

E. GUZIK<sup>1</sup>, D. KOPYCIŃSKI<sup>1</sup>, A. ZIÓŁKO<sup>2</sup>, A. SZCZĘSNY<sup>1\*</sup>

## THE METHOD OF INOCULATION OF HIGH-QUALITY GREY CAST IRON INTENDED FOR MASSIVE CASTINGS FOR BOTTOM AND DISTANCE PLATES AS WELL COUNTERWEIGHTS MANUFACTURED AS VERTICAL CASTINGS

The technology of producing castings of high-quality inoculated cast iron with flake graphite particles in the structure is a combination of the melting and inoculation process. Maintaining the stability of the strength and microstructure parameters of this cast iron is the goal of a series of studies on the control of graphitization and austenitic inoculation (increasing the number of primary austenite dendrites), and which affects the type of metal matrix in the structure. The ability to graphitize the molten alloy decreases with its holding in the melting furnace more than an hour. The tendency to crystallize large dendritic austenite grains and segregation of elements such as Si, Ni and Cu reduce the ductility properties of this cast iron. The austenite inoculation process may introduce a larger number of primary austenite grains into the structure, affecting the even distribution of graphite and metal matrix precipitation in the structure. Known inoculation effects the interaction (in low mass) of additives: Sr, Ca, Ba, Ce, La, produces  $MC_2$  carbide). Addition of Fe in the inoculant influences the number and shape of austenite dendrites. Hybrid modification combines the effects of these two factors. The introduction of nucleation sites for the graphite eutectics and primary austenite grains result in the stabilization of the cast iron microstructure and an increase in mechanical properties. The obtained test results set the direction for further research in this area in relation to the production of heavy plate castings in vertical and horizontal pouring.

*Keywords:* High-quality cast iron; heavy castings; massive casting; graphitizing inoculation; hybrid inoculation

### 1. Introduction

The article is the result of a research project in Foundry Krakodlew S.A. in Krakow (Poland). The project concerned the development of a technology for casting massive plate castings in a vertical arrangement. These castings weigh up to 80 tons. In ingot moulds, during operation, changes in the structure of the cast iron may occur, which may lead to cracking of these castings. That problems are not encountered when using another type of heavy (or massive) castings, such as counterweights, the weight of which reaches 30 tons. The casting crystallization should be carried out in such a way as to increase its resistance to thermal fatigue and cracking under the influence of thermal stresses. The above-mentioned issues are particularly relevant to heavy bottom plates. Due to technical and technological considerations, gray modified cast iron with flake graphite still has the advantages that characterize this cast iron to be used for bottom plates. Examples of that plates produced in Krakodlew S.A.

foundry is shown in Fig. 1. Therefore, high-quality inoculated gray cast iron with flake graphite was selected for the tests. In this case, pay attention to even distribution of structural phases, such as graphite and metal matrix, in the casting volume. The appearance of at least one type of problem, e.g. segregation of elements, graphite flotation, variable proportion of perlite and ferrite and shrinkage porosity always leads to a very large number of defective castings with reduced service life and large financial losses of the foundry.

Currently, every producer of massive castings wants to resign from machining, which in this case constitutes high costs in the technology of obtaining a finished product. Currently, there is no known technology for obtaining this type of castings without mechanical processing. However, it seems that a significant reduction in processing time could be obtained if foundries vertical positioned heavy plates. At the same time, it should be realized that this method of casting may result in difficulties in keeping the mass or the shape of the casting (the phenomenon

<sup>1</sup> AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY, DEPARTMENT OF ENGINEERING OF CAST ALLOYS AND COMPOSITES, FACULTY OF FOUNDRY ENGINEERING, AL. MICKIEWICZA 30, 30-059 KRAKOW, POLAND

<sup>2</sup> KRAKODLEW S.A., 1 UJASTEK ST., 30-969 KRAKOW, POLAND

\* Corresponding Author: [ascn@agh.edu.pl](mailto:ascn@agh.edu.pl)





Fig. 1. Examples of bottom plates – weight range: up to 80 tons (a) and counterweight – weight range: up to 30 tons (b) produced in Foundry Krakodlew S.A.

of deformation of the casting surface). It can be assumed that the above issues apply not only to the material which is cast iron [1-4], but also cast steel [5-11] and other groups of materials, including types of alloy cast iron [4,12-15], to be used for heavy castings. Unfortunately, there is a small number of articles on that castings, which mainly refer to ductile iron [16-19] and the methods of their production in a vertical arrangement in a casting mold [16]. When producing castings, it should be mentioned that porosities can significantly reduce strength and utility properties of castings. Fig. 2 shows photos of the porosity that may appear in cast iron.

## 2. Aspect of inoculation of cast iron intended for heavy castings

In order to obtain a cast without defects, the inoculation procedure must be carried out in a proper and appropriate way. The mechanism of cast iron inoculation is closely related to the process of nucleation and eutectic growth, and there are several hypotheses in this regard. According to one, the introduction of inoculants into the molten alloy causes the formation of heterogeneous-endogenous graphite nuclei in the form of oxides

( $\text{SiO}_2$ ), nitrides, sulfides or carbides. Proponents of this hypothesis believe that the procedure of inoculation cast iron is actually a process of deoxidizing the bath with silicon. This hypothesis is contradicted by the fact that ferrosilicon without Ca and Al admixtures does not give the inoculation effect. The most probable hypothesis is B. Lux's theory, which is presented in Fig.3a. The instability of carbides as graphite nucleation sites under the influence of oxygen and sulfur contained in molten cast iron is shown in Fig. 3b. The scheme of gray cast iron inoculation is shown in Fig. 4.

## 3. Phase composition of industrial inoculants

Fig. 5 shows the results of tests of industrial inoculants of their phase composition and distribution. It can be seen that iron exists only in the form of  $\text{FeSi}_2$  phase and is a separate phase in the inoculant. It cannot be ruled out that the  $\text{FeSi}_2$  phase may be a suitable substrate for the nucleation of primary austenite dendrites. Moreover, elements such as Sr, Al, Ba can also form substrates for heterogeneous nucleation of austenitic grains in the molten alloy.

