Available online at www.sciencedirect.com

ScienceDirect

Ciência & Tecnologia dos MATERIAIS

http://ees.elsevier.com/ctmat

Ciência & Tecnologia dos Materiais 29 (2017) e27–e33

Special Issue "Materiais 2015"

The use of thermal analysis to predict the dendritic coherency point on nodular cast iron melts

Vítor Anjos[a, *], Rüdiger Deike[b], Carlos Silva Ribeiro[c]

[a]*OCC GmbH; Eickner Str.111, 41063 Mönchengladbach – Germany*
[b]*Universität Duisburg-Essen, Institut für Technologien der Metalle, Friedrich-Ebert-Str.12, 47119 Duisburg /Laar – Germany*
[c]*CEMUC FEUP, Universidade do Porto, Faculdade de Engenharia, Rua Dr. Roberto Frias, 4200-465 Porto – Portugal*

Abstract

Nowadays there is a growing interest in studying the influence of primary austenite structure and the formation of graphite particles during solidification to correlate with the formation of microshrinkage or even shrinkage defects on castings.
In order to obtain information on the development of the dendritic structure, advanced thermal analysis techniques were applied in the study of hypoeutectic and close eutectic melt compositions to determine the occurrence of the dendritic coherency point (DCP). The occurrence of the DCP has a major importance on metal yield through the design of the in-gate and feeding system of a casting as it determines the available time for feeding and take advantage of the internal expansion inside the mould cavity due to graphite precipitation.
To correlate the thermal analysis results and the solidification sequence of the metallographic constituents to identify the occurrence of the DCP, quenching of the thermal analysis sample during solidification was performed to freeze the solidification process at a given characteristic stage of the solidification curve. This has allowed the observation of the stable solidification structure that has developed until the time of quenching.
The work performed allowed a better understanding of the solidification structure of hypoeutectic nodular cast iron melts and the occurrence of the dendritic coherency point.

© 2017 Portuguese Society of Materials (SPM). Published by Elsevier España, S.L.U. All rights reserved.

Keywords: Dendritic coherency point (DCP); thermal analysis; nodular cast iron.

1. Introduction

As in any material, the knowledge of the solidification process is important to optimize mould filling, casting soundness and the microstructure. Primary austenite is characterized by being the first crystals to nucleate and grow from the liquid, in melts with hypoeutectic compositions [1,2]. The growth of primary austenite follows a typical non-planar solidification interface. The most usual non-planar interfaces are cellular and dendritic that often lead to micro-segregation and formation of secondary phases.

Dendritic coherency is a phenomenon in solidification of alloys with dendritic growth that corresponds to the moment when the individual dendrites first impinge with their neighbours and is often referred to in literature as *"dendritic coherency point"* or simply DCP [3,4].
The importance of this solidification phenomenon is that it will influence greatly the ability of the liquid melt to flow in the mould cavity. During solidification, the formation of the so called "mushy zones" occurs, that correspond to areas of coexistence of liquid and solid phases. As long as the solid phase is free (in suspension) to flow with the liquid and the existing columnar structure is short, there will be a good flow of melt through the mould cavity. However, once dendritic coherency is reached, a rigid

* Corresponding author.
E-mail address: vitor.anjos@inbox.com (Vítor Anjos)

© 2017 Portuguese Society of Materials (SPM). Published by Elsevier España, S.L.U. All rights reserved.
http://dx.doi.org/10.1016/j.ctmat.2017.05.003

structure is formed, similar to an interconnected skeleton. After the occurrence of the dendritic coherency, the flow of melt is only possible through the inter-dendritic spacing of the solid structure, that will become smaller as the solidification proceeds and the dendrites continue to ramify and coarsen.

Once dendritic coherency has occurred, the interdendritic feeding mechanism starts and the flow of the melt is highly conditioned (feeding velocity will decrease) by the presence of a solidified "skeleton" structure. The point of dendritic coherency is often considered in literature as the most critical point in the feeding of a casting because it is the one where more constrains to the melt fluidity and feeding velocity are imposed [2].

