Influence of cryogenic treatment on microstructure and hardness of austempered ductile iron

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*Abstract: The article presents the properties and microstructure of austempered ductile iron as well as the process of obtaining it. It also shows investigations regarding the additional process of cryogenic treatment * aimed at transformation of metastable austenite into martensite. The fundamental feature of ADI is the presence of a substantial amount of austenite in its microstructure. This austenite, when present in certain quantities, is not stable and is subject to transformation into martensite during cold working or cryogenic treatment. Investigations reveal that when subjected to basic observation with the aid of a light microscope the microstructure of ADI does not differ substantially regardless of whether it underwent cryogenic treatment or not. However, hardness testing, observations with the aid of a scanning electron microscope, as well as measurement of the content of martensite by the DM test method indicate that certain expected changes have taken place in the microstructure.*

Key-Words: austempered ductile iron, cryogenic treatment, austenite, martensitic transformation

1 Introduction

The term: Austempered Ductile Iron (ADI) is not yet well known to all metallurgical engineers, and perhaps it is because of this fact that constantly new ideas keep on coming up, aiming at improvement of its already excellent properties. This is especially valuable in terms of the application of this material. Suffice it to say, in 2003 global production of ADI reached 220,000 tons [1], and by the year 2010 it may have reached 300000 tons [2]. These figures alone speak of the gigantic interest by purchasers of castings made from ADI which are used for manufacturing of machine components and whole systems in industrial branches such as automotive, agricultural, defence, etc. ADI, however, still hides several secrets which continue to intrigue scientists worldwide. The present article may be helpful in solving at least one of them.

2 Problem Formulation

Austempered ductile iron is a material classified according to the European Standard [3] which describes a very broad range of its properties. The most representative of these are the commonly quoted good ductility, exceeding 10%, with tensile strength higher than 900 MPa or very high tensile strength, recorded at 1600 MPa for a different grade of ADI. Austempered ductile iron, however, features several other properties which predestine it for application to diverse structural components. The following are its best known properties: capability of vibration damping, 10% lower density in comparison with steel, good machinability prior to heat treatment, propensity for strengthening of casting surfaces during burnishing or shot peening, etc.

The reason why austempered ductile iron exhibits such good properties is the appropriately carried out heat treatment cycle, comprising austenitization and quenching with isothermal transformation (Fig. 1). Both these processes are extremely important from the point of view of microstructural transformations and, by the same token, properties of ADI. Austenitization determines the content of carbon in austenite and its homogenization, while the isothermal transformation, taking place after quenching from the austenitizing temperature, determines the morphology of the mixture of ferrite and austenite (ausferrite), observed at ambient temperature and finally responsible for the properties of ADI (Fig. 2). Such a description of heat treatment of ductile iron seems to be very simple to carry out and leads to obtaining proper effects. As investigations reveal, it is not always possible to fulfill minimum criteria described by the specification (Fig. 3).

Control of the heat treatment process, as follows from several investigations, boils down mainly to determining the appropriate amount of austenite in the matrix of the required grade of austempered ductile iron [4]. In the light of new investigations carried out by the authors of this article, the above fact may be restated in a manner that is more precise. Why this need? The first reason for this is the question about the non-uniformity of mechanical properties of ADI which reveals itself even with a constant amount of austenite in the ausferritic matrix. The second reason may be the lack of unequivocal justification of non-uniformity of austenite surrounded by platelets of ferrite at ambient temperature. Such austenite will occur in at least three forms: stable supercooled austenite (approx. >1.6% C), metastable supercooled austenite (approx. 1.0 ÷ 1.6% C), as well as untransformed austenite (approx. <1.0% C) enriched in additional alloys: Mo, Mn. Each one of these austenite will feature a different characteristic and its proportion will be able to determine the final properties of the entire casting.

