

Comparing the possibilities of austenite content determination in austempered ductile iron

D. Myszka^{a*}, T. Giętka^b

^aInstitute of Precision Mechanics, ul. Duchnicka 3, 01-796 Warszawa

^bDepartment of Materials Science and Engineering, Mechanical Engineering Faculty, University of Technology and Life Sciences, al. Kaliskiego 7, 85-796 Bydgoszcz, Poland

*Corresponding author. E-mail address: myszkadawid@wp.pl

Received 20.07.2011 accepted in revised form 27.07.2011

Abstract

The article presents various methods for assessment of the austenite volume fraction in Austempered Ductile Iron (ADI). Tests were carried out on two types of ADI, i.e. unalloyed and alloyed with the addition of 0.72%Cu and 0.27%Mo, heat treated under different conditions of isothermal transformation to obtain different austenite volume fractions. The test material was then subjected to metallographic examinations, X-ray diffraction (XRD) analysis, an analysis using the author's genuine programme of artificial neural networks, image analysis and magnetic measurements. The results were compared with each other indicating the possibility of a quantitative measurement of austenite and other phases present in cast iron. It was found that different methods of measurement are not fully consistent with each other but show similar results of the austenite content.

Keywords: Austempered Ductile Iron (ADI), Austenite content

1. Introduction

Ausferritic ductile iron, classified recently by a European standard [1], is the cast material that offers a very wide range of interesting properties. The most representative ones, cited most commonly, include large elongation reaching over 10% for the tensile strength higher than 900MPa, or very high tensile strength of 1600MPa recorded in another ADI grade. Besides these properties, ausferritic ductile iron has also a number of other favourable features which make it applicable for various structural components. The best known are the damping capacity, density by 10% lower than that of steel, good machinability prior to heat treatment, hardenability of the casting surface during rolling or ball peening, etc.

The excellent properties of ausferritic ductile iron are due to a properly conducted heat treatment cycle consisting of austenitising and austempering. Both these treatments are extremely important, as they are responsible for the time-related changes of microstructure, and hence for the ADI properties. Austenitising stabilises the carbon content in austenite and makes it more homogeneous, while isothermal transformation, following the rapid cooling from austenitising temperature, is shaping the morphology of a ferrite/austenite mixture (ausferrite), formed at ambient temperature and ultimately responsible for the ADI properties.

Both temperature and time of austenitising have a great influence on the kinetics of this phenomenon and on the results achieved, the most important one being certainly the stabilisation of carbon content in the forming austenite. Temperature allows

adjusting the achievable value of the equilibrium concentration of carbon in austenite, while time controls when (and if at all) this moment will occur. The higher is the austenitising temperature, the faster is the process of the cast iron matrix austenitising. The solubility of carbon originating from the precipitates of graphite increases, austenite becomes more homogeneous, and its grains are growing faster.

Ensuring a reasonably long time of austenitising helps in achieving the equilibrium carbon content in austenite, which is characterised by a specific temperature M_s . The research shows that for the cast iron grades that are convertible into ADI, the M_s temperature will be at a level of 150 to 200°C. This has a significant impact on changes that occur during the isothermal transformation.

The effect that carbon content stabilised in austenite during the austenitising process may have on the kinetics of isothermal transformation can be explained through analysis of the individual stages of this transformation. Isothermal transformation begins after rapid cooling of casting from the austenitising temperature to a temperature of 230 to 400°C (Fig. 1). Figure 2 shows schematically the formation of ductile iron matrix morphology during isothermal heat treatment, compared with the change in M_s temperature. At point A, where the isothermal annealing is very short, the ductile iron will be characterised by an almost completely martensitic matrix with but very scarce precipitates of the lamellar ferrite. This will be due to a nearly complete transformation of the thermally unstable austenite. At point B, the ductile iron will be characterised by a matrix composed of the lamellar ferrite precipitates and undercooled metastable austenite containing

