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PREDICTION OF CHUNKY GRAPHITE IN NODULAR CAST IRON ON THE BASE OF NUMERICAL SIMULATION AND EXPERIMENTAL DATA

Chunky graphite has been recognized for a long time as one of the major problems in production of heavy section nodular cast iron. A great number of studies have been conducted to describe the chunky graphite formation, but a clear understanding of its appearance and a safe mastering of the melt preparation to avoid chunky graphite are not yet available. In the present work the cooling curves were recorded in large cone blocks and standard TA cup. According to measured data from the cone block, melt characteristics and heat transfer coefficient between casting and mould were adjusted in the ProCAST[®] simulation software. For a near-eutectic nodular cast iron test melt with 0.7 wt. % Ni, relationship between the area of the cone block affected by chunky graphite and simulation software results has been observed, i.e., thermal modulus and time to solidus.

Keywords: chunky graphite, nodular cast iron, numerical simulation, cooling curve, microstructure

1. Introduction

Over the last fifteen years, the need for heavy section ductile iron castings have increased in different industry sectors. This refers primarily to the manufacture of castings for wind turbines, manufacture of containers for permanent storage of nuclear waste and industry of heavy-duty vehicles [1-4]. Chunky graphite (CHG) is a graphite degeneration which normally appears in the thermal centre of large castings and decreases the mechanical properties, especially tensile strength, elongation and fracture toughness [3-5]. On macro-scale, it is optically visible on cut or machined surfaces as a black spot. Microscopic observation shows that CHG consists of large cells of highly branched and interconnected graphite strings. The main causes of a CHG occurrence are low cooling rate and chemical composition of the melt [1-3]. With slower cooling rates and increasing section thickness, the size of nodules increases and their count per unit of area decreases. In such zones, with combination of higher concentrations of certain element, chunky graphite will almost always develop as a result of degenerated graphite form [5]. According to [1,3,6] CHG formation is favoured by the presence of Ce, Ca, Si, Ni, Al, Mg, Cu and P, especially in the absence of the elements Bi, Sb, As, Sn, Pb, B, Cu and O. A great number of studies have been conducted to describe the

CHG formation, but a clear understanding of its appearance and a safe mastering of the melt preparation to avoid CHG are not yet available.

Thermal analysis has been used for a long time as important tool to control melt preparation and to evaluate the solidification behavior of cast iron. Later, thermal analysis become imperative as it gives the possibility of correlating cooling curve characteristics with microstructure features and prediction of mechanical properties. In nodular cast iron production, thermal analysis is the key tool for obtaining information regarding graphite morphology in the structure and its relation with the potential to avoid or minimize the occurrence of defects. In last few years researchers are working on the possibility of correlating cooling curve characteristics recorded on TA cups with predicting CHG formation in heavy-section nodular cast iron parts [4,7,8]. Another possibility is the use of simulation to predict the occurrence of CHG. This correlations with CHG formation still need to be investigated.

In the present work the cooling curves were recorded in the cone blocks and standard TA cup. According to measured data from the cone block, melt characteristics and heat transfer coefficient between casting and mould were adjusted in the ProCAST[®] simulation software. Correlation of the simulation results with the experimentally obtained CHG area was discussed.

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2. Experimental

The test castings used in this study were large cone blocks with diameter of 300 mm and a height of 350 mm, to enable investigation of the influence of different cooling rates on CHG formation, Fig. 1.



Fig. 1. Cone pattern with feeder

Two moulds, each of them containing one cone, were produced from sodium silicate bonded sand. For both moulds, KALPUR direct pouring process was used. In this study near-eutectic nodular cast iron test melt with 0.7 wt. % Ni was used. Nickel is known as chunky graphite promoter. Preliminary results have proven that Ni promotes the chunky graphite formation in nodular cast iron. For this investigation it was necessary to have sample containing chunky graphite.

