, FeSiZr and FeSiSr were used in the tests. The effect of the inoculant used on the change in $Te_{min}$ temperature and nucleation rate was analysed. The carbon equivalent ranged between 3.86 and 3.92 for all melts. The tests were carried out during the melting of GJL 250 grey cast iron. The cooling curves for individual inoculants are shown in Fig. 10.

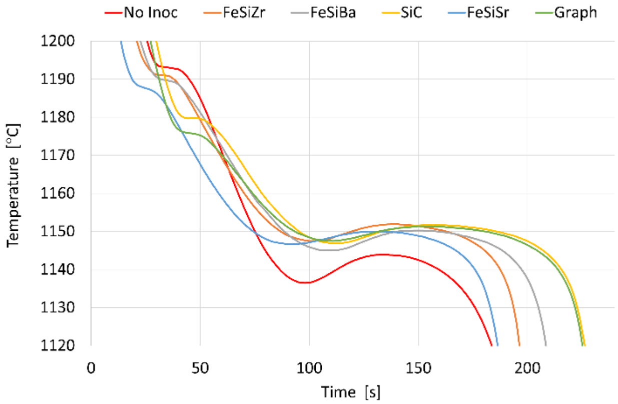


Fig. 10. Cooling curves without and after primary inoculation

Fig. 11 presents the change in $Te_{min}$ temperature after the inoculation process and Fig. 12 shows the effect of the inoculation on the number of nuclei. The red bars refer to temperatures before the process and the blue bars to temperatures after the process.

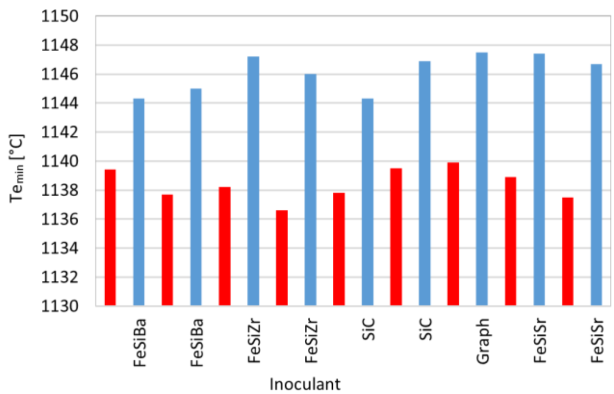


Fig. 11. Minimum eutectic temperature ($Te_{min}$) after the introduction of different inoculants

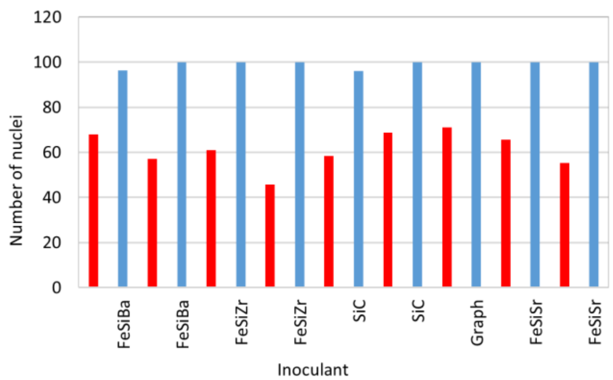


Fig. 12. Change in the number of nuclei after the introduction of different inoculants

In the case of ductile cast iron, the magnesium treatment process is followed by the graphitising inoculation (secondary inoculation), which can be carried out by introducing the PE core wire after the completion of the magnesium treatment process by the flexible wire method or performed directly on the metal stream when pouring the moulds. Below are presented the cooling curves for ductile cast iron after the inoculation by the introduction method on the metal stream when pouring large moulds using the Itaca XLStream system (Fig. 13). FeSiBa, FeSiCe, FeSiZr and FeSiBi inoculants were used for the tests. The effect of the inoculant used on the change in Temin temperature was also analysed (Fig. 14). The carbon equivalent ranged between 4.26 and 4.32 for the melts carried out. The tests were carried out during the melting of EN-GJS 500-7 and EN-GJS 600-3 ductile cast irons.

