Determination of Eutectoid Transformation Temperatures in Ductile Cast Iron

W.O. Orłowicz, A. Trytek*, M. Mróz, M. Tupaj
Department of Casting and Welding, Rzeszow University of Technology, ul. W. Pola 2, 35-959 Rzeszów, Poland
*Corresponding author. E-mail address: trytek@prz.edu.pl

Received 30.06.2015; accepted in revised form 15.07.2015

Abstract

The paper proposes a methodology useful in verification of results of dilatometric tests aimed at determination of temperatures defining the start and the end of eutectoid transformation in the course of ductile cast iron cooling, based on quenching techniques and metallographic examination. For an industrial melt of ductile cast iron, the effect of the rate of cooling after austenitization at temperature 900°C carried out for 30 minutes on temperatures $T_{Ar1}^{start}$ and $T_{Ar1}^{end}$ was determined. The heating rates applied in the study were the same as the cooling rates and equaled 30, 60, 90, 150, and 300°C/h. It has been found that with increasing cooling rate, values of temperatures $T_{Ar1}^{start}$ and $T_{Ar1}^{end}$ decrease by several dozen degrees.

Keywords: Ductile cast iron, Eutectoid transformation, Cooling rate

1. Introduction

Heat treatment becomes nowadays an obligatory component of technological processes applied in manufacturing ductile cast iron castings with diversified matrix microstructure, e.g.: ferritic, pearlitic, martensite, ausferritic, or combined with various volume shares of these microstructure components. Developing technologically correct heat treatment schedules depend on precise knowledge of temperatures defining both start and end of eutectoid transformation occurring at both heating and cooling. Temperatures defining $A_1$ transformation range in ductile cast iron depend mainly on silicon and manganese content as well as on heating, austenitization, and cooling conditions. Even the change in cooling rate within the range from 30°C to 300°C alone may result in lowering the temperatures limiting the eutectoid transformation range by several dozen degrees Celsius [1,3]. This has a very significant effect on kinetics of the transformation in both anisothermal and anisothermal-isothermal conditions. According to [1], an increase of manganese content in cast iron decreases $A_1$ transformation limiting temperatures whereas high silicon content causes a distinct increase of these temperature limits. The increase of the heating rate from 15°C/h to 300°C/h results a noticeable (up to several dozen degrees) increase of temperatures determining the transformation range ($T_{Ar1}^{start}$ and $T_{Ar1}^{end}$), whereas similar increase of the cooling rate causes similarly significant decrease of $A_1$ transformation temperatures ($T_{Ar1}^{start}$ and $T_{Ar1}^{end}$).

The austenitization temperature adopted in [1] was 900°C. The author has assessed $A_1$ transformation temperatures according to recommendations of applicable Polish standard [2], i.e. by means of drawing tangents to curves representing elongation changes as a function of temperature.

Results reported in [3], obtained with the use of the same methodology, have confirmed similar nature of the effect caused by silicon and manganese and the cooling rate as this presented in [1]. Additionally, it has been stated that the increase of the austenitization temperature from 910°C to 960°C resulted in a decrease of temperature $T_{Ar1}^{start}$ by several degrees Celsius.
Authors of paper [4], appreciating significance of the knowledge of temperatures \( T_{Ac_1}^{\text{start}} \) and \( T_{Ac_1}^{\text{end}} \) and the effect of heat treatment within the eutectoid transformation temperature range at heating on mechanical properties of ductile cast iron, reported that the heat treatment carried out within the \( T_{Ac_1} \) temperature range allowed to improve mechanical properties of ductile cast iron. Despite acknowledging the significance of identification of temperatures \( T_{Ac_1}^{\text{start}} \) and \( T_{Ac_1}^{\text{end}} \), authors of the study do not quote respective values.

Authors of the study presented in [5] have demonstrated that by means of heat treatment carried out within the eutectoid transformation temperature range, it is possible to change pearlite and ferrite percentage shares in the ductile cast iron matrix. Based on empirical formulas developed in [6] and DTA method applied therein, they have determined the eutectoid transformation temperature range for ductile cast iron with various silicon content ranging from 1.88% to 3.19%. They have also noted that for cooling conditions used in DTA method, eutectoid transformation temperatures \( T_{Ar} \) were by 70–80°C lower than the values of these temperatures has been passed over.

Authors of paper [7] studied the susceptibility to cracking observed in ductile cast irons with microstructure shaped with the use of a heat treatment within the eutectoid transformation temperature range at cooling. However, the issue of assessment of values of these temperatures has been passed over.