Test melt was prepared in medium frequency induction furnace. Sandwich treatment of base melt at 1480°C with 1.8 wt. % FeSiMg (44-48 wt. % Si, 3.5-3.8 wt. % Mg, 0.9-1.1 wt. % Ca, 0.5-1.2 wt. % Al, 0.6-0.8 wt. % RE, Fe bal.) and 0.2 wt. % cover alloy (46-50 wt. % Si, 1.8-2.2 wt. % Ba, 0.4-0.6 wt. % Ca, 0.5-1.0 wt. % Al, Fe bal.) was carried out. To raise Ni, 0.7 wt. % Ni was added in the ladle. 0.4 wt. % of Ce based commercial inoculant (70-76 wt. % Si, 1.5-2.0 wt. % Ce, 0.75-1.25 wt. % Al, > 1 wt. % O and S, Fe bal.) was used for in-stream inoculation. The metallic charge consisted of a mixture of 70 wt. % grey pig iron (Sorelmetal[®]), 21 wt. % returns, 9 wt. % steel scrap, 0.1 wt. % SiC and 0.2 wt. % FeSi. SiC (~92 wt. % SiC) was added to increase C and Si contents and the nucleation ability of the melt. FeSi 70 % was added to increase silicon content.

The melt treatment for spheroidization and inoculation are described in previous work [9]. Pouring temperature for both moulds was 1380°C and pouring time 27 s.

Both moulds were equipped with three K-type thermocouples, two in the mould cavity (position 1 and 2) and one in the mould (position 3) for recording the cooling curves during solidification, Fig. 2. Horizontal position of thermocouples is determined with distances a, b and c which amounted 46 mm, 36 mm and 52 mm in first mould and 35 mm, 21 mm and 69 mm in the second mould. Melt was poured through the feeder located at the top of the casting so the risk of the fracture of the quartz-tube was too high and it was not possible to place thermocouple in thermal centre of the casting.

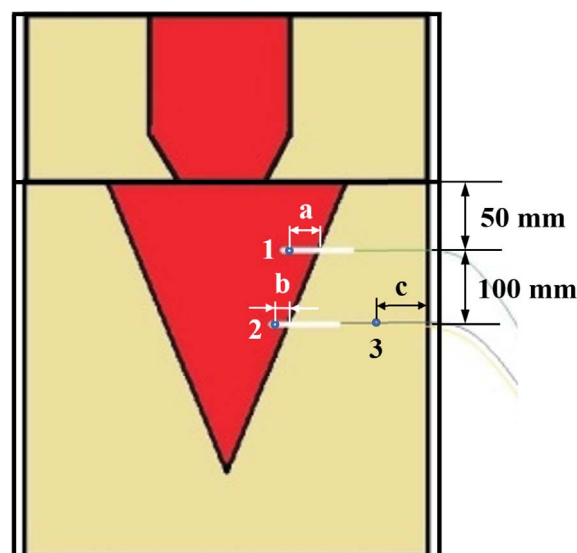


Fig. 2. Location of thermocouples in the mould

According to measured data from the cone blocks, melt characteristics and heat transfer coefficient between casting and mould were adjusted in the ProCAST[®] simulation software.

Just before pouring the melt in the mould, the chilled coupon was analysed by optical emission spectrometer (ARL 3460). Carbon and sulphur content were determined with carbon/sulphur analyzer (ELTRA CS-800). Chemical composition before in-stream inoculation is listed in Table 1. It was calculated for in-stream inoculation to raise Si content approximately 0.29 wt.% giving the carbon equivalent, CE, of 4.18 using equation $CE = \% C + 0.33 (\% Si + \% P)$.