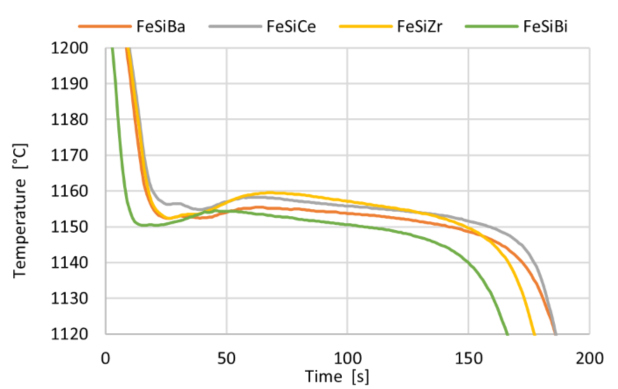


Fig. 13. Cooling curves for ductile cast iron after inoculation with various inoculants

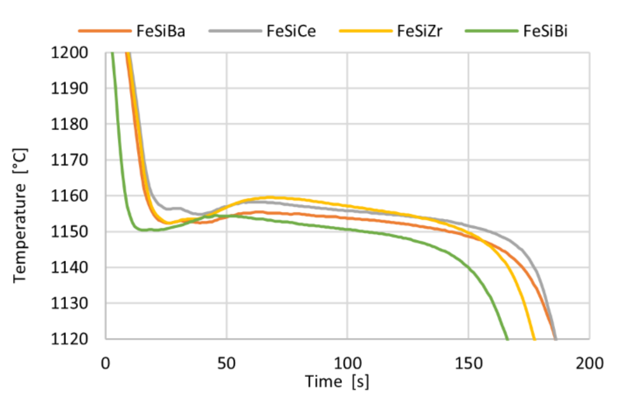


Fig. 14. Minimum eutectic temperature ($Te_{min}$) after the introduction of different inoculants in the secondary inoculation process

In all cases, the number of nuclei reached 100. During these experiments, the Y samples used for testing of mechanical properties and metallographic examinations were poured. Fig. 15 shows the number of graphite precipitates obtained after inoculation by various inoculants. The average number of precipitates in ductile cast iron was 223 for the inoculant containing Zr, 215 for the inoculant containing Ce, 186 for the inoculant containing Bi and 179 for the inoculant containing Ba. Based on the research carried out and taking into account the conditions prevailing in the Iron Foundry in Śrem, the use of inoculants containing FeSiZr and FeSiCe is suggested for secondary inoculation. In non-modified cast iron, the number of precipitates ranged between 42 and 46.

When analysing the strength properties, it should be noted that strengths exceeding those set out in the standards for these two grades of cast iron (500 and 600 MPa, respectively) were obtained in all cases. High elongation values were also obtained (ranging from 4 to 11% for GJS 600-3 and from 7 to 17% for GJS 500-7).

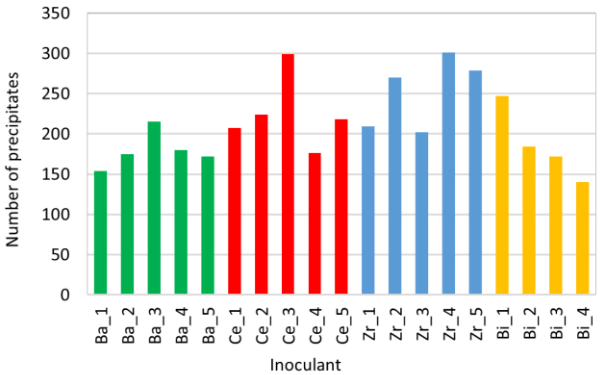


Fig. 15. Effect of secondary inoculation and inoculant used on the number of graphite precipitates in ductile cast iron

# 4. Summary and conclusions

The experiments conducted revealed a very high usefulness of the derivative and thermal analysis (DTA) in the melting of both grey and ductile cast iron. The number of parameters acquired when recording the cooling curve, enriched with its derivative and the results of chemical analysis, and during the examinations of mechanical and metallographic properties make it an irreplaceable tool for assessing metallurgical quality.

The research carried out confirmed the very high relationship between Temin and the nucleation index (number of nuclei) obtained from DTA curves and the actual number of precipitates obtained from the metallographic tests.

The research showed that in order to achieve a high metallurgical quality of cast iron being melted, the appropriate quality of charge materials, as well as the accuracy and repeatability of their dosing, should be ensured. Then, a short time of melting and the holding of the metal in the furnace and in the ladle should be provided. This will result in less consumption of inoculants and consequently lower production costs.

Based on the research carried out, it was found that all the inoculants tested provide the assumed process parameters, which means that their use would depend on their current market availability and price.

By analysing the inoculation methods, it can said that the best results in the primary inoculation process can be achieved by using the automated Itaca OptiDose system, which provides both accurate weighing and precise dosing into the metal stream. For secondary inoculation, a very good process flow as well as indicators are provided by the Itaca XL Stream system.

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# References

[1] Stefanescu, D.M., Suarez, R. & Kim S.B, (2020). 90 years of thermal analysis as a control tool in the melting of cast iron. *China Foundry*. 17(2), 69-84. https://doi.org/10.1007/s41230-020-0039-x.