Papers [8, 9] report on heat treatment of ductile cast iron including austenitization within the range of eutectoid transformation temperatures \( T_{Ac_1} \) aimed at obtaining a characteristic dual matrix structure. In both cases, the authors referred to an assessment of the eutectoid transformation end temperature \( T_{Ac_1} \) quoted in paper [10] where the calculated temperature \( T_{Ac_1}^{\text{end}} \) was 840°C, whereas the presented microstructure patterns for samples quenched from temperature 900°C after austenitization for a period of 20 minutes revealed presence of significant quantities of ferrite. This is an evidence of incomplete course of the austenitization process. Therefore, the presented results indicate uselessness of the formula given in [10] for the purpose of determining the eutectoid transformation temperature in ductile cast iron employed in both papers.

It follows from the presented review of available literature that a number of authors suggest the possibility to establish general relationships applicable to assessment of the effect of chemistry and heating, austenitization, and cooling conditions on the eutectoid transformation start and end temperatures, but precise determination of their values for any specific cast iron grade would require carrying out relevant tests in each individual case.

If follows from experience of authors of the present paper gained in the area of kinetics of phase transitions that the use of the dilatometric test method verified by means of the structure freezing could be considered a reliable an effective way of assessing phase transition temperatures.

The purpose of the study presented in this paper is to prove usefulness of the method for both scientific research and industrial practice.

2. Experimental

The material used in the present study was a cast iron melted in OXITERM gas-fueled rotary furnace. The liquid metal was transferred to a 1000-kg ladle filling it up to half height. In the course of filling the ladle with liquid alloy, Foundrisil master alloy was dosed on the stream of metal as the first stage of inoculation. Spectral analysis of the sample taken from the ladle has shown that percentage content of individual alloying elements fell within the range of the assumed chemical composition. This was a base on which the spheroidization operation was further performed by means of wire method with the use of master alloy Mg24Si50Ce3 in the core. Next, the ladle was topped up with liquid metal as before. After mixing, a sample of liquid metal has been taken for analysis of chemical composition and the process test (bar test) was executed to assess the effect of spheroidization. Positive results were the base for carrying out the second stage of modification. Results of the bar test with the use of longitudinal ultrasonic wave velocity measurements were also positive so the decision to pour the moulds could be taken. The metal was poured into moulds from 90-kg distribution ladles. The melt at temperature 1380°C was cast into a Y-block sand mould prepared in accordance with ISO 1083.

In the course of casting the wedges, alloy samples for chemistry analysis were taken. Composition of the cast iron was as follows: 3.48% C; 2.55% Si; 0.069% Mn; 0.04%Ni; 0.026% Cr; 0.014%Ti; 0.009% S; 0.037% P; 0.041% Mg, and Fe to balance. The material was characterized with ferritic-pearlitic matrix (Fig. 1).

![Fig. 1. Microstructure of the cast iron used in the study: ferrite, pearlite, spheroidal graphite. Magnification ×200, etched in 4% \( HNO_3 \).](image-url)

After cutting off 20-mm thick layers from bases from the wedge-shaped castings, 8-mm thick slices were then cut off from which samples for dilatometric tests were prepared. The samples had the diameter \( d = 4.0,05 \) mm and length \( l = 20.0,05 \) mm. In each sample, a 50-mm deep bore with diameter \( d = 2 \) mm was made for mounting the tip of Ni-CrNi thermocouple which was attached to the sample with the use of capacitance welder. The diameter of thermocouple wires was 0.3 mm. Each thermocouple was checked by means of the comparative method with Class S PtRh-Pt standard thermocouple.
The studies on eutectoid transformation temperatures were carried out with the use of LS4 dilatometer with computer-based recording of elongation and temperature values with the use of dedicated software. In the course of the test, the sample together with thermocouple and a quartz follower coupled with an induction elongation gauge were places in shielding atmosphere of argon.

Around the quartz tube introduced into the dilatometer heating furnace, cast-iron reference specimens for microstructure freezing were placed in straight-through steel tubes at the same height as the dilatometric sample. The samples were welded to thin wires with diameter 0.3 mm ends of which were mounted in clamps located on the upper surface of the furnace.

In the first stage of the study concerning the effect of cooling rate on temperatures $T_{Ar_1^{start}}$ and $T_{Ar_1^{end}}$, their values were determined by means of the tangents method, like in papers [1] and [3]. In the second stage, correctness of the method was verified by means of the sample freezing technique. For the purpose of verification, several temperature values were selected around the critical temperature $T_{Ar_1}$, e.g. $T_{Ar_1^{start}} + 6°\text{C}$, $T_{Ar_1^{start}} + 3°\text{C}$, $T_{Ar_1^{start}} - 3°\text{C}$, and $T_{Ar_1^{start}} - 6°\text{C}$. At the moment of displaying these values on the computer monitor, successive clamps were released and specimens, through openings at tube bottoms being earlier uncovering, fell one after another into the vessel filled with water (Fig. 2).