One method to determine the time of occurrence of the dendritic coherency point is using advanced thermal analysis, being already used often for aluminium alloys [3]. There are three methods normally used to determine the dendritic coherency point. Bäckerud *et al.* [5] proposed a thermal analysis method that uses two thermocouples: one positioned at the thermic centre and another close to the samples surface, where the dendritic coherency point corresponds to the first significant minimum in the temperature difference curve (ΔT) between both cooling curves (Fig. 1 a)).

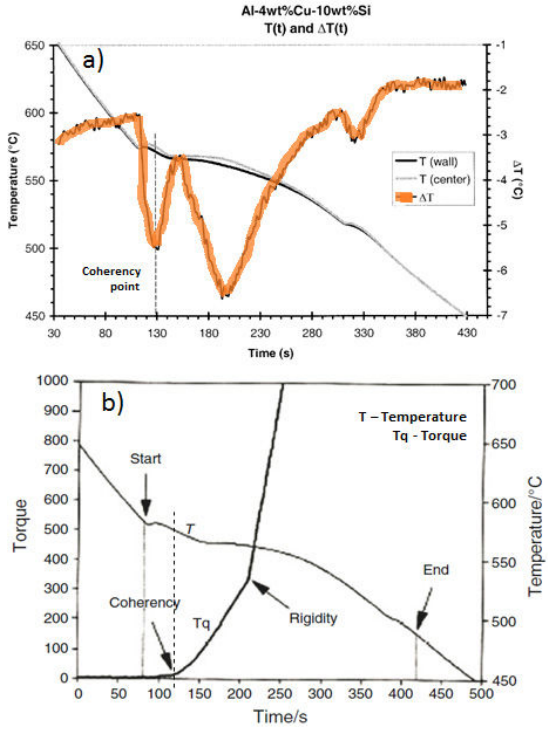

Fig. 1. Determination of the dendritic coherency point using thermal analysis following the models proposed by Bäckerud (a) [5] and Chai (b) [6].

The second method, developed by Chai *et al.* [6], is based on rheological technique, that uses a paddle stirrer to identify the dendritic coherency point due to the changes in the mechanical strength (increase in torque) when the mushy zone is formed (Fig. 1 b)).

The third method, presented and tested by Djordjevic *et al.* [4], uses the model proposed by Bäckerud, but with only one thermocouple at the centre of the sample. Djordjevic indicated in his research that the one thermocouple technique can recognize the dendritic coherency point as good as the two thermocouples technique. He used the temperature difference curve from the two thermocouples method from Bäckerud to find a match in a single dT/dt versus temperature curve from a single thermocouple system, indicating that this curve reflects the thermal conductivity change during solidification and therefore can be used to determine the dendritic coherency point (Fig. 2 a)) [4].

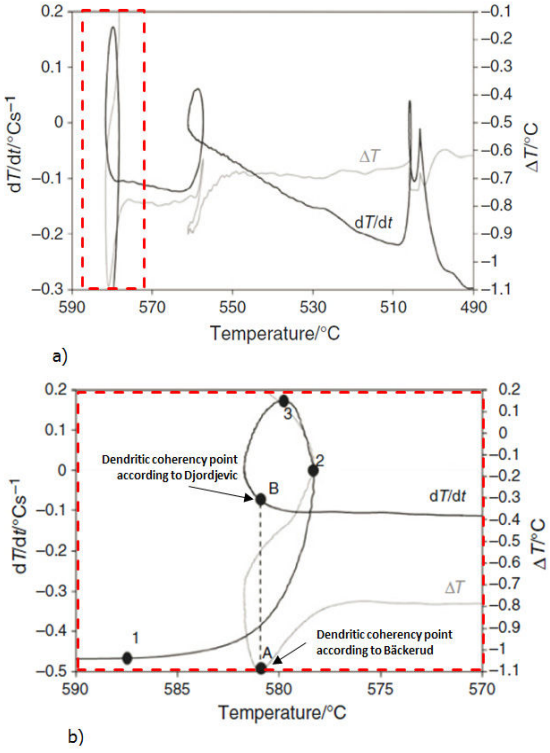

Fig. 2. Method of dendritic coherency point determination according to Djordjevic for aluminium alloys: a) dT/dt curve versus temperature and ΔT versus temperature curve (following the method from Bäckerud); marked red is the region of primary dendritic nucleation and growth; b) Detail view of the region marked red in figure a), with indication of the dendritic coherency point [4].