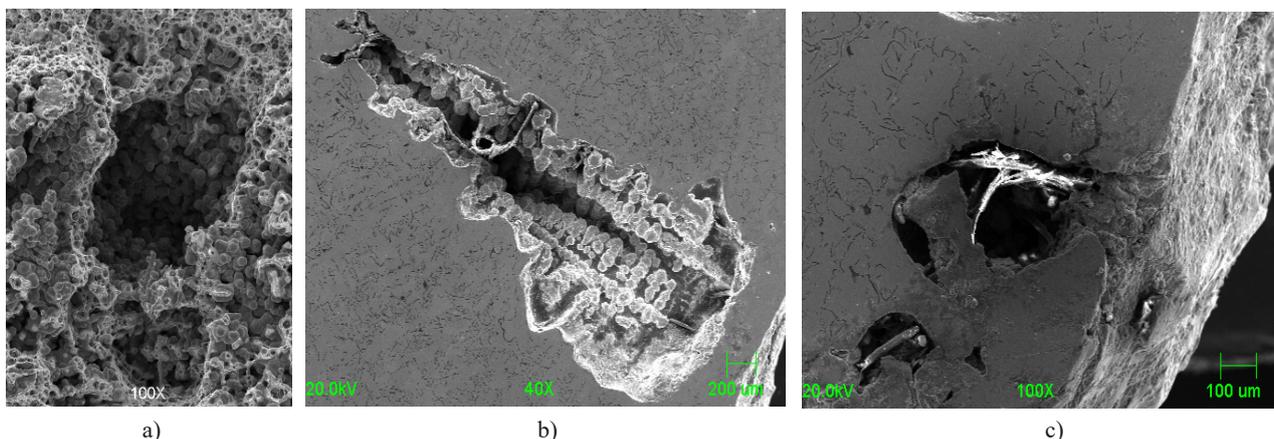


Fig. 2. Types of porosity defects in the microstructure of a gray cast iron casting: interdendritic porosity – a), shrinkage porosity – b), gas porosity – c)

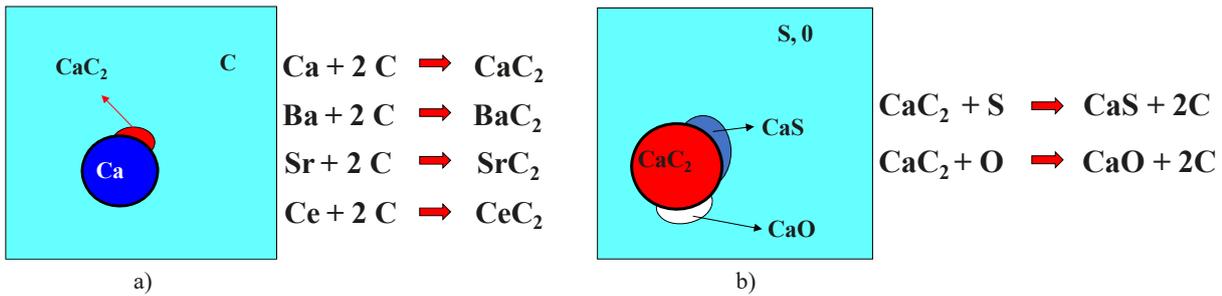


Fig. 3. Diagram of the formation of carbides as graphite nucleation sites (a) and the mechanism of their instability during the reaction with sulfur and oxygen (b)

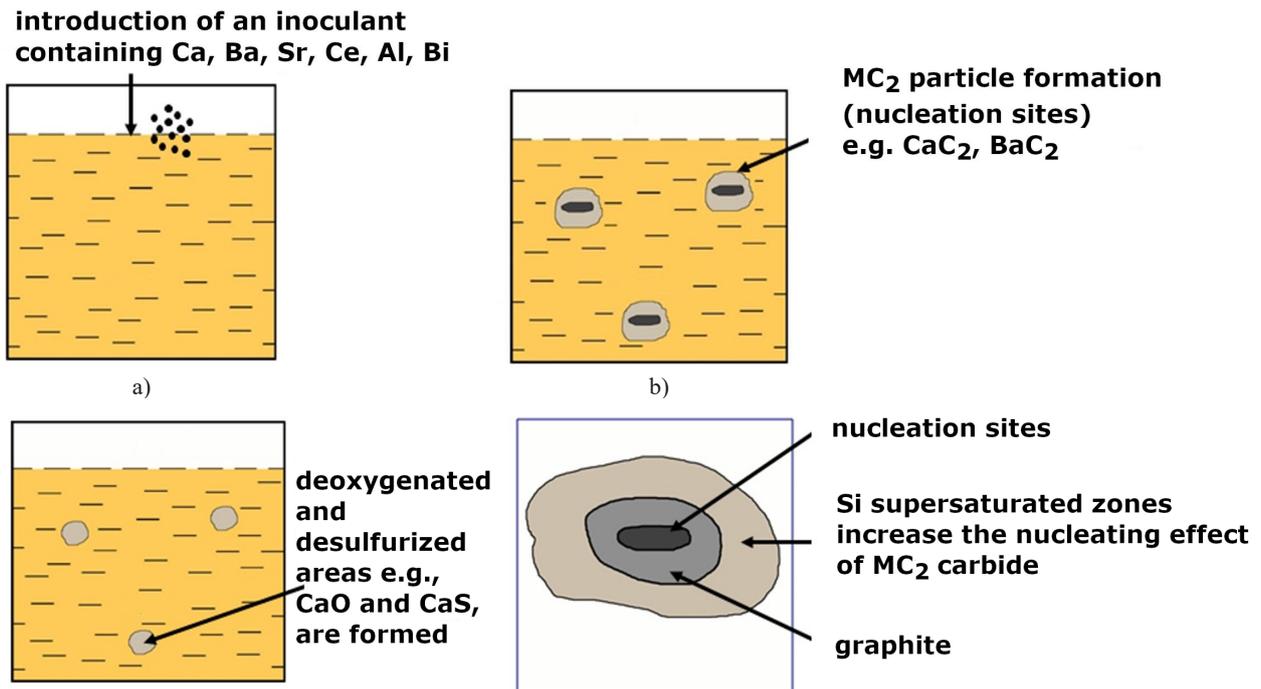


Fig. 4. Scheme of the inoculation procedure: introduction of an inoculant containing Ca, Ba, Sr, Ce, Al, Bi (a); formation of deoxygenated and desulfurized areas, e.g. CaO and CaS (b); formation of MC<sub>2</sub> particles (nucleation site) (CaC<sub>2</sub>, BaC<sub>2</sub>)