1,0÷1,6% C, formed by carbon saturation from the areas of the growing ferrite. At point C, austenite is already saturated with carbon to a level above 1,6% C, which ensures its stability, and as such will be called undercooled stable austenite. Proceeding further, the transformation starts the process of the precipitation of excessive amounts of carbon in the form of carbides and the formation of a bainitic ductile iron matrix – points D-E. In reality, the above mentioned forms of microstructure are very different in nature and due to microsegregations in cast iron can occur together at practically all stages of the isothermal transformation. Hence follow different phase morphologies in a matrix called ausferritic, i.e. composed of a mixture of lamellar ferrite and carbon-saturated austenite. Therefore, the unreacted austenite may appear also in the areas with elevated content of manganese and molybdenum, where these two elements will act as its stabilisers, hindering effectively the transformation at ambient temperature.

As indicated by the results of numerous studies, the optimum ADI properties depend on the properly adjusted temperature and time of isothermal transformation, providing the highest content of austenite in matrix [2]. For precise determination of the austenite content, the X-ray diffraction method is usually applied. It is, however, suitable only for research purposes, consumes a lot of time and requires highly skilled specialists, and since there are other research methods available, the authors have decided to describe and compare them in this study.

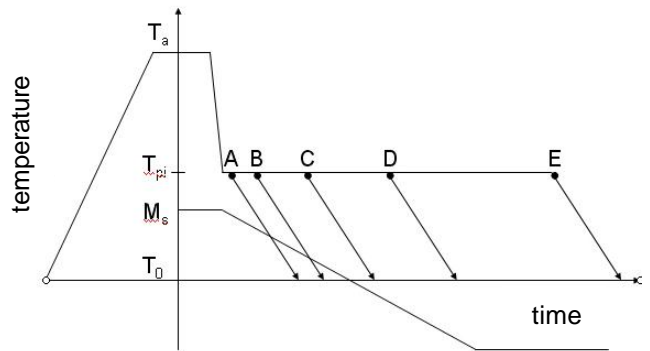


Fig. 1. Flow chart of isothermal transformation in heat-treated ductile iron

2. Methodology

Studies were carried out on unalloyed ductile iron and on ductile iron with small additions of Cu and Mo. Samples of unalloyed ductile iron used in the present research were melted in an industrial, hot blast and acid-lined cupola. Spheroidisation of cast iron was performed with ML5 magnesium alloy rods introduced to the cupola receiver. The liquid metal was inoculated with FeSi master alloy on the tapping spout, and then cast into green sand moulds. The YII shaped ingots were cast separately according to PN EN 1563: 2000. The metal in as-cast state had a ferritic-pearlitic (10%) matrix structure and the correct spheroidal shape of graphite (112 precipitates /mm² of the metallographic section surface area). For further research it was assumed that the base iron for austempering will have a ferritic structure. To produce this structure, the ingots were subjected to a two-stage ferritising annealing. The ferritising treatment covered the lower parts of the YII ingots (stage I at 1050°C/4h and stage II at 680°C/6h).

Specimens for mechanical tests of a \varnothing 10 mm diameter were austenitised in a chamber furnace at a temperature of 900°C, then after 1h the specimens were taken out and put in a furnace with the salt bath at a temperature of 400°C, where they were austempered for 30 min.

On the other hand, specimens of ductile iron with additions of the alloying elements were cut out from the blade of a shot blasting machine, cast in investment process from the ductile iron obtained by melting the metal in a medium frequency induction furnace of 500kg capacity, followed by spheroidising treatment carried out with NiCuMg17 master alloy in a Sandwich process.

Castings of the chemical composition shown in (Tab. 1) were subjected to microstructural examinations (Fig. 2), and then the heat treatment was carried out. Austenitising of specimens was carried out for 2 hours at 900°C, while austempering was conducted in a fluidised bed at 370°C for 60min and at 400°C for 30min. Ausferritic ductile iron of the matrix microstructure shown in (Fig. 3) and mechanical properties presented in Table 2 was produced. Static tensile tests were carried out on specimens with a \varnothing 7mm diameter of the measuring part, while cylindrical \varnothing 12mmx10mm specimens were used for a quantitative phase evaluation done with a LUCIA image analysis programme, for magnetic examinations with ferritoscope, and for an X-ray

analysis on a Rigaku device with an attachment for the analysis of residual austenite; photos were taken with an Olympus IX70 metallographic light microscope.