It seems that the most desired type of austenite in ADI will be that which is thermally and mechanically stable at ambient temperature [4]. However, undoubtedly more interesting from the point of view of obtaining the best properties from ADI is the metastable austenite, especially that in which the TRIP (Transformation Induced Plasticity) effect occurs. Such austenite, as the result of the effect of stresses, transforms into deformational austenite, the appearance of which in certain steels enables the obtaining of yield strength up to 2200 MPa with 20÷25% relative elongation [5].

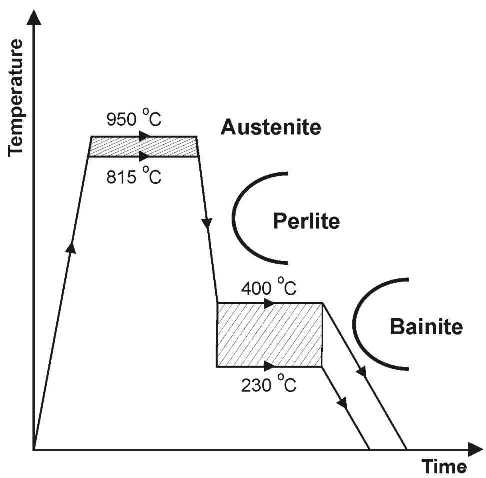


Fig.1. Schematic of heat treatment of austempered ductile iron

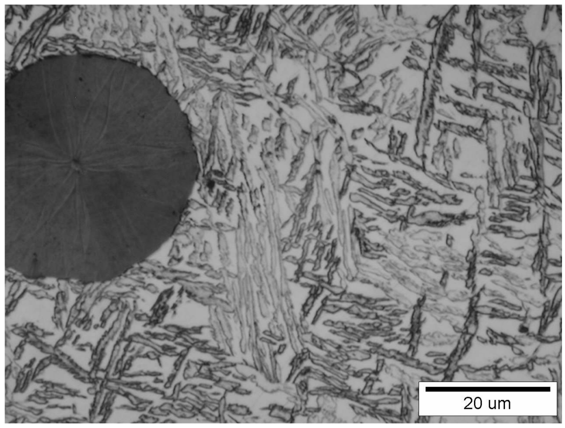


Fig.2. Microstructure of ADI.

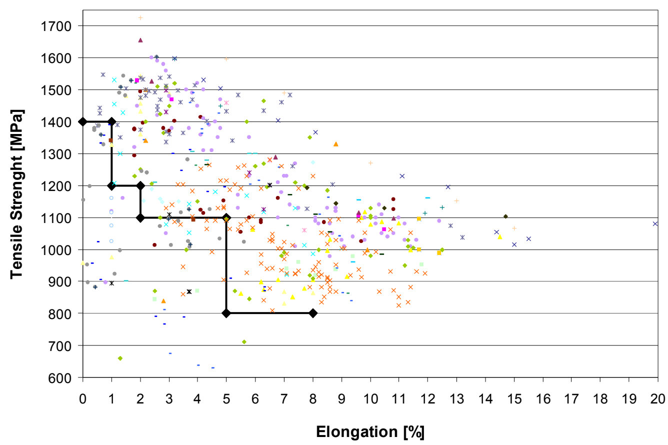


Fig. 3. Plot illustrating the dependence of tensile strength on elongation obtained empirically (points on the plot) and their comparison with minimal values of the European Standard (lines on the plot) [6].

Metastable austenite may have another interesting feature. It will transform to martensite as the result of cryogenic treatment. The employment of this type of treatment may result, besides changes in the properties of ADI, in revealing, by an indirect method, the amount of metastable austenite which underwent transformation to martensite.

Currently, the Institute of Precision Mechanics is conducting investigations aiming at the enhancement of service properties of ADI, as well as those of other structural materials. One of the ways of doing this is the employment of cryogenic treatment.

Investigations presented in this article were aimed at revealing the microstructure and carrying out hardness measurements of specimens before and after cryogenic treatment of ADI for various heat treatment parameters.