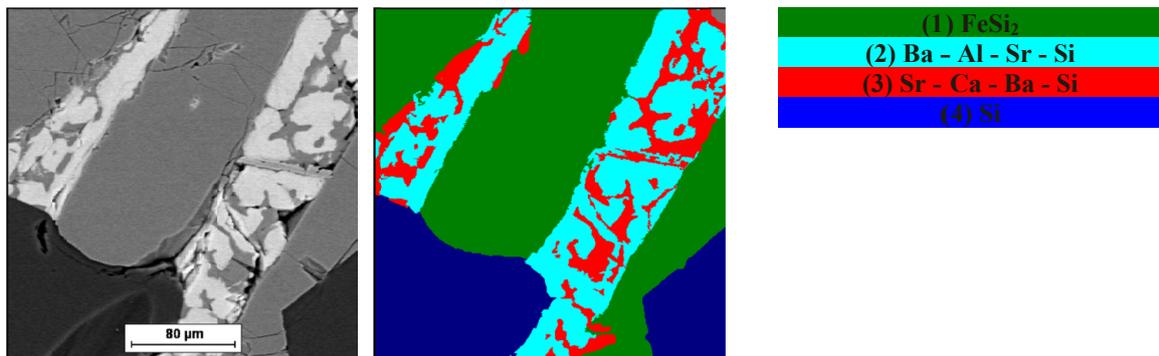


Fig. 5. Assessment of the phase composition of a typical industrial inoculant with additives: Sr, Ba, Al. using a scanning microscope with EDS. Chemical composition of phases (wt%): (1) 52.39% Si, residual% Fe; (2) 9.04% Al, 1.27% Ca, 40.38% Ba, 3.83% Sr, residual Si; (3) 34.13% Ca, 2.10% Ba, 20.59% Sr, residual Si; (4) 100% Si

In the industrial practice of iron foundry, the inoculation effect is considered mainly as the influence on the graphite eutectic grains, while its influence on the crystallization of austenite dendrites (primary austenite grains) is largely neglected. It was

proved in [3,4] that the inoculation procedure affects not only the grains of graphite eutectics, but also the grains of primary austenite. Fig. 6 shows the correlation between the number of primary austenite grains and the number of graphite eutectic

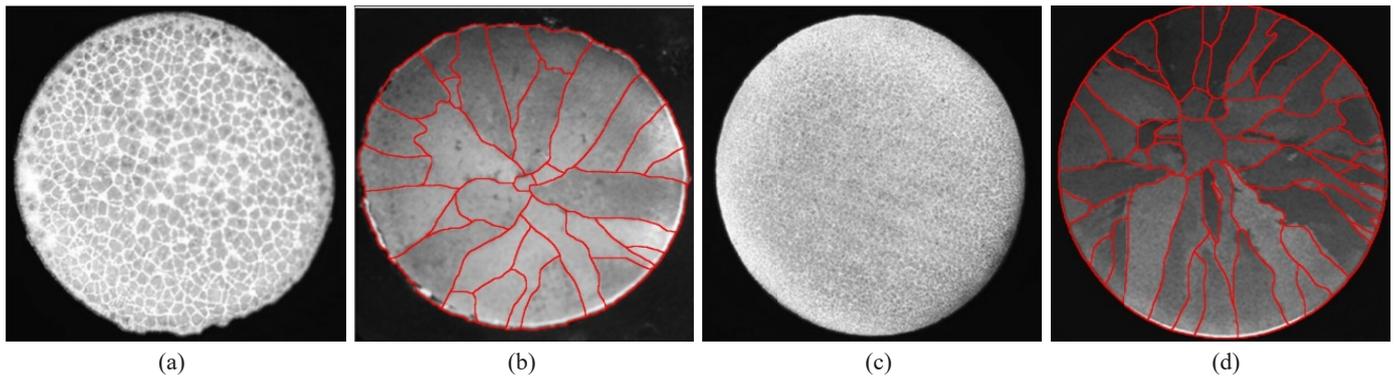


Fig. 6. Macrostructure [3,4] of rolls (diameter 30 mm) of gray cast iron with flake graphite after heat treatment using the DAAS method: before inoculation (a, b); after inoculation (c, d); eutectic grains (a, c); primary austenite grains (b, d)

grains in gray cast iron before and after inoculation. Therefore, in the further part of the work, we will treat the inoculation as a procedure influencing the number of eutectic grains of graphite and primary austenite grains.

The most common method is to introduce the inoculant into the ladle and apply a secondary inoculation when pouring the mold. Mostly, foundries use high-melting inoculants containing 75%Si in ferrosilicon. They crystallize in the temperature range from 1221°C to 1315°C and can be introduced at the temperature of the molten alloy of about 1350°C. Less frequently used are low-melting inoculants which composition is based on 50%Si. These crystallize in the temperature range from 1210°C to 1221°C. Due to the low pouring temperature in the case of the production of massive castings, the potentially better results should be obtained by using low-melting inoculants in the case of introducing its to the metal stream when pouring the mold. The amount of the inoculants used in the metallurgical process ranges from 0.2 to 0.7% of the weight of melted cast iron. The equilibrium phase diagram [20] of Fe-Si alloys (Fig. 7) shows that FeSi alloys with a composition of 70% and 50%Si have a melting point of 1220°C and 1350°C, respectively. The presented research on the optimization of the physicochemical state of molten alloy was aimed at increasing the number of graphite

eutectic grains per unit area and thus improving the mechanical and functional properties of castings.

Properly carried out inoculation can contribute to the elimination of defects in the production of heavy castings. The studies presented below were aimed at developing a 3-stage inoculation technology, the scheme of which is presented in TABLE 1 and Fig. 8.