Table 1.

Chemical analysis of ductile iron used in the studies [% wt.]

Material designation	C	Si	Mn	Mg	Cu	Mo
A	3,65	2,59	0,18	0,06	-	-
B	3,40	2,80	0,28	0,055	0,72	-

Table 2.

Mechanical properties of ductile iron after heat treatment

Sample	R _m [MPa]	R _{0,2} [MPa]	A ₅ [%]
ADI A 400_030	976	716	12,3
ADI B 400_030	832	540	5,7
ADI A 370_060	961	598	15,7

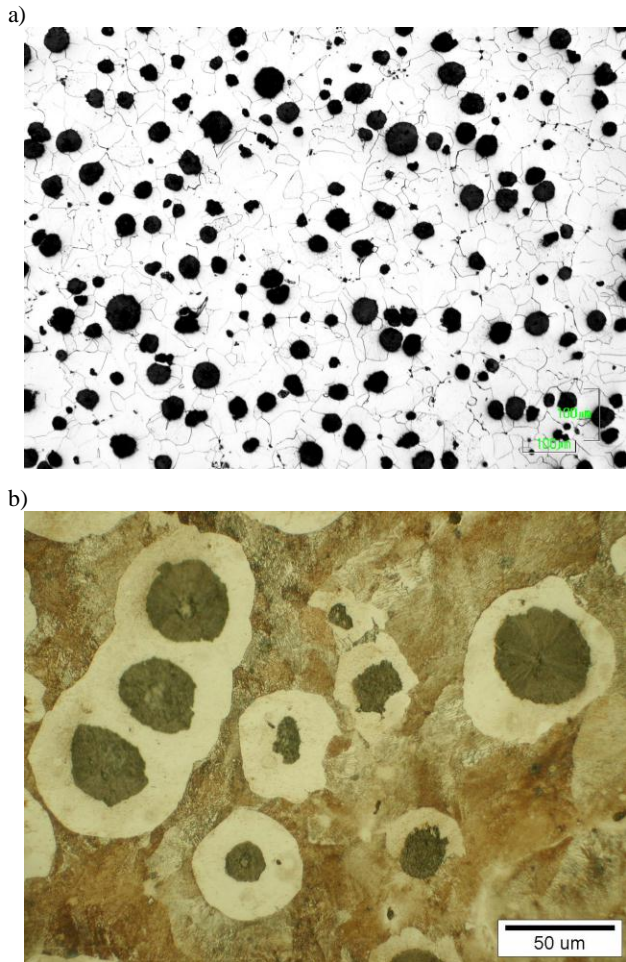


Fig. 2. Microstructure of ductile iron before heat treatment: a) unalloyed - A; b) alloyed with additions of Cu and Mo - B