TABLE 1

Chemical composition of the melt

Mass fraction, wt. %						
C	Si	Mn	S	P	Mg	Co
3.51	1.75	0.16	0.013	0.032	0.036	0.017
Ni	Cr	Cu	Sn	Mo	Ti	Al
0.72	0.03	0.016	0.004	0.003	0.007	0.0095

At the time of pouring, one standard cup for thermal analysis (TA-cup) was also filled with the test melt and cooling curve was recorded using ATAS[®] system. The obtained cooling curve was subsequently analyzed for determining the characteristic solidification temperatures.

Each cone block was afterwards sectioned along the vertical symmetry plane for evaluation of the zone affected by CHG. The macrograph was taken and used for the determination of CHG area in the cross-section. The area affected by CHG was analysed using software for automatic image processing (Analysis® Materials Research Lab). Also, samples from different zones in vertical central axis of the cone block were prepared for the metallographic analysis by means of optical microscope (Olympus BX 61). The microstructure was analyzed in different zones of the cone block, with and without CHG.

3. Results and discussion

Fig. 3 shows the cooling curve obtained with TA cup. All the characteristic temperatures evaluated from the cooling curve are listed in Table 2. The recorded cooling curve of the spheroidized melt showed that the solidification proceeds in two steps, much like description given by Chaudhari et al. for near-eutectic alloys [10]. The initial eutectic reaction (starting from T_{ES}) is related to the coupled growth of the austenite and primary graphite nodules, while the second solidification step (starting from the T_{EU}) is referred to the nucleation of secondary nodules and growth of the corresponding eutectic nodular graphite cells. The TA curve shows two significant thermal arrests. The first thermal arrest corresponds to the liquidus temperature, T_L . Second observed thermal arrest corresponds to the bulk eutectic plateau. T_{EU} and T_{ER} stands for the minimum and maximum bulk eutectic temperatures. The temperature of the initial eutectic reaction T_{ES} (eutectic start) was not recorded because ATAS® system was not equipped with this modul. Using this curve, the nucleation of the melt can be estimated and it has also been observed in work [7] a relationship between the recalescence measured on TA cup and the volume of the blocks affected by CHG.

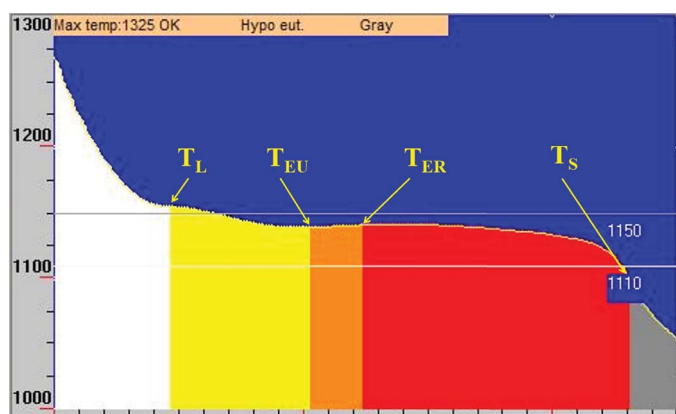


Fig. 3. Cooling curve recorded on TA cup

TABLE 2

Characteristic temperatures (°C) evaluated on TA cup

T_L	T_{EU}	T_{ER}	T_S	GRF1	GRF2
1155	1139	1141	1096	94	37

Cooling curve and first derivative on cone block were recorded in mould 2 with thermocouple at position 1 ($a = 35$ mm), Fig. 2. After analysis of all measurements only one cooling curve was appropriate for further investigation. It is very difficult to obtain good experimental cooling curve in foundry conditions. According to data from one experimental cooling curve, Fig. 4, it is possible to adjust heat transfer coefficient and melt characteristics in simulation software and calculate cooling conditions of any location in investigated casting. All characteristic temperatures obtained from the first derivative of the cooling curve for spheroidized melt with in stream inoculation are listed in Table 3. Observed characteristic temperatures on experimental cooling curve are: T_N – nucleation temperature, T_L – liquidus temperature, T_{ES} – eutectic start temperature, T_{EU} – minimum eutectic temperature, T_{ER} – maximum eutectic temperature and T_S – solidus temperature. Cooling curve obtained from the cone block has the same overall features as the corresponding recorded on TA cup, i.e., eutectic solidification proceeds in two steps, thus the two reactions have almost merged in one single thermal arrest for cone block castings. According to literature and first derivative this arrest is related to the start of bulk eutectic reaction. Slightly arrest apparent for pre-eutectic reaction was observed. In cooling curve obtained with TA cup, this pre-eutectic reaction