The research presented in the article is part of the stage 2 research. The research was carried out in Foundry Krakodlew S.A. to develop a special inoculation method dedicated to massive castings, including large-size plates produced in a vertical position. This method using the cored wire method consisted in introducing a steel tube with a graphitizing inoculant and the steel tube itself. In order to simplify the tests in laboratory conditions, the inoculation procedure was carried out by introducing a graphitizing inoculant together with a steel bell. The conducted research has shown that this method of treatment – hybrid inoculation, is more effective in reducing the undesirable D-type interdendritic graphite (according to the PN-EN ISO 945 standard) than in the standard inoculation. The research and analysis of the results presented in the article allowed for the development of an innovative method of hybrid inoculation of high-quality cast iron with flake graphite dedicated to the technology of production massive (heavy) castings.

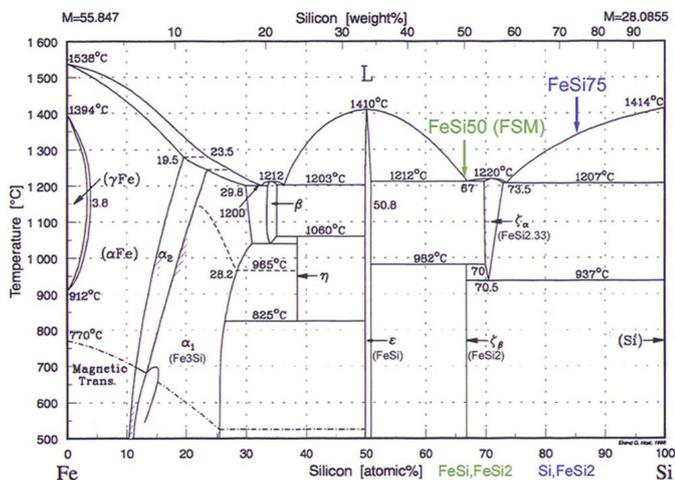


Fig. 7. Equilibrium phase diagram of Fe-Si alloys with the designation of the high-melting FeSi75 and low-melting FeSi50 inoculant

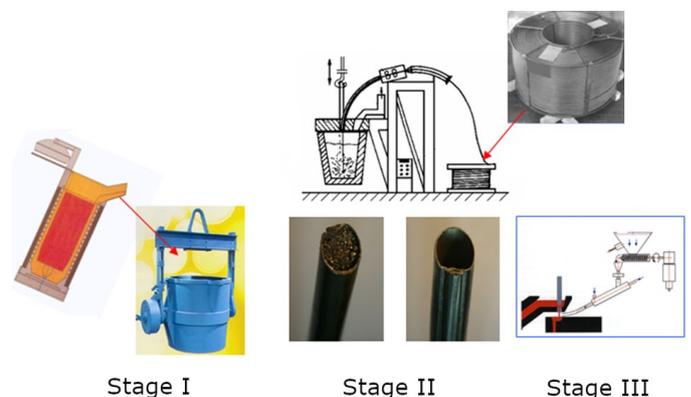


Fig. 8. Scheme of inoculation applied in Foundry Krakodlew S.A.: I – stage (when pouring from the furnace into the ladle), II – stage (introducing wire steel with and without a graphitizing inoculant using cored wire method), III – stage (on a stream of metal when pouring molds)

TABLE 1

Scheme of the 3-stage inoculation procedure

Inoculation		
Stage I	Stage II	Stage III
in the ladle	with the cored wire method	when pouring the mold

4. Experimental

At the beginning, it was checked whether the change of the forming method (from horizontal to vertical) could affect the casting defects. For this purpose, an earlier study carried out in the Krakodlew S.A. foundry, in which slabs of ductile iron was produced, was used. Information on these studies was published

in [21]. The research published there showed a large difference between the crystallization temperatures of the molten alloy depending on the location of the thermocouples in the casting and defects such as porosity were observed.

To control the technological process of casting the castings, computer simulation was used with the use of ProCAST software for the chemical compositions of cast iron presented in TABLE 2. Fig. 9 show the results of a computer simulation with the ProCAST software for a board with dimensions; 100×500×500 mm.

The next step was to make a laboratory melt using the molten alloy inoculation method with a steel bell. In the crucible of a 15 kg medium frequency induction furnace, the cast iron charge was melted to a liquid melt state. After superheating the melt bath to 1500°C (T1), the molten alloy was held for 3 minutes and then the temperature was lowered to 1400°C (T2). At this



Fig. 9. The simulation results showing the areas of shrinkage porosity in the reference cast iron (a, b, c) and the inoculated cast iron (d, e, f). Chemical compositions assigned in Table 2; No. 1 (a, d), No. 2 (b, e), No. 3 (c, f)

TABLE 2

Chemical composition of inoculated cast iron with flake graphite

Chemical composition	No 1	No 2	No 3
C	3.70	3.70	3.40
Si	2.00	1.70	1.66
Mn	0.6-1.0	0.6-1.0	0.6-1.0
Cr	0.2	0.2	0.2
Cu	to 0.6	to 0.6	to 0.6
Sn	0.1	0.1	0.1
Ni	0.2	0.2	0.2
S	0.04-0.07	0.04-0.07	0.04-0,07
P	0.2	0.2	0.2
Eutectic saturation	$S_c = 1.02$	$S_c = 0.99$	$S_c = 0.91$
Carbon Equivalent	$CE = 4,33$	$CE = 4,25$	$CE = 3.93$

temperature, a inoculation procedure was carried out using an industrially selected inoculants and to modification of the primary structure (grains of primary austenite dendrites) using a 100 g steel bell. These processes took place in a crucible located in a switched-on induction furnace. The casting moulds were standard rollers with a diameter of  $\text{Ø}30 \times 270$  mm and a “Y” type ingot. A summary of data for individual melts is presented in TABLE 3.