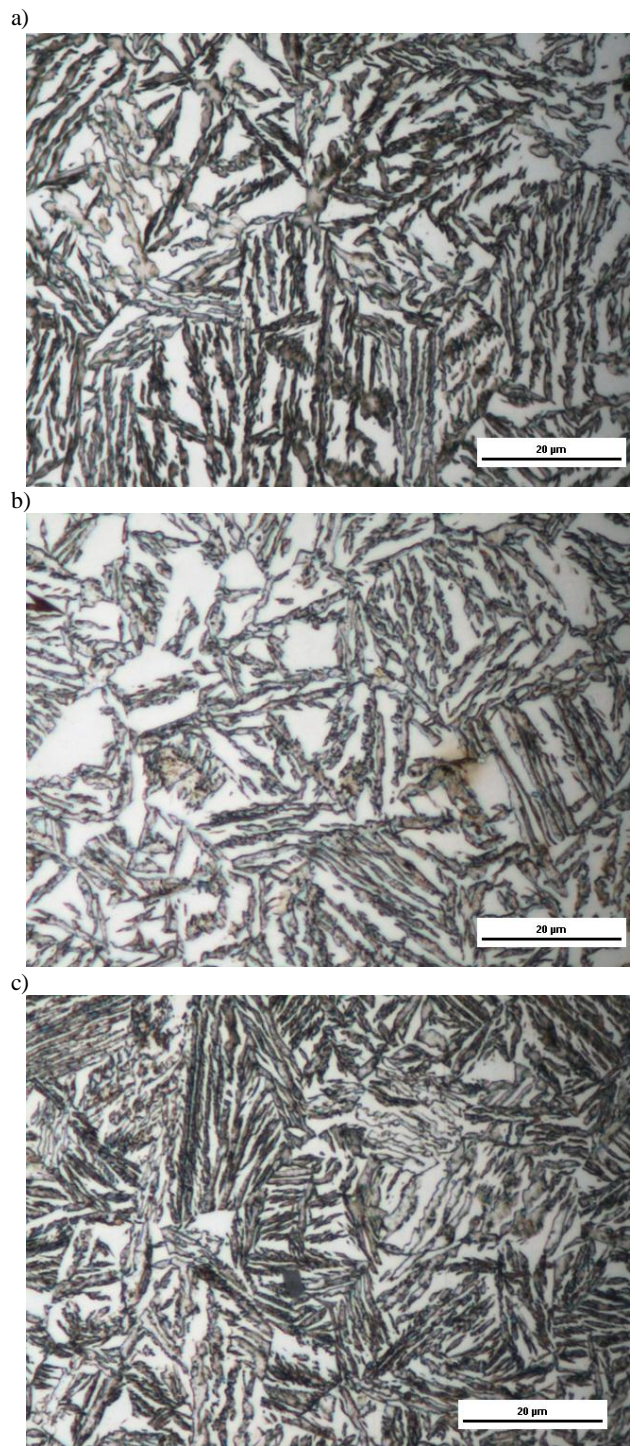


Fig. 3. Microstructure of ausferritic ductile iron: a) ADI A 400_030, b) ADI B 400_030; c) ADI B 370_060

3. Measurements of austenite

Table 3 presents the cumulative results of austenite measurements for different methods used in the research. The successive numbers represent different methods of measurements carried out on the cast iron designated as ADI B 370_060. In each case, a value averaged for minimum three measurements is given (in addition to image analysis, where all the three values are given because of a large scatter of the results).

Table 3.
The results of quantitative measurements of the austenite content in B 370_060 ADI

Method of measurement	Cast iron type	Austenite, %	Ferrite, %	Graphite, %
Measurement by XRD		25,8±1%	-	-
Image analysis	ADI B 370_060	19,1 ^{Fig. 4a}	-	9,7±0,4%
		24,2 ^{Fig. 4b}		
		29,3 ^{Fig. 4c}		
Artificial neural network		29,0±1%	-	-
Magnetic measurement		32,1	58,3±1,5%	-

The basic method in quantitative research of the austenite content in cast iron is X-ray diffraction. The X-ray diffraction analysis for studies of sample A were performed on DRON - 1.5 diffractometer, using iron filtered CoK_α radiation. The following operating parameters of the diffractometer were applied:

- voltage on tube - 36 kV;
- current on tube - 6 mA;
- collimator ports system I - 2, II - 2 III - 2 mm and Soller slits were used for the primary and diffracted beam;
- detector - scintillation counter fed with a voltage of 700 V.

Diffraction pattern recording was performed in an angular range of $2\theta - 49,5 \div 53,5$. In this range, using a step of $0,01^\circ$, the diffraction lines of (110) α phase and (111) γ phase were recorded. The intensity of each interferential line was determined three times by planimetry the area under a curve to the background level.

X-ray diffraction studies were carried out on metallographic polished sections prepared for microscopic examinations.