An attempt was also undertaken to find a correlation between the results of microstructural investigations and hardness measurements with transformations taking place in the ausferritic matrix following cryogenic treatment.

3 Experimental Procedure

Investigations were carried out on alloy cast iron, smelted in a medium frequency induction furnace of 120 kg capacity and modified by FeSi75 of 3-7 mm granulation, added in an amount of 1% relative to the mass of the furnace charge. As a result of that, nodular cast iron was obtained, with a chemical composition presented in Table 1, in the form of YII castings with a 25 mm base.

Table 1. Chemical composition of ductile iron [wt.%]

C	Si	Mn	P	S	Ni	Cu	Mo	Mg
3.64	2.25	0.18	0.035	0.01	1.55	0.8	0.18	0.045

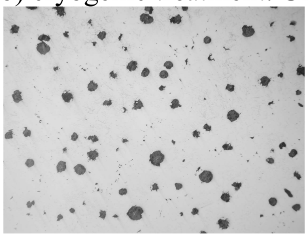
Specimens were machined out of the measurement portion of the castings and had a shape of rods of 50 mm length and cross-section of 10 x 10 mm. The specimens were austenitized at a temperature of 900°C for 2 hours, and next quenched isothermally in a fluidized bed at temperatures of 260, 300 and 360°C for 60 and 120 min. After the completion of the heat treatment process, the specimens were subjected to cryogenic treatment in liquid nitrogen for a time of 30 min.

All specimens of ADI before and after cryogenic treatment were subjected to microstructure observations with the aid of an OLYMPUS IX70 light metallurgical microscope and a HITACHI S-3500N scanning electron microscope. The specimens were mounted in thermosetting phenolic resin by compacting at elevated temperature. They were subsequently ground on emery papers from #240 to #2400 and next polished on special polishing wheels with a 3 μm diamond suspension and an aluminium oxide suspension of 0.1μm size. The specimens were etched in a 3% solution of nitric acid in ethyl alcohol. On remaining portions of the specimens HB hardness measurements were made employing a versatile Rockwell-Brinell hardness tester, while the content of martensite, transformed as a result of cryogenic treatment, was measured by a novel method called the DM-test [12].

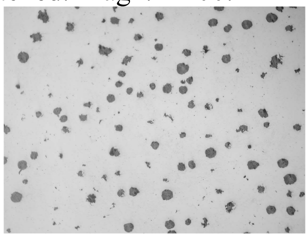
4 Results and Discussion

Metallographic investigations carried out with the aid of the light microscope, presented in Tables 2, 3 and 4, did not reveal any significant differences between the microstructure of ADI before and after cryogenic treatment. It is also difficult to evaluate micrographs in terms of contents of the particular phases due to varying intensity of their etching response and subjective character of such observations. Nevertheless it is possible to state that visible changes had taken place in the microstructure of ADI following cryogenic treatment, proving occurrence of transformations in the morphology of graphite and ferrite.

Table 2. Austempered ductile iron before (a) and after (b) cryogenic treatment. Unetched. Magn. x 100.

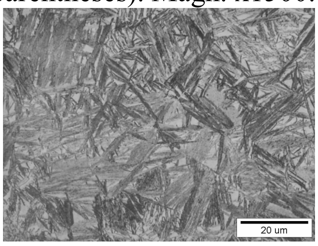


a)

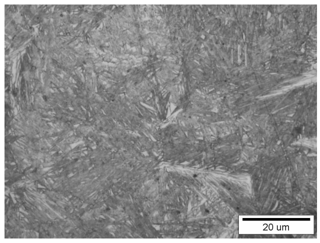


b)

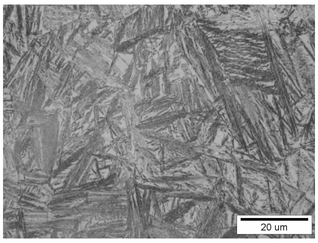
Table 3. Microstructure of the matrix of ADI before cryogenic treatment (heat treatment parameters – temperature and time of isothermal quenching - given in parentheses). Magn. x1500.