The course of curves of cooling and crystallisation of inoculated cast iron is shown in Fig. 10. That curves are characteristic for eutectic cast iron chemical composition. The degree of subcooling  $T$  (defined as the difference between the equilibrium crystallization temperature of graphite eutectic and the minimum temperature at the beginning of cast iron crystallization) is for, respectively: B1 = 18.63°C, B2 = 18.27°C, B3 = 10.25°C,

TABLE 3

Technological parameters of the performed laboratory melts

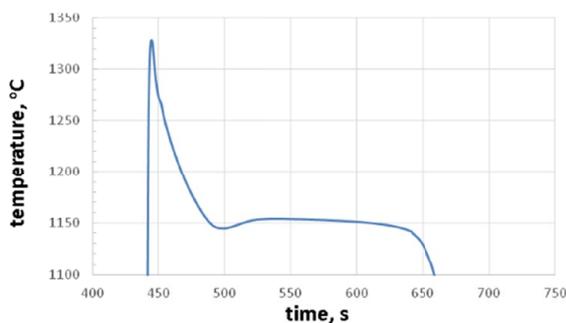
Melt No	Inoculant	Chemical composition of cast iron, % mas.						
B1	Inolate	C	P	S	Si	Mn	Cr	Cu
	mass %	3.72	0.02	0.06	1.98	0.68	0.17	0.19
B2	Superseed	C	P	S	Si	Mn	Cr	Cu
	mass %	3.73	0.02	0.06	2.05	0.70	0.17	0.19
B3	Zircinoc	C	P	S	Si	Mn	Cr	Cu
	mass %	3.69	0.03	0.05	2.01	0.67	0.18	0.20
B4	Zircinoc	C	P	S	Si	Mn	Cr	Cu
	mass %	3.64	0.04	0.06	2.09	0.60	0.23	0.27

Steel bell mass – 100g  
Overheating temperature (T1) 1500°C  
Inoculation temperature (T2) 1400°C

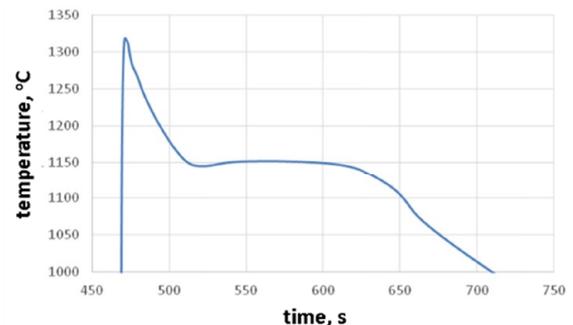
B4 = 15.83°C. It is worth noting that the decrease in the degree of subcooling  $T$  for the inoculated cast iron in relation to the initial state is insignificant.

## 5. Results and discussion

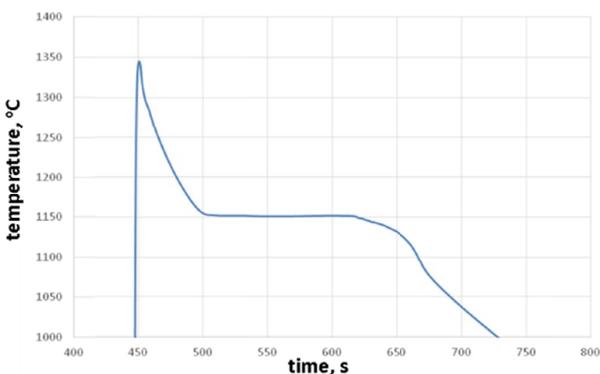
Figures 11-14 show the microstructures in the investigated castings made during experimental melting carried out under semi-industrial conditions at the Experimental Foundry in the Foundry Department of the AGH University of Science and Tech-



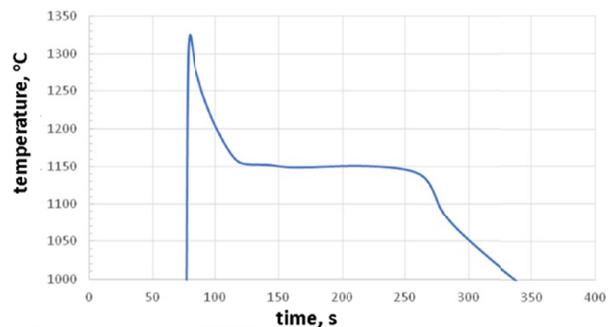
a)



b)



c)



d)

Fig. 10. Cooling and crystallization of inoculated grey cast iron for the melts: a) B1, b) B2, c) B3, d) B4