[2] Jura, S., Sakwa, J. & Borek, K. (1980). Application of thermal and differential analysis for determination of chemical composition parameters. *Krzepnięcie Metali i Stopów*. 3, 16-24. (in Polish).

[3] Jura, S., Sakwa, J. & Borek, K. (1980). Differential analysis of solidification and crystallization processes of gray cast iron. *Krzepnięcie Metali i Stopów*. 3, 25-35. (in Polish).

[4] Jura, Z. & Jura, S. (1990). Calorimetric curve and heat source in thermal and derivational analysis of cast iron solidification process. *Krzepnięcie Metali i Stopów*. 16, 126-139. (in Polish).

[5] Jura, Z. & Jura, S. (1996). The theory of the TDA method in the study of Al alloys. *Krzepnięcie Metali i Stopów*. 28, 57-88. (in Polish).

[6] Jura, S., Studnicki, A., Przybył, M. & Jura, Z. (2001). Application of the ATD method to assess the quality of ductile cast iron. *Archiwum Odlewnictwa*. 1(1), 93-102. (in Polish).

[7] Gawroński, J., Szajnar, J., Jura, Z. & Studnicki, A. (2004). Professor Stanisław Jura, creator of the theory and industrial applications of diagnostics and wear of metals and alloys. *Archiwum Odlewnictwa*. 4(SI 16), 1-74. (in Polish).

[8] Pietrowski, S. & Władysiak, R. (1996). TDA Inspection of piston silumins. *Krzepnięcie Metali i Stopów*. 28, 160-173. (in Polish).

[9] Pietrowski, S. & Gumienny, G. (2002). Methodology for preparing the quality assessment of ductile cast iron using the TDA method. *Archiwum Odlewnictwa*. 2(6). (in Polish).

[10] Pietrowski, S. & Gumienny, G. (2002). Evaluation of the quality of ductile cast iron EN-GJS-400-15 by the TDA method. *Archiwum Odlewnictwa*. 2(6), 257-268. (in Polish).

[11] Chisamera, M., Riposan, I., Stan, S., Stefan, E. & Costache, G. (2009). Thermal analysis control of in-mould and ladle inoculated grey cast irons. *China Foundry*. 6(2), 145-151.

[12] Erturka, S.O., Kumruoglub, L.C., Ozel, A. (2017). Determination of feederless casting limits by thermal analysis in cast iron. *Acta Physica Polonica A*. 131(3), 370-373. DOI: 10.12693/APhysPolA.131.370.

[13] Seidu, S.O. (2013). Thermal analysis of preconditioned ductile cast iron. *International Journal of Current*

*Engineering and Technology*. 3(3), 813-818. ISSN 2277-4106.
[14] Cojocaru, A.M., Riposan, I. & Stan, S. (2019). Solidification influence in the control of inoculation effects in ductile cast irons by thermal analysis. *Journal of Thermal Analysis and Calorimetry*.138, 2131-2143. https://doi.org/10.1007/s10973-019-08808-2.
[15] Petrus, Ł., Bulanowski, A., Kołakowski, J., Brzeżański, M., Urbanowicz, M., Sobieraj, J., Matuszkiewicz, G., Szwalbe, L., Janerka, K. (2020). The influence of selected melting parameters on the physical and chemical properties of cast iron. *Archives of Foundry Engineering*. 20(1), 105-110. DOI: 10.24425/afe.2020.131290.
[16] Petrus, Ł., Bulanowski, A., Kołakowski, J., Sobieraj, J., Paruch, T., Urbanowicz, M., Brzeżański, M., Burdzy, D. & Janerka. K. (2021). Importance of TDA thermal analysis in an automated metallurgical process. *Journal of Casting & Materials Engineering*. 5(4), 89-93. https://doi.org/10.7494/jcme.2021.5.4.89.
[17] ProserviceTech. Retrieved June, 30, 2022 from http://www.proservicetech.it/itacax-thermal-analysis-final-iron-quality-control/.
[18] Novacast. Retrieved June, 30, 2022 from https://www.novacast.se/product/atas/.
[19] Heraeus. Retrieved June, 30, 2022 from https://www.heraeus.com/en/hen/products_and_solutions_hen/foundry/thermal_analysis/thermal_analysis.html.
[20] Vesuvius. Retrieved June, 30, 2022 from https://www.vesuvius.com/content/dam/vesuvius/corporate/Our-solutions/our-solutions-master-english/foundry/Newsletter/Issue2/FP-new-issues/FERROLAB%20V.pdf.