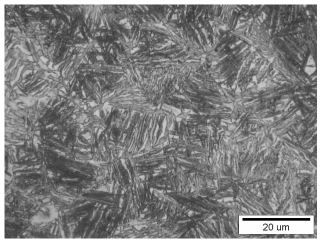
260°C/60 min.



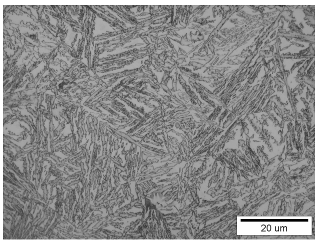
260°C /120min.



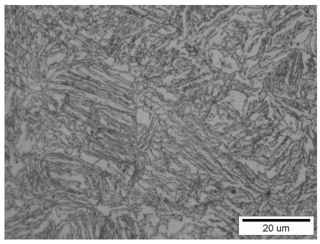
300°C /60min.



300°C /120min.

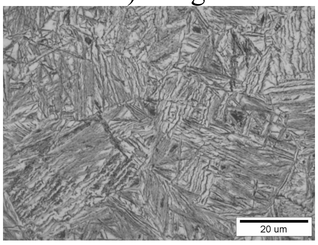


360°C /60min.

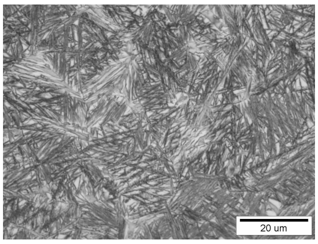


360°C /120min.

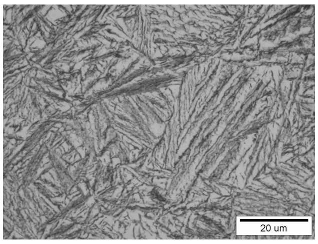
Table 4. Microstructure of the matrix of ADI after cryogenic treatment (heat treatment parameters – temperature and time of isothermal quenching - given in parentheses). Magn. x1500.



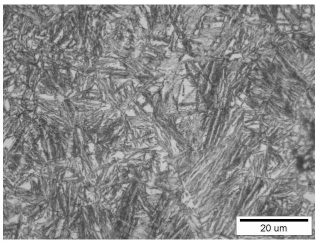
260°C /60min.



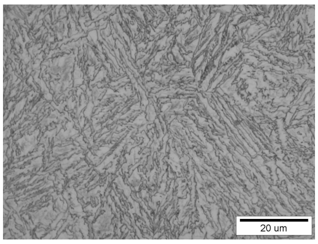
260°C /120min.



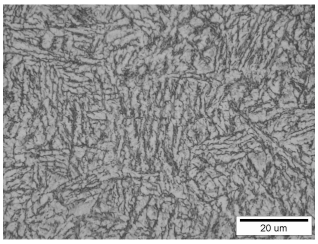
300°C /60min.



300°C /120min.



360°C /60min.



360°C/120min.

In-depth observations may, however, suggest a conclusion that austenite transforms into martensite in special microregions. Such localities are most clearly visible between close-neighbouring precipitations of nodular graphite (Fig. 4) in ductile iron austempered at 260°C. On the other hand, in ADI austempered at 360°C and subsequently cryogenically treated, darker fields are seen when viewed in the Nomarski differential interference contrast mode. They may point to the transformation of certain microregions between ferrite platelets (Fig. 5).

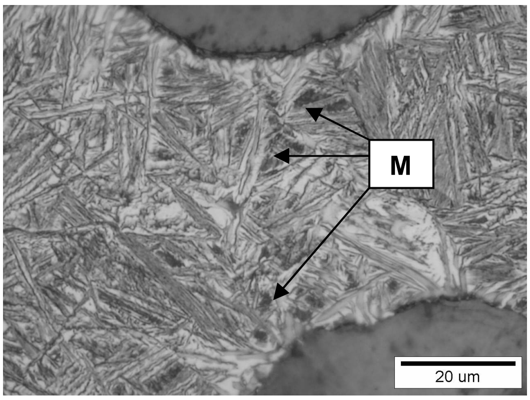


Fig.4. Miecrostructure of matrix of ADI between graphite nodules. M – martensite. Magn. x1500.