The determination of the dendritic coherency point for cast iron, using thermal analysis, would allow a better determination of the time available for liquid and mass feeding at a given region of the casting before

restrictions due to the development of the dendritic network occur. This would therefore allow a more precise dimensioning of the feeding system and specially the feeder necks to freeze just before the occurrence of dendritic coherency.

2. Experiment and Results

In this experiment, the method for determination of the dendritic coherency point, usually applied to aluminium alloys, was applied to hypoeutectic nodular cast iron melts. The identification of the DCP in solidification curves from nodular cast iron was later validated by freezing tests through quenching samples during solidification to observe the dendritic structure before and after the identified DCP.
Once the main objective is to use the thermal analysis curves to estimate the DCP, the method proposed by Bäckerud (that is also the base method behind Djordjevic's model) was applied to a standard nodular cast iron melt.

2.1. Experimental procedure for determination of DCP in SG melts using thermal analysis

In the experiments, a dual-chamber thermal analysis cup AccuVo® (Fig. 3) was used, where in one chamber the hot junction of the thermocouple was placed at the centre, and in the other chamber the hot junction is close to the wall of the cup, as presented in Fig. 3 c). Using this cup, the two thermocouples experimental principal used by Bäckerud is recreated.

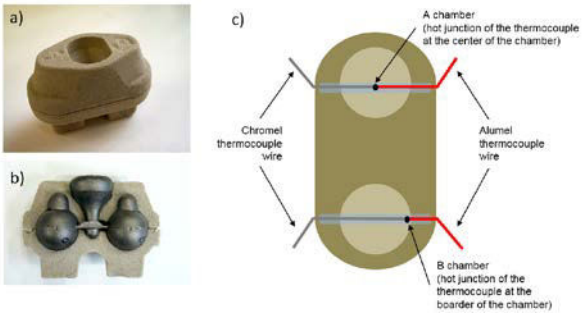

Fig. 3. Thermal analysis cup AccuVo from OCC GmbH: a) external view of the cup, b) image from the internal sample body shape and c) top view of the bottom half of the AccuVo cup showing the disposition of the two termocouples' hot junction for the DCP determination test.

This experiment was performed using a nodular cast iron melt, at a foundry that uses hypoeutectic melt compositions. The sampling to the thermal analysis cup was done after nodularization and inoculation treatment of the melt.

Two samples were made in this test to achieve two thermal analysis curves with different liquidus temperatures. One sample was taken from a melt with higher liquidus temperature (DCP_Hypo_1) and another from a melt with lower liquidus temperature. The liquidus temperatures and the main chemical elements content of the melt are presented in Table 1.

Table 1. Main spectrometer data and liquidus temperature from the samples used on the dendritic coherency point trials, all with hypoeutectic solidification morphology.

Melt type	DCP_Test_1	DCP_Test_2
Liquidus temperature (°C)	1190	1152
C (%)	3.23	3.52
Si (%)	2.21	2.43
Mn (%)	0.34	0.30
P (%)	0.012	0.012
S (%)	0.006	0.006
Cu (%)	0.031	0.032
Sn (%)	0.010	0.010
Mg (%)	0.032	0.043

2.2. Results from the experiment to determine the DCP in SG melts using thermal analysis

The solidification curves obtained from the tests are presented in Fig. 4. In the thermal analysis software results, the red curve refers to the thermocouple in the centre, and the green curve the thermocouple at the border.
From Fig. 4 a), sample DCP_Test_1, a small detachment between both solidification curves can be observed between the liquidus (Liq) and eutectic low point (EuLo). Following Bäckerud's model, a curve representing the temperature difference between the solidification curve in the centre and in the wall is also plotted together with the two solidification curves. According to Bäckerud's model, the dendritic coherency point will correspond to the first minimum in the temperature difference curve occurring after the start of solidification (liquidus temperature). The dendritic coherency point is also indicated in the same figure. A projection of this point in the 1st and 2nd derivative curves (bottom graphic from Fig. 4 a)) shows that it approximately matches the first maximum in the 1st derivative curve and the zero of the 2nd derivative, that correspond to the MIR point (MIR is the point of maximum cooling speed between the liquidus point and the low eutectic temperature point in the solidification curve) in the solidification curve from the centred thermocouples.