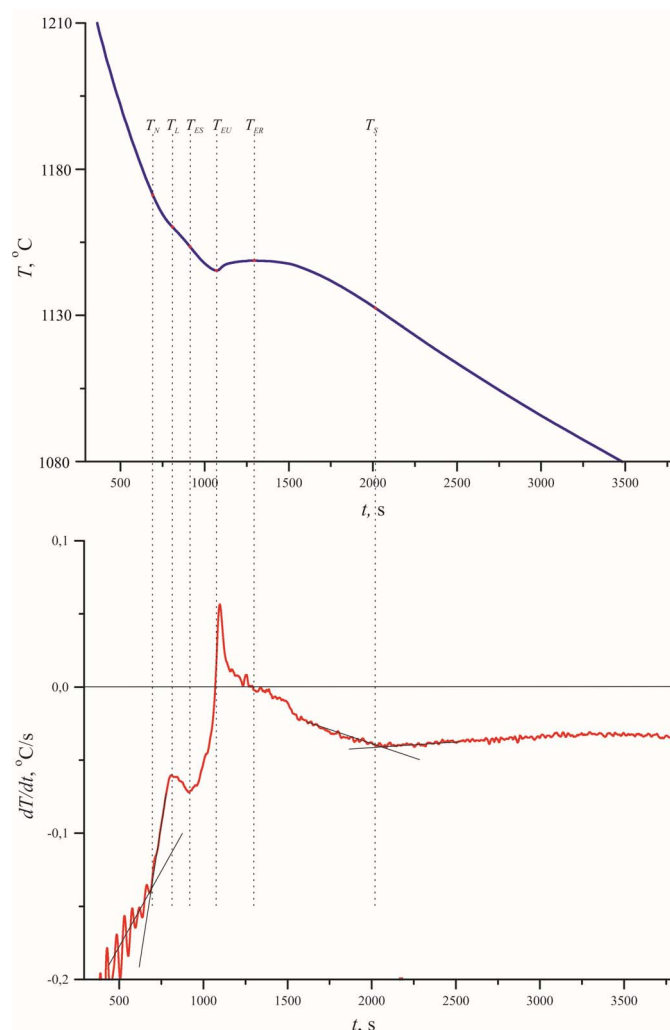


Fig. 4. Cooling curve recorded on the cone block and its first derivative

is apparent as thermal arrest. All the characteristic temperatures are shifted to higher values in cone block because of the lower cooling rates. Also, a higher recalescence value is observed on the block. Although first (T_L) and second (T_{ES}) arrest were hardly observed on the cooling curve or did not show up, they can be obtained by means of its first derivative. Plotting of first derivative curve offers easier interpret of cooling curve and is useful in detecting various events during solidification. According to the [8] and previous work the recorded cooling curve appear to be representative of the solidification at the scale of blocks.

TABLE 3

Characteristic temperatures ($^{\circ}\text{C}$) evaluated on cone block

T_N	T_L	T_{ES}	T_{EU}	T_{ER}	T_S
1168.3	1160	1152.3	1145.4	1148.7	1132.2

Using cooling curve parameters measured in the cone block, melt characteristics and heat transfer coefficient between casting and mould were adjusted in the ProCAST[®] simulation software. Simulated cooling curve adjusted according measured data from the cone block is also shown in Fig. 5 and is in good agreement with experimental cooling curve, which means that simulated curve can be used to interpret solidification behavior of the melt at every point in the casting.