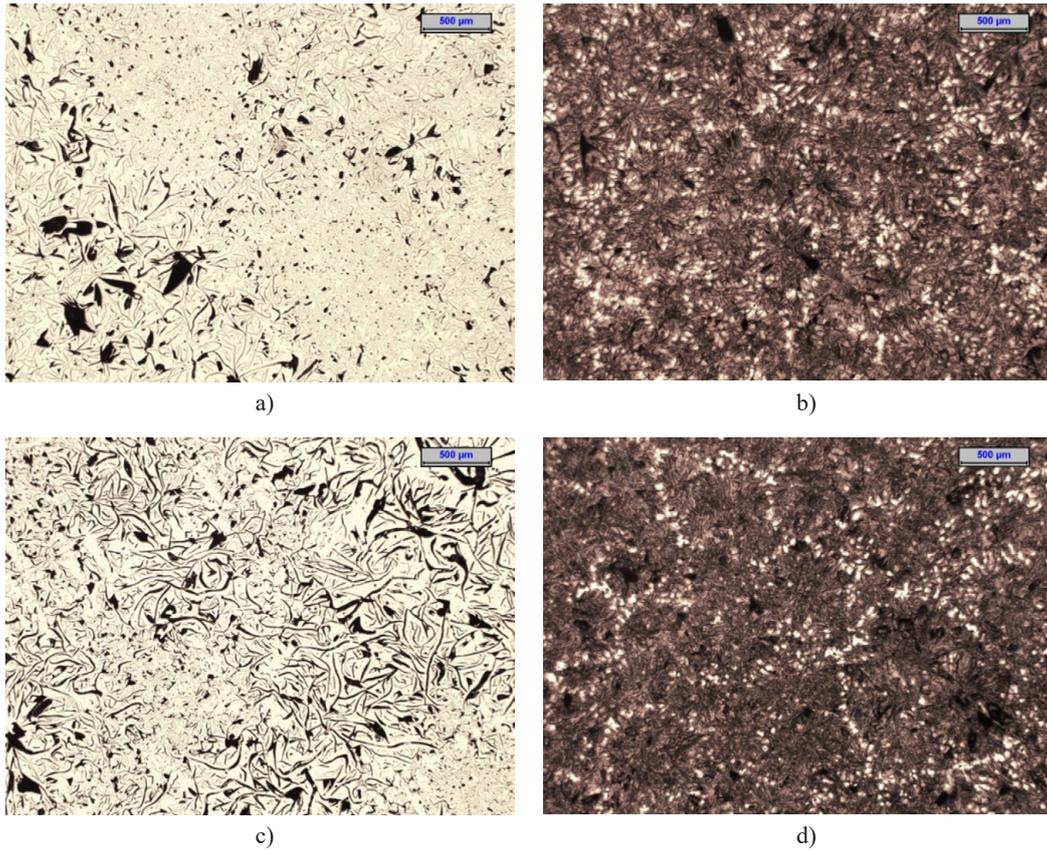


Fig. 11. Microstructure of cast iron (melt No 1B) after inoculation: „Y“-type ingot – a,b), roll casting with a diameter of Ø30 mm – c,d); nital etched – b,d) not etched – a,c)

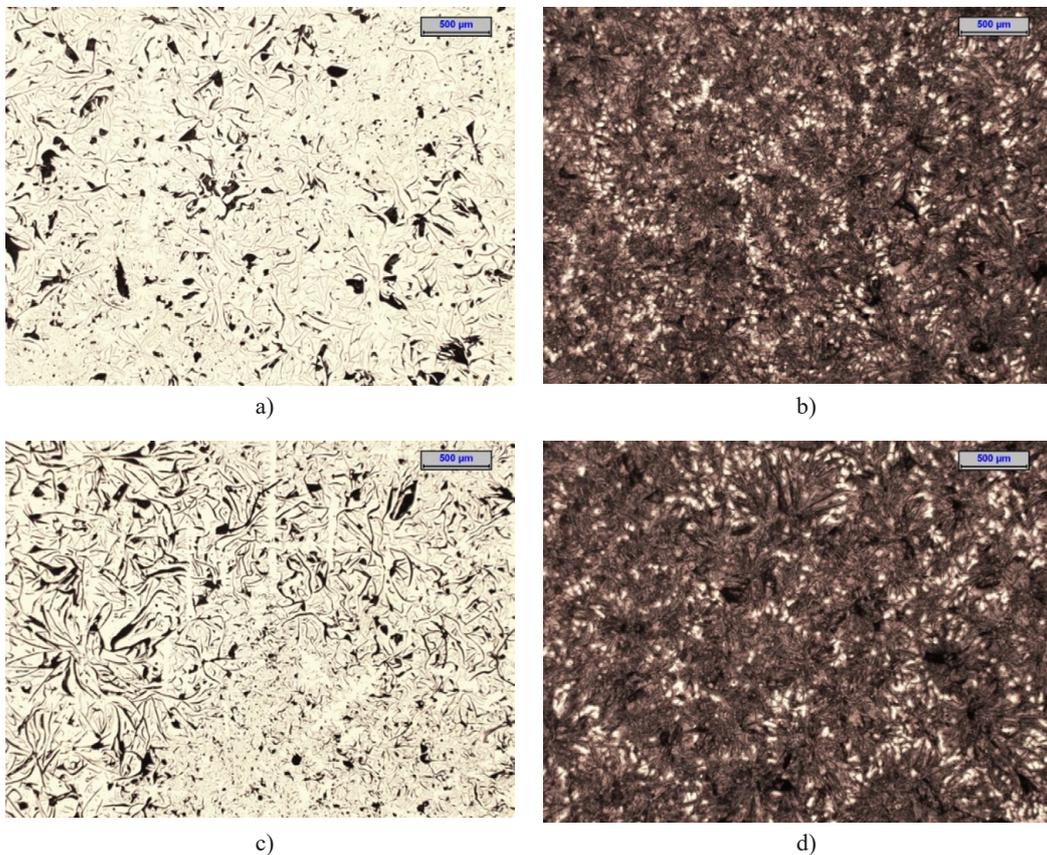


Fig. 12. Microstructure of cast iron (melt No 2B) after inoculation: „Y“-type ingot – a,b), roll casting with a diameter of Ø30 mm – c,d); nital etched – b,d) not etched – a,c)