More exactly, and after a detailed observation of the 1st derivative curve, the dendritic coherency point matches the point where the cooling rate of the red curve turns higher than the one from the green curve (the 1st derivative lines cross each other), which occurs just less than a second after the maximum point.

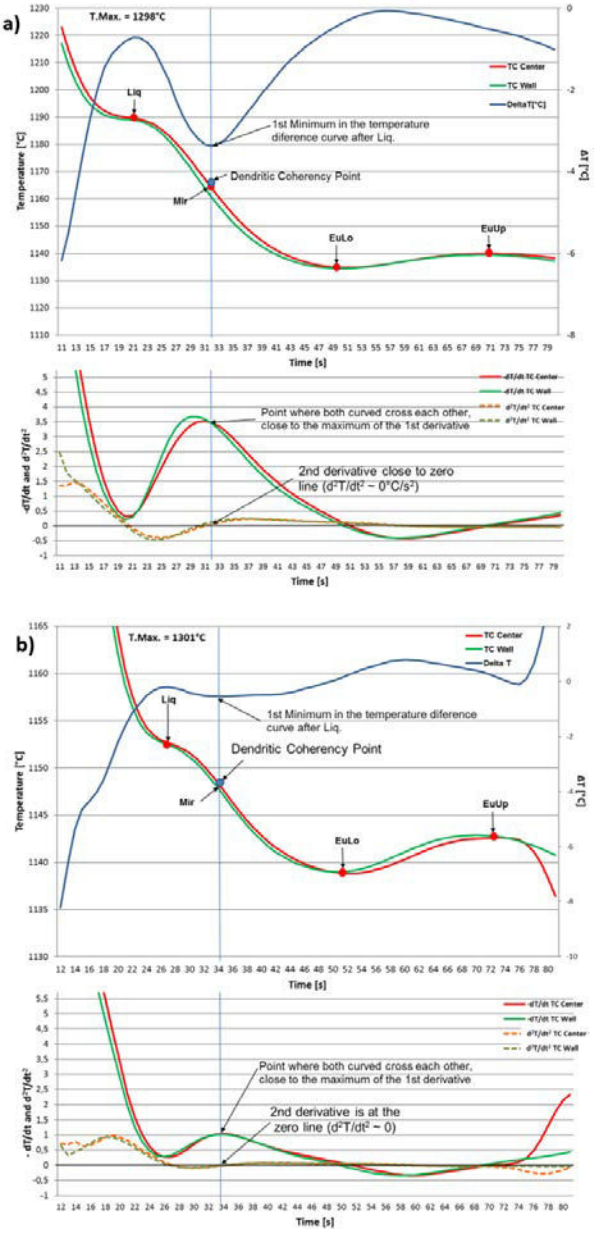

Fig. 4. Determination of the dendritic coherency point, using Bäckerud's model, for the samples: DCP_Test_1 (a) and DCP_Test_2 (b).

In Fig. 4 b), for a sample DCP_Test_2 with a lower liquidus temperature than DCP_Test_1, the same observations made for Fig. 4 a) are valid. Also here an exact match exists between the dendritic coherency point and the moment when the cooling rate of the red curve turns higher than the cooling rate of the green curve. In this specific case, it occurs shortly before the maximum cooling rate is achieved. The DCP on DCP_Test_1 occurs 12s after the liquidus temperature, and on DCP_Test_2 it occurs 8s after the liquidus temperature. This indicates that decreasing liquidus temperature will reduce the time interval to reach DCP, after solidification start.