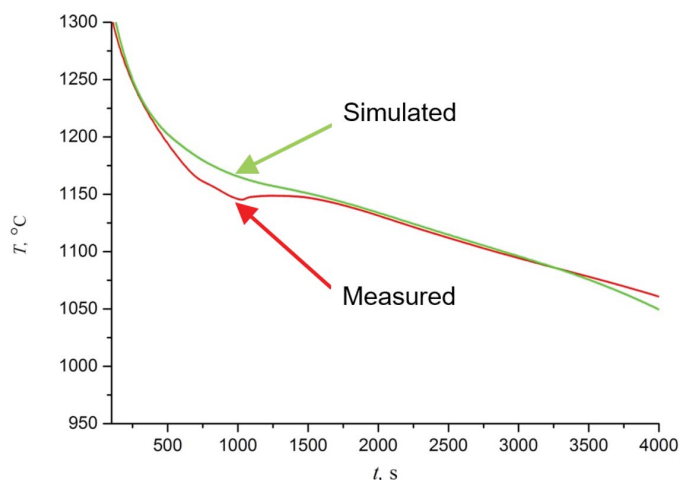


Fig. 5. Cooling curve recorded on the cone block and simulated cooling curve

In a second step of analysis, metallographic inspection of the cross-sections of the cone blocks was performed. The appearance of CHG can be easily seen in the cross-section of the cone block as darker zone, Fig. 6a). Higher Ni content caused CHG formation in heavy-section nodular iron casting. CHG appeared concentrated in the thermal centre of the cone block, below the feeder. CHG affected area amounted 13% of the cross-section.

Results of simulation showed that calculated thermal modulus has highest value in the area near the top of the casting, Fig. 6b), and amounts 3.61 cm. This area indicates high possibility of CHG occurrence. Aside this area the thermal modulus is

lower. As it can be seen, the predicted area of CHG occurrence shows good agreement with the experimental results.

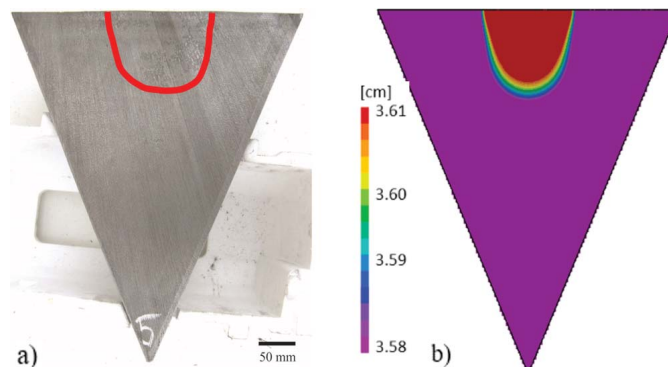


Fig. 6. Cross-section of the cone block and thermal modulus (cm)

Another numerical simulation result; time to solidus, could also be used as indicator of CHG formation, Fig. 7. Three different positions on central axis of the cone block (4, 5 and 6) with three different calculated times to solidus (3650 s, 1600 s and 500 s) were chosen for microstructure analysis. Position 4 is related to the area where CHG formation was observed, while positions 5 and 6 are outside the area affected by CHG. Micrographs taken are shown on Fig. 8.