The volume fraction of austenite (V_γ) in the matrix of austempered ductile iron was determined from formula [3]:

$$V_\gamma = \frac{1}{1 + I_\alpha \cdot I_\gamma^{-1} \cdot R} \cdot 100 \% \quad (1)$$

where:

- V_γ - volume fraction of austenite, %;
- I_α - total relative intensity of diffraction line of (110) α

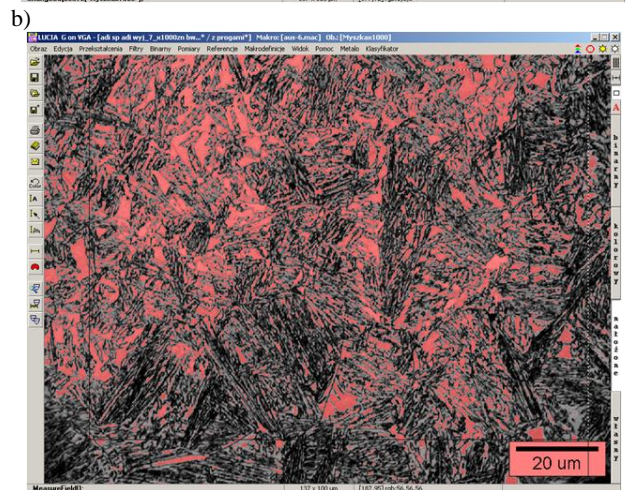
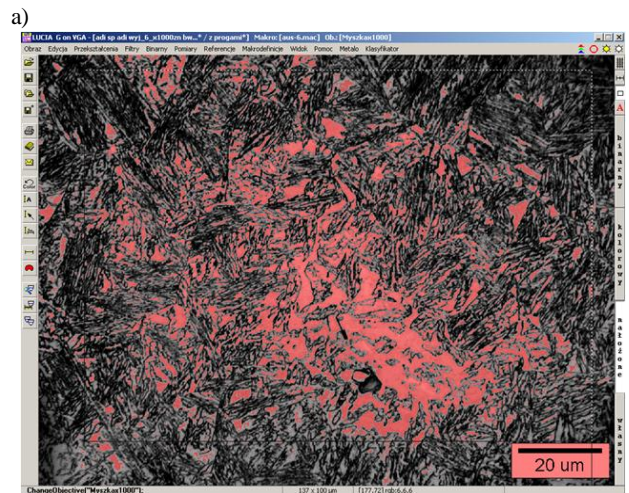
phase, planimetry on X-ray pattern, imp;

- I_γ - total relative intensity of diffraction line of (111) γ phase, planimetry on X-ray pattern, imp;

- R - constant adopted from [3]; for these measurements its value was 0,85.

By means of the above described methods it is possible to determine the quantitative contribution of each phase present in ADI. However, the article focuses only on studies of the austenite.

Another classic method for the measurement of austenite content in ausferritic ductile iron is by analysis of metallographic images [4-8]. Studies of B 370_060 ADI have indicated that, depending on the type of the image used, the results of such measurements can differ quite considerably. Figure x shows three images describing the examined cast iron. The results of these measurements presented in the form of three numerical values in Table 3 show that the evaluation of austenite content (white phase) is not consistent and strongly depends on the choice of location and on the quality of the metallographic image, and therefore is very subjective. An analysis of this type can be successfully implemented in determination of the spheroidal graphite content in cast iron (Fig. 4), due to a strong contrast that graphite offers when observed against the background of a clear matrix.