The results from Fig. 4 indicate that the MIR point on the thermal analysis curve from hypoeutectic nodular cast iron is the point of DCP. Based on this information and in order to have a microstructural evidence that confirms this finding, quenching tests were made on a hypoeutectic melt, before and after reaching the MIR point during solidification, in order to observe the dendritic structure in development.

2.3. Experimental procedure for validation of DCP point determined through thermal analysis

The quenching test was made by immersion of the thermal analysis cup, after taking the melt sample, in a solution of brine and ice. It is expected that this rapid solidification of the melt sample will cause the following transformation of the different phase constituents in the sample:

- Liquid metal → Cementite (Fe_3C)
- Austenite → Martensite or Pearlite
- Graphite → Graphite

For the execution of this experiment, a thermal analysis system was equipped with two stations using the thermal analysis AccuVo® cup. From the same ladle, a sample was taken from the melt after treatment (nodularization and inoculation) and poured simultaneously into the two AccuVo® cups. At one cup, the thermal analysis test proceeded normally until the end of solidification, and the other cup was used to be immersed in the brine water for interrupting the normal solidification process. The acquisition of the cooling curve data of the sample quenched continued after the sample was immersed in brine until the end of solidification was reached.

The iron used for these trials was a hypoeutectic (determined by thermal analysis) GJS400 grade, and the trials procedure is illustrated in Fig. 5.

The test was repeated two times (for two ladles) in order to allow the interruption of the normal melt solidification before and after DCP.

All samples were later analysed at the laboratory for metallographic evaluation.

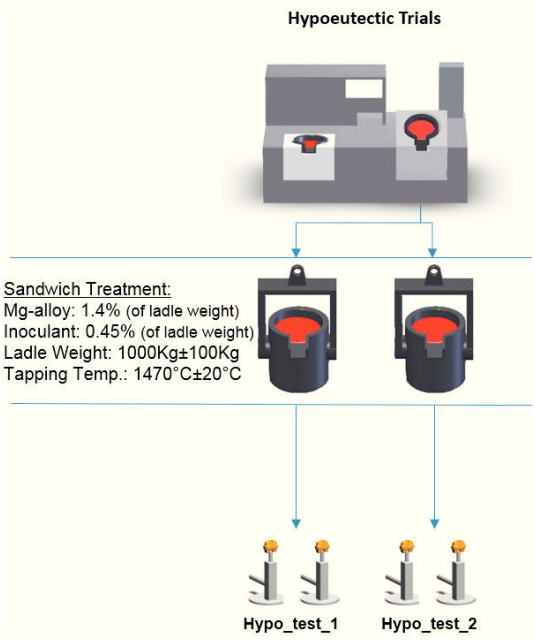

Fig. 5. Illustration of the trials procedure for the quenching of the thermal analysis sample.

The liquidus temperature and main chemical elements content of the melt are presented in Table 2.

Table 2. Chemical analysis from the melt after treatment, used in the quenching test.

Melt type	Test_1	Test_2
Liquidus temperature (°C)	1145	1145
C (%)	3.55	3.53
Si (%)	2.62	2.60
Mn (%)	0.43	0.42
P (%)	0.009	0.009
S (%)	0.004	0.004
Cu (%)	0.031	0.033
Sn (%)	0.012	0.014
Mg (%)	0.046	0.049

2.4. Results from the test to validate DCP point determined through thermal analysis

The solidification curves from the two quenching tests are presented in Fig. 6.
In the first test (Hypo_Test_1), presented in Fig. 6 a), the quenching was done after the plateau corresponding to the liquidus temperature and before the MIR point. On the second test (Hypo_Test_2), the quenching was performed immediately before the low eutectic point and after MIR (Fig. 6 b)).