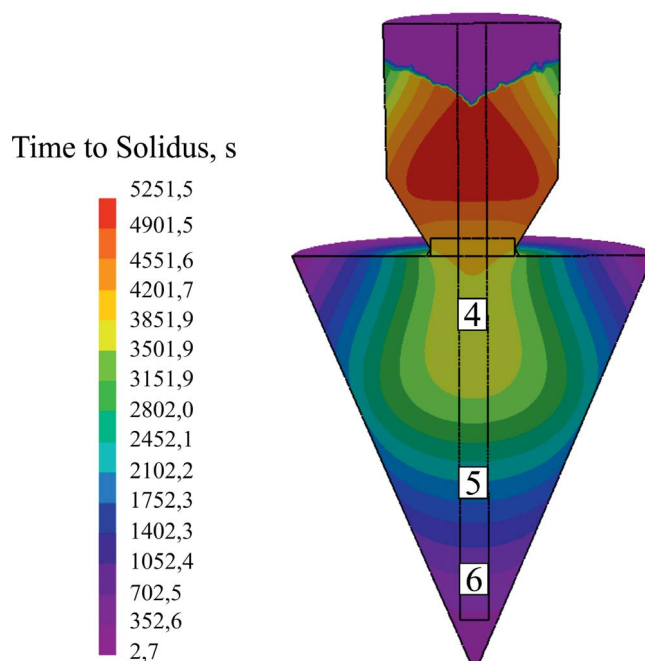


Fig. 7. Time to solidus results at cross-section of the cone block

Microstructure examination of chunky graphite, position 4, revealed that chunky graphite formed eutectic cells. Aside the CHG cells, usual microstructure with nodular graphite can be observed. These nodules are larger in size and reduced in number than the nodules outside the CHG zone. On positions 5 and 6, with lower times to solidus, graphite nodules with good nodularity are dominant. They are equally distributed in the metal

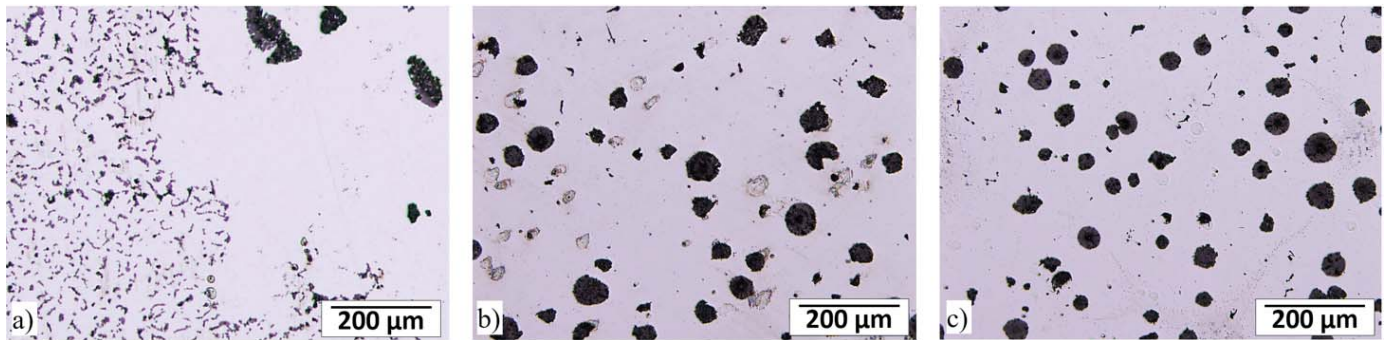


Fig. 8. Micrographs with various time to solidus. a) position 4, 3650 s, b) position 5, 1600 s and c) position 6, 500 s

matrix, with some degenerative nodules of smaller dimensions in position 5. Outside the thermal centre of the casting no chunky graphite was detected.

Optical micrograph at position 1 of the thermocouple, where experimental cooling curve was recorded is shown in Fig. 9. Time to solidus at the location of the thermocouple corresponds to location 5 on central cone axis and the micrographs are well matched.

According simulated times to solidus and microstructure analysis of cone block, below 2600 s to solidus, there is no danger of CHG formation. Positions close to thermal centre of the cone block, where time to solidus amounts from 3800 s to 2800 s, are prone to CHG formation. The higher the time to solidus, the higher the possibility of CHG formation.

After the comparison of macro- and microstructural features with the simulation software results, it could be observed that thermal modulus and time to solidus are related with prediction of CHG affected area in nodular iron castings.