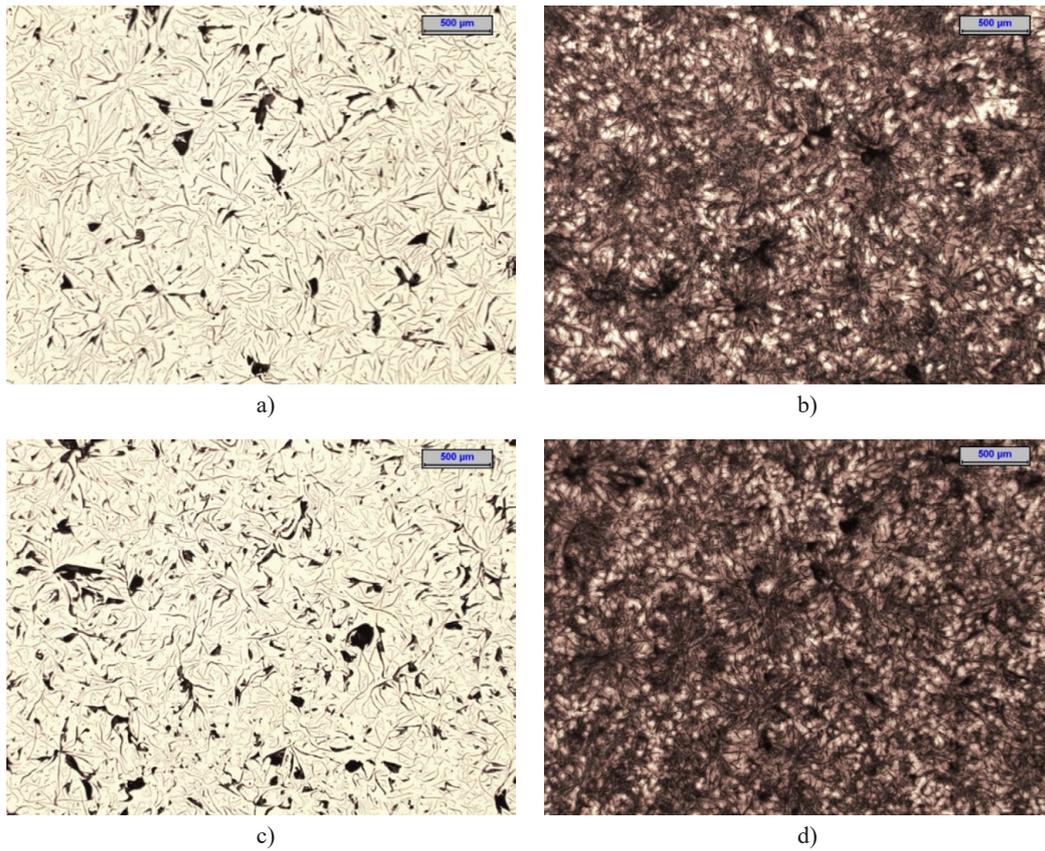


Fig. 13. Microstructure of cast iron (melt No 3B) after inoculation: „Y“-type ingot – a,b), roll casting with a diameter of Ø30 mm – c,d); nitral etched – b,d) not etched – a,c)

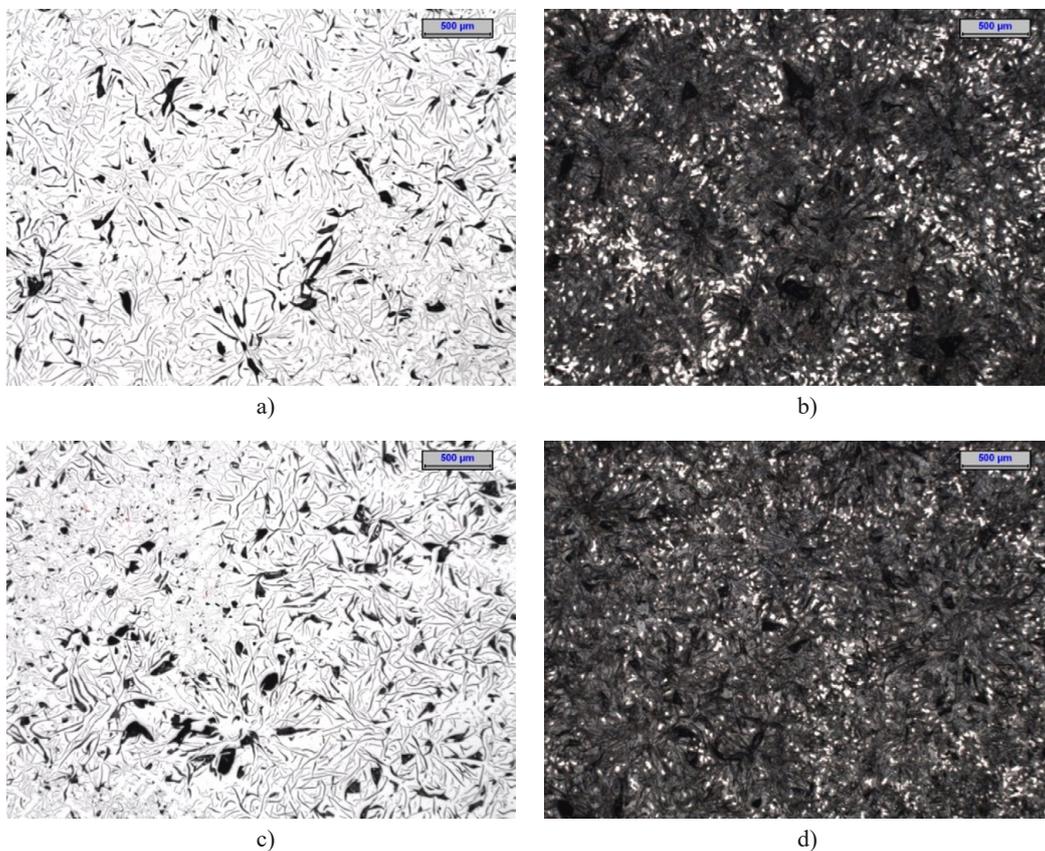


Fig. 14. Microstructure of cast iron (melt No 1B) after inoculation: „Y“-type ingot – a,b), roll casting with a diameter of Ø30 mm – c,d); nitral etched – b,d) not etched – a,c)

Results of the metallographic analysis

No melt	Cast	N <sub>A</sub> , cm <sup>-2</sup>	%A	%B	%D	A-L <sub>max</sub>	Class	B-L <sub>max</sub>	Class	D-L <sub>max</sub>	Class
B1	Y	450	50	20	30	319	3	461	3	53	6
	Ø30	357	55	15	30	367	3	724	2	67	5/6
B2	Y	440	50	20	30	453	3	643	2	135	4/5
	Ø30	324	40	30	30	376	3	672	2	98	5
B3	Y	324	80	20	0	288	3	418	3	-	-
	Ø30	232	87	10	3	319	3	448	3	75	5
B4	Y	73	386	3	20	569	2	90	7	283	5
	Ø30	87	344	3	10	636	2	102	3	253	5

N<sub>A</sub> – number of eutectic grains;  
 % A, B, D – percentage share of particular types of graphite distribution,  
 class – graphite size class according to PN-EN-ISO 945-1,

L<sub>max</sub> – maximum graphite dimensions,  
 Y – results obtained in “Y”-type ingot casting,  
 Ø30 – results obtained in roller casting of Ø30 mm diameter

nology. During the melting of cast iron, various inoculants were checked in terms of their influence on the uniform distribution of flake graphite obtained during research melts 1B-4B. The microstructure parameters are summarised in TABLE 4. The study shows that superheating the metal at 1500°C for three minutes gives much better results in terms of graphite homogeneity compared to the superheating temperature of 1450°C. Superheating of the metal contributes to obtaining a physico-chemical state of the molten alloy, providing a more favorable microstructure from the point of view of the separation of graphite flakes, appropriate distribution and size.