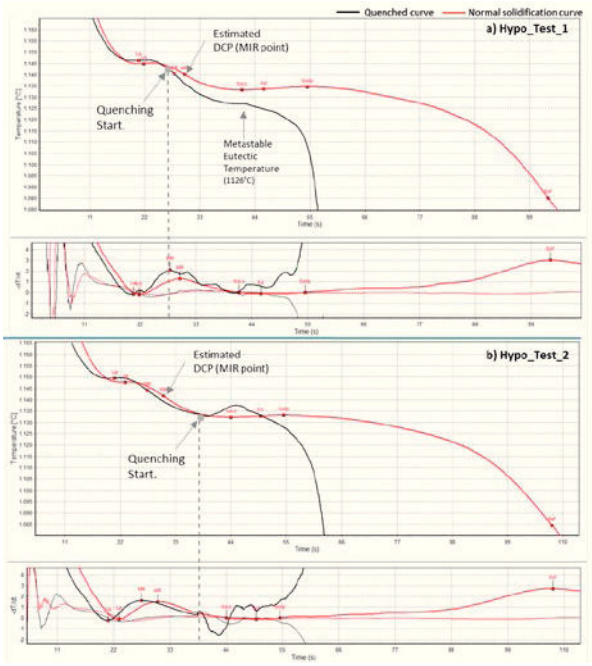

Fig. 6. Solidification curves for the quenching tests before MIR point (a) and after MIR point (b).

The microstructures of the quenched samples are presented in Fig. 7. It is observed that the dendritic structure is more developed in the Hypo_Test_2 sample, compared to Hypo_Test_1, as a result of the late interruption of solidification.
From the Hypo_Test_1 sample, the following qualitative observations are made:

- There is a much lower amount of martensite and graphite (corresponding to solid phase when sample was quenched);
- The dendrite arms are long and spaced between each other;
- The secondary arm branching is almost non-existing;
- There are wide areas of cementite (corresponding to liquid phase before quenching) in the microstructure;
- There are small graphite nodules along the primary arms of the dendrites, with diameters up to 24 µm enveloped in martensite (austenite before quenching).

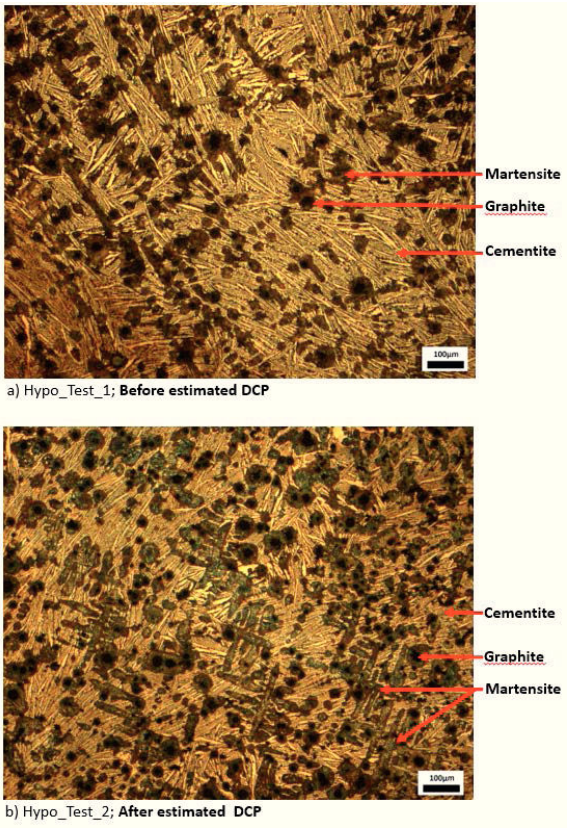

Fig. 7. Micrographs from the samples: Hypo_Test_1 (a) and Hypo_Test_2 (b), at the centre of the samples (Nital 4%).

From the sample Hypo_Test_2, the following qualitative observations are made:

- There is a higher amount of martensite and graphite (corresponding to solid phase when sample was quenched);
- The main dendrite arms are longer and spaced between each other;
- The secondary arm branching is more pronounced;
- There are less areas of cementite (corresponding to liquid phase before quenching);
- There is a higher density of graphite particles, comparing with sample Hipo_Test_1, with diameter up to 36 µm. Nodules are no longer found only alongside the dendrite arms.