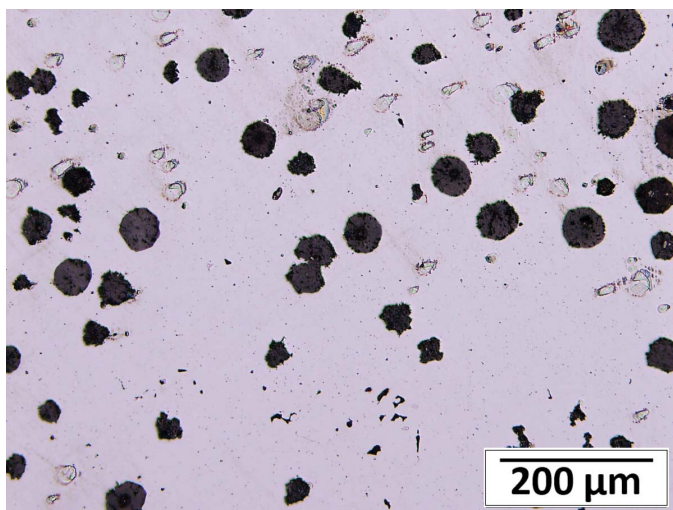


Fig. 9. Micrograph at position 1 of the thermocouple

4. Conclusion

Chunky graphite has been recognized for a long time as one of the major problems in production of heavy section nodular cast iron. Prediction of CHG formation on the base of numerical

simulation and experimental data was investigated. For a given melt a relationship between the area of the cone block affected by CHG and simulation software results, i.e., thermal modulus and time to solidus has been observed. If the chemical composition of the melt is known, experimental cooling curve data can be used to adjust simulation for estimating critical areas which are susceptible to the formation of CHG. This can be used in designing complex geometry of heavy-sectioned castings to avoid CHG.

Acknowledgements

This work is partially supported by the foundry MIV d.d. Varaždin.

REFERENCES

- [1] J. Lacaze, L. Magnusson Åberg, J. Sertucha, Review of microstructural features of chunky graphite in ductile cast irons, in: Keith Millis Symp. on Ductile Cast Iron 2013, AFS, Nashville (2013).
- [2] I. Riposan, M. Chisamera, S. Stan, *China foundry* 7 (2), 163-170 (2010).
- [3] R. Källbom, K. Hamberg, L.E. Björkegren, Chunky Graphite – Formation and Influence on Mechanical Properties in Ductile Cast Iron, in: Proc. of the Gjutdesign 2005, VTT Symposium (2005).
- [4] P. Ferro, A. Fabrizi, R. Cervo, C. Carollo, *J. Mater. Process Tech.* 213, 1601-1608 (2013). DOI:10.1016/j.matprotec.2013.03.012.
- [5] H. Loblich, *Giessereiforschung* 58 (3), 2-11 (2006).
- [6] O. Knustad, L. Magnusson Åberg, Chunky Graphite – effects and theories on formation and prevention, in: F. Unkić (ed.), International Foundrymen Conference – IFC 2014, Opatija (2014).
- [7] J. Sertucha, R. Suarez, I. Asenjo, P. Larranaga, J. Lacaze, I. Ferrer, S. Armandariz, *ISIJ International* 49 (2), 220-228, (2009).
- [8] P. Larrañaga, I. Asenjo, J. Sertucha, R. Suarez, I. Ferrer, J. Lacaze, *Metall. Mater. Trans. A*, 36A, 654-661 (2009). DOI: 10.1007/s11661-008-9731-y
- [9] I. Mihalic Pokopec, P. Mrvar, B. Bauer, *Materials and Technology* 51 (2), 275-281 (2017). DOI:10.17222/mit.2015.355.
- [10] M.D. Chaudhari, R.W. Heine, C.R. Loper, *AFS Transactions* 82, 379-386, (1974).