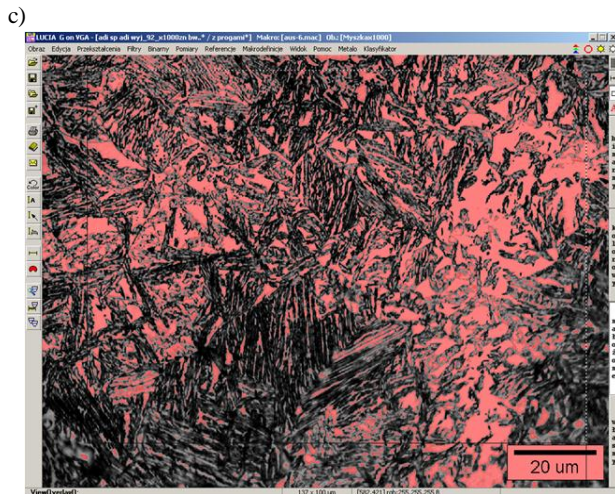


Fig. 4. Metallographic images of the B 370_060 ADI matrix when analysed by LUCIA programme

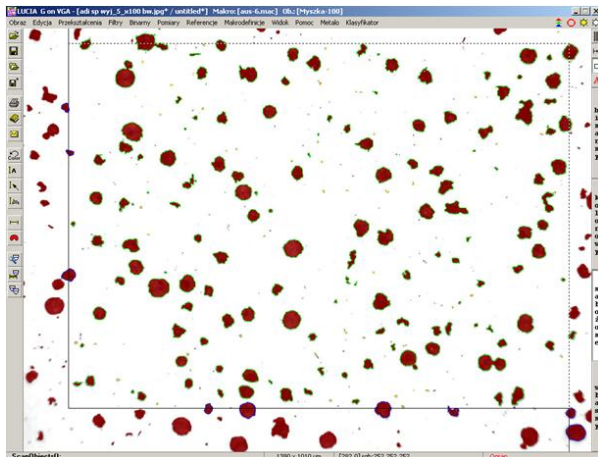


Fig. 5. Photograph of unetched B 370_060 ADI analysed by LUCIA programme

Another method to determine the content of austenite in ausferritic ductile iron is by forecasting using artificial neural networks. The estimation of the austenite content occurs as a result of the operation of a genuine intelligent programme developed at Warsaw University of Technology, based on a large amount of data collected from various experiments carried out on the ADI, processed and stored in a special database. The estimation is of an approximate character and is not the result of a direct measurement. Yet, looking at the values in Table 3, the results of that prediction appear to be similar to the results of an X-ray diffraction analysis and to one of the values obtained by image analysis. Artificial neural network also provides a number of additional options for an analysis of the austenite content in ausferritic ductile iron. One of the most interesting is a wide-range correlation between the austenite content and heat treatment parameters (Fig. 6). For some specific cases, from these charts, one can conclude about trends associated with the presence of this phase in cast iron, and hence about the properties of the cast iron itself.

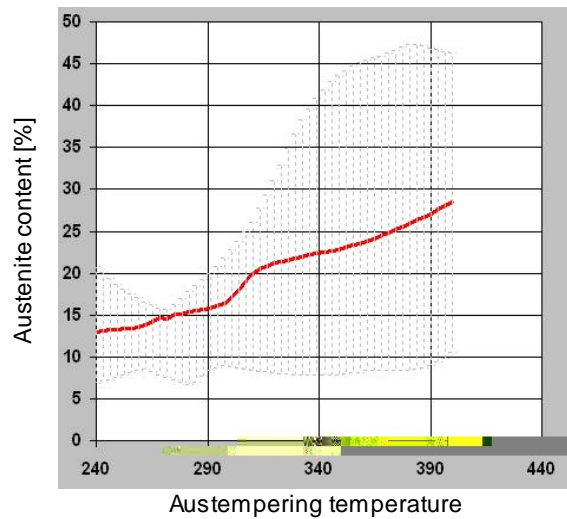


Fig. 6. Austenite content vs temperature of isothermal transformation (excluding the effect of other factors such as temperature and time of austenitising or time of isothermal transformation)

One of the most interesting methods recently considered in the measurements of austenite content in ausferritic ductile iron is a magnetic method. It consists in determination of the content of ferromagnetic phases, which in cast iron can be ferrite and/or martensite. In ADI, one can expect the presence of ferrite as a product of isothermal transformation and hence conclude that the percent indication of ferromagnetic phase will define the content of ferrite. Yet, to transpose this value to the quantitative content of a paramagnetic phase, i.e. austenite, it is necessary to know the percentage of graphite, which is also paramagnetic. The value of this content can be determined by measurements such as e.g. an image analysis, where the determination of graphite content is a basic measurement carried out for cast iron. Therefore, the authors have decided to include in Table x also the austenite content estimated by a magnetic technique. The results of the magnetic method assisted by image analysis stand out from the measurements taken by other methods. However, it is difficult to conclude about the lack of precision because other results are characterised by a large scatter, too.