![Fig. 2. Schematic diagram of the set-up for structure freezing in ductile cast iron samples by means of quenching: 1- straight-through steel tubes; 2- control cast iron dilatometric sample; 3- cast-iron samples for structure freezing tests suspended on wires; 4- clamps fixing sample suspending wires; 5- unwoven cloth obscuring the bore of tubes; 6- charcoal to create shielding atmosphere; 7- vessel filled with water](image)

Based on results of metallographic examination, $T_{Ar_1}$ values determined by means of the dilatometric test method were established more precisely. In case of temperature $T_{Ar_1^{start}}$, the clue was presence of trace ferrite precipitations (Fig. 3), and in case of $T_{Ar_1^{end}}$, presence of trace hardening products.

![Fig. 3. (a) An assessment of the eutectoid transformation start temperature $T_{Ar_1^{start}}$ by means of the dilatometric test method. (b) Microstructure of a sample with structure frozen as a result of quenching from the eutectoid transformation start temperature assessed by means of the dilatometric test method. Hardening products with traces of ferrite precipitations visible at the graphite surface. Etched in 4% HNO$_3$](image)

Results of assessment of eutectoid transformation start and end temperature values at cooling are shown in Fig. 4. The obtained results indicate that with increasing cooling rate, temperatures of both start and end of the eutectoid transformation decrease. The cooling rate increase from $30°\text{C/h}$ to $300°\text{C/h}$ resulted in lowering temperature $T_{Ar_1^{start}}$ by $27°\text{C}$ and temperature $T_{Ar_1^{end}}$ by $38°\text{C}$.

![Fig. 4. Graph showing the relationship between cooling rate and eutectoid transformation temperatures](image)
3. Conclusions

The study reported in this paper proved that changes in the rates at which the alloy is heated up to and cooled down from the austenitization temperature have a significant effect on the eutectoid transformation start and end temperatures. The increase of the cooling rate from 30°C/h to 300°C/h caused reduction of temperature $T_{A1}^{start}$ by 27°C and temperature $T_{A1}^{end}$ by 38°C.

In view of the above, correct determination of the eutectoid transformation temperature range at heating and at cooling for cast iron coming from individual melts with diversified chemistry should be considered a point of departure for developing heat treatment schedules and fine adjustment of their parameters aimed at obtaining an assumed microstructure and thus the required mechanical properties of the material.

References

[1] Bylica, A. (1971). *The effect of silicon and manganese and heating rate on A1 transformation temperatures in ductile cast iron*. Habilitation dissertation. Politechnika Śląska, Gliwice, Poland. (in Polish).

[2] Polish Standard PN-68/H-04500.

[3] Orłowicz, A.W. (1980). *The effect of silicon and manganese and cooling rate on A1 transformation temperatures in ductile cast iron*. Doctoral dissertation. Politechnika Śląska, Gliwice, Poland. (in Polish).

[4] Sołn Cecil, L.A., Ivanova, L.P., Miniajło, T.L. & Pawluczenko, A.A. (1976). Wlianije temperaturyna nagariuwa na mikrostrukturu i svojstwa nieligierowanego czuguna. *Metallowiedienije i Temiczeskaja Obrabotka Metallow*, 6, 29–32.

[5] Hummer, R. & Westerhalt, W. (1979). Untersuchungen zur Wärmebehandlung von Gusseisen mit Kugelgraphit unter besonderer Berucksichtigung der Hertestellung von GGG-50. *Giesserei Praxis*, 1–2, 15–20.

[6] Drapal, S. (1963). Eutektoide Umwandlung von Austenit im Gusseisen und Temperguss. *Giesserei Techn. Wiss. Beih.*, 15, 87–97.

[7] Voigt, R.C., Eldoky, L.M. & Chiou, H.S. (1986). Fracture of ductile cast irons with dual matrix structures. *AFS Transactions*, 94, 645–656.

[8] Katecampe, K., Cerah, M. & Erdogan, M. (2006). Effect of martensite volume fraction and its morphology on the tensile properties of ferritic ductile iron with dual matrix structures. *Journal of Materials Processing Technology*, 178, 44–51.

[9] Sahin, Y., Erdogan, M. & Cerah, M. (2008). Effect of martensite volume friction and tempering time on abrasive wear of ferrite ductile iron with dual matrix. *WEAR*, 265, 196–202.

[10] Andrews, K.W. (1965). Empirical formulae for the calculation of some transformation temperatures. *J. Steel Inst.* 203, 721.