In what concerns the graphite structure, the micrographs from the samples are presented in Fig. 8. From the micrographs, it is visible that, in the sample quenched before MIR (Fig. 8 a)), some graphite particles had already solidified. When the sample is etched (Fig. 7 a)), these particles appear, as expected, along the main dendrite arms. For the samples quenched before the eutectic low, the micrograph presented in Fig. 8 b) shows a higher density of graphite particles that appear more randomly distributed. This might suggest that a higher number of eutectic graphite particles have been formed.

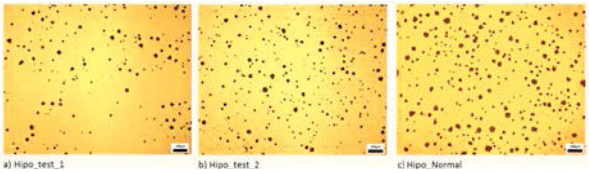

Fig. 8. Micrographs without etching from the quenched samples: Hipo_Test_1 (a), HIpoTest_2 (b) and normal solidification AccuVo-Cup sample (c).

3. Discussion

The determination of the dendritic coherency point presented in Fig. 4 indicates that, for hypoeutectic curves, the DCP occurs approximately at the MIR point between the liquidus temperature and the eutectic low temperature. The micrographs from the quenching test in hypoeutectic morphology melts (Fig. 7 a)) show that, when quenching was done before the estimated DCP (shortly before the MIR point), the dendritic structure has still wide gaps between arms, almost no secondary arm branching and wide areas that were still liquid when the sample was quenched. This indicates that, at that stage, there is yet no dendritic coherency. The sample quenched after the estimated DCP (Fig. 7 b)) shows that the dendritic structure is denser, with lower primary dendrite arm spacing, strong branching with impingement and the liquid regions were smaller when quenching was made, indicating that DCP had already occurred.

For hypoeutectic solidification morphology, the liquidus temperature will influence the time interval between the start of solidification and DCP. Lower liquidus temperatures will result in shorter time intervals to reach DCP.

A surprising observation resulted from Fig. 8 a), where nodules with a diameter up to 24 µm have nucleated alongside the dendrite arms before reaching the MIR. This effect might be due to the segregation of carbon alongside the dendrite arms that will locally increase the carbon content to values around the eutectic carbon content, creating conditions for a graphite nodule to nucleate and grow. For the sample quenched after the MIR point and before the eutectic low temperature (Fig. 8 b)), it is observed that nodules

start also to nucleate from the bulk liquid (they appear randomly dispersed in the microstructure), indicating that the eutectic transformation is taking place.

4. Conclusions

– The DCP in SG melts with hypoeutectic compositions occurs at the point identified as MIR (between liquidus and eutectic low temperature), where the cooling rate in that section of the curve reached a maximum;

– This information, on the time required for a given melt to reach the DCP, is of great importance for the development of the gating and feeding system of a casting;

– The easy determination of the DCP point, by using thermal analysis in foundry environment, can serve as an important information input for the design and simulation of casting's feeder and gating system;

– The results from the quenching test for hypoeutectic samples revealed also that the precipitation of graphite nodules occurs along the primary dendrites, before the eutectic reaction and even before the DCP.

References

[1] ASM Handbook, Castings, vol. 15, ASM International, 2008, pp. 54-55, 317-327, 835.
[2] D.M. Stefanescu, Science and Engineering of Casting Solidification, Springer Science+Business Media, New York, USA, 2009, pp. 1-4.
[3] N.L. Veldman, A.K. Dahle, D.H. St. John, L. Arnberg, Metall. Mater. Trans. A 32A (2001) 147.
[4] M. B. Djurdjevic, J. H. Sokolowski, J. Therm. Anal. Calorim. (2012) 875.
[5] L. Bäckerud, B. Chalmers, Trans. Metall. Soc. AIME 245 (1969) 309.
[6] L. Arnberg, L. Bäckerud, Solidification Characteristics of Aluminium Alloys, Dendrite Coherency, American Foundrymen's Society, Des Plaines, Illinois, USA 3, 1996.