Table 4 shows other studies carried out on the ductile iron austempered at 400°C, with the time of transformation amounting to 30 minutes. The table comprises only the results of measurements obtained by the methods considered to be the most accurate. From these results it follows that, despite the identical heat treatment conditions, the content of individual phases differs when various methods are used for the measurement. This indicates slightly different type of transformations occurring in unalloyed and alloyed material. Looking at the results disclosed in table x it was found that the quantitative contribution of individual phases was not clearly defined, either, since the obtained individual values could not be summed up.

Table 4.

The results of quantitative measurements of the austenite content in A 400_030 ADI and B 400_030 ADI

Cast iron type	Austenite		Ferrite	Graphite
	Measured by XRD	Predicted by neural network	Magnetic measurement	Image analysis
ADI A 400_030	30,5	25,7	67,0	7,8
ADI B 400_030	33,9	37,0	60,2	7,6

4. Conclusions

Summing up the results of the tests carried out on the ausferritic ductile iron it can be concluded that the quantitative measurement of austenite content is always a rough estimate and as such is reliable to a very limited extent only. It has been indicated that other than the classic methods of the austenite content measurement, i.e. prediction by an artificial neural network and magnetic technique, are also applicable in an approximate determination of its content. The use of different methods to measure the austenite content and a large number of the results of these measurements enable its volume fraction to be determined with great probability.

The studies of austenite presented in this article do not allow for the results of the measurements of metastable austenite, which is also present in ausferritic ductile iron. It can be formed during preparation of samples for measurements, e.g. during grinding. This is an extremely important aspect, which should always be taken into account during austenite measurements. The presence of metastable austenite, and especially its transformation into martensite, which is a ferromagnetic phase, may have a significant impact on both the measurement methods, i.e. by X-radiation and magnetic force.

References

- [1] PN-EN 1564, „Founding – Austempered Ductile Cast Iron”, 2000; (in Polish).
- [2] www.ductile.org
- [3] D. Senczyk, The Laboratory from X-Ray Structure Analysis. Pub. School Engineering, Poznań (1974) (in Polish).
- [4] A. Kowalski, M. Kuder, Use of Computer Programme to the Analysis of Painting LUCIA to the Quantitative Opinion of the Structure of Cast Iron ADI. Innovations in Founding, Part I, Cracow (2007) 281-290 (in Polish).
- [5] T. Giętka, S. Dymski, The attempt at evaluation of the ADI microstructure with the use of the image in cast iron. Archives of Foundry Engineering, vol. 10, Issue 2, (2010) s.57-62.
- [6] K. Miernik, R. Bogucki, A. Gądek, W. Dziadur, The Influence of Conditions of the Thermal Processing on Content of the residual austenite in Ductile Cast Iron of Isothermal Transformation (ADI), Conference Materials XXX the School Engineering Material, Kraków-Ustroń-Jaszowiec (2002) (in Polish).
- [7] L. Wojnar, K. Kurzydowski, J. Szala, The Practician of the Analysis of the Painting. Publishing house Polish Company Stereology, Cracow (2002) (in Polish).
- [8] Ł. Rauch, J. Kusiak, Automatic analysis of microstructure photographs. Scientific Papers, Warsaw University of Technology (2007), no. 216, s. 165–170.
- [9] J. Aranzabal, I. Gutierrez, J.M. Rodriguez-Ibabe, J.J. Urcola, Influence of the amount and morphology of retained austenite on the mechanical properties of an austempered ductile iron. Metallurgical and materials transactions A, vol. 28A, (1997), s. 1143 – 1156.