The study shows that the inoculation of the primary structure (austenite dendrite grains) and the inoculation of the graphite eutectic, contributed to an increase in the homogeneity of the microstructure of the cast iron, as shown by the results in terms of the shape and size of the flake graphite particles. Analysis of the curves of crystallization and cooling of cast iron and their first derivatives indicates that the procedure of eutectic and double inoculation (concerning austenite dendrite grains and graphite eutectic) increases the temperature of the end of cast iron crystallization. This effect was observed in all cases and is accompanied by a significant reduction in the presence of the so-called “overcooled” areas, where graphite nucleates to form significantly fragmented and undesirable in the microstructure of cast iron interdendritic graphite type D according to ISO 945-2010. Comparison of the modifier effects, gave the most favorable results when the Zircinoc type inoculant was used. In combination with a double inoculation (i.e. a combination of Zircinoc modifier + steel “bell”), it resulted in a significant decrease in the volume proportion of D-type graphite, down to a level of 5% in standard casting shafts. At the same time, the degree of undercooling has the lowest value and the highest solidus temperature. Analysis of the effect of different doses of Zircinoc inoculant (together with the addition of “steel bell”) indicates that the higher the inoculant dose, the proportion of undesirable D-type graphite separation in the microstructure decreases. The lowest value was obtained for cast iron with 0.5% Zircinoc. However, the use of 0.35% inoculant resulted in only a slight increase in the proportion of D-type graphite release, to 5%.

## 6. Conclusions

The use of a inoculant with a high capacity to produce large amounts of graphite eutectic grains in combination with steel introduced by the inoculation process results in a significant reduction of shrinkage type defects when pouring vertically oriented plate moulds.

From high melting graphitising inoculants based on FeSi75 (their melting temperature in the bath is 1350°C) such as; Inolate, Superseed or Zircinoc, as, the best effect of the so called primary modification was obtained when Zircinoc and then Superseed were introduced into the bath at their consumption of 0.35% to the mass of molten alloy.

A significant decrease in the volume proportion of D-type graphite, up to a level of 5% in the microstructure, was observed in favour of uniformly distributed A-type graphite in the metal matrix. From this case, it is concluded that during the dissolution of the steel bell in the liquid melt, steel (iron) crystallization sites are obtained for the nucleation of the hypoeutectic austenite dendrite grains, which in both cases have the same A1-type crystal lattice.

## Acknowledgements

The publication was created during the implementation of the project RPMP.01.02.01-12-0055 / 18 in Foundry Krakodlew S.A. in Cracow financed by the Regional Operational Program of the Małopolskie Voivodeship, Poland.

## REFERENCES

- [1] E. Guzik, Procesy uszlachetniania żeliwa – wybrane zagadnienia. Archives of Foundry, PAN Katowice (2001).
- [2] M. Benedetti M.E. Torresani, V. Fontanari, D. Lusuardi, Metals 7, 88 (2017).
- [3] J. Dorula, D. Kopyciński, E. Guzik, A. Szczęsny, D. Gurgul, Materials 14, 21, 6682, 1-17 (2021).

- [4] D. Kopyciński, Shaping of structure and mechanical properties in cast iron for operation under harsh conditions: (selected issues). Archives of Foundry Engineering. Wydawnictwo Komisji Odlewnictwa PAN Gliwice (2015).
- [5] Q. Wang, G. Cheng, Y. Hou, Metals **10**, 4-15 (2020).
- [6] Q. Wang, S. Chen, L. Rong, Metall. Mater. Trans. **51**, 2998-3008 (2020).
- [7] B. Kalandyk, R. Zapała, S. Sobula, M. Górny, Ł. Boroń, Metallurgija-Metallurgy **53**, 4, 613-616 (2014).
- [8] B. Kalandyk, R. Zapała, Archives of Foundry Engineering **13**, 4, 63-66 (2013).
- [9] D. Tęcza, R. Zapała, Archives of Foundry Engineering **18**, 1, 119-122 (2018).
- [10] G. Tęcza, Materials **15**, 3, 1-11 (2022).
- [11] S. Sobula, S. Krański, Archives of Foundry Engineering **21**, 4, 82-86 (2021).
- [12] M. Celis, B. Domengès, E. Hug, J. Lacaze, Materials Science Forum **925**, 173-180 (2018).
- [13] R. Gilewski, D. Kopyciński, E. Guzik, A. Szczęsny, Materials **14**, 20, 5993, 1-15 (2021).
- [14] R. Gilewski, D. Kopyciński, E. Guzik, A. Szczęsny, Appl. Sci.-Basel. **11**, 20, 9527, 1-15 (2021).
- [15] D. Kopyciński, D. Siekaniec, A. Szczęsny, M. Sokolnicki, A. Nowak, Archives of Foundry Engineering **26**, 4, 74-77 (2016).
- [16] A. Szczęsny, D. Kopyciński, E. Guzik, G. Soból, K. Piotrowski, P. Bednarczyk, W. Paul, Acta Metallurgica Slovaca **26**, 74-77 (2020).
- [17] M.M. Mourad, S. El-Hadad, Journal of Metallurgy **9**, 1-11 (2015).
- [18] E. Foglio, M. Gelfi, A. Pola, D. Lusuardi, Key. Eng. Mat. **754**, 95-98 (2017).
- [19] F. Kavicka, B. Sekanina, J. Stetina, K. Stransky, V. Gontarev, J. Dobrovska, Materials and Technology **43**, 73-78 (2009).
- [10] O. Kubaschewski, H. Okamoto, Phase diagrams of binary iron alloy, ASM International, Materials Park (1993).
- [21] A. Szczęsny, D. Kopyciński, E. Guzik, G. Soból, K. Piotrowski, P. Bednarczyk, W. Paul, Metallurgica Slovaca **26**, 74-77 (2020).