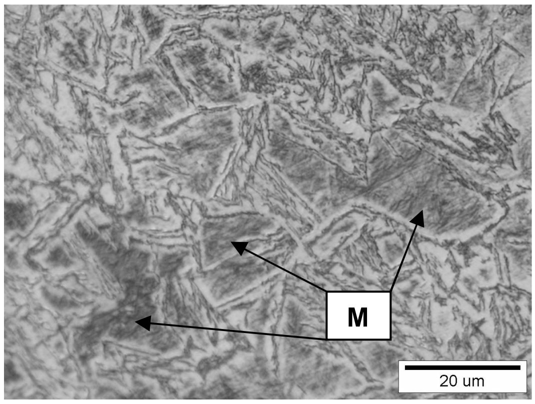


Fig. 5. Martensite (M) in matrix of ADI. Magn. x1500.

Interesting observations regarding the darker microregions on metallurgical mounted specimens may be made thanks to electron microscopy. On specimens of ADI austempered at 360°C and subsequently subjected to cryogenic treatment one can observe fields of austenite partially transformed to martensite, which is not seen in the ADI matrix that is not cryogenically treated (Figs. 6 and 7). That the observed phase is martensite can be inferred from the morphology of lens-like platelets arranged in characteristic zigzags (Fig. 7) [5] as well as from earlier investigations [11]. Noticeable also is the absence of transformation of austenite into martensite in regions between the ferrite platelets. Such a character of phenomena suggests that in these regions occurs saturation with carbon sufficient to lower the M_S temperature of austenite to below -196°C. Subjected to martensitic transformation, stemming from cryogenic treatment in liquid nitrogen are bigger fields of austenite, where carbon saturation is smaller.

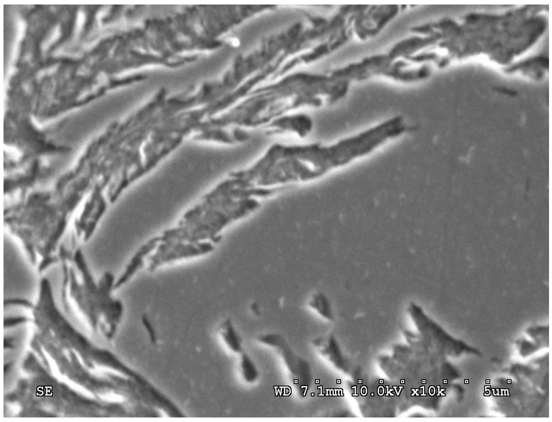
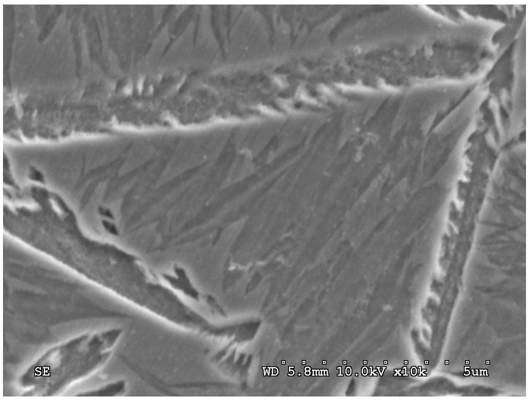


Fig.6. Microstructure of ADI matrix after isothermal quench at 360˚C. Magn. x10000.



Rys.7. Microstructure of ADI matrix after isothermal quench at 360˚C and cryogenic treatment in liquid nitrogen. Magn. x10000.

A more intense character of martensitic transformation in ADI obtained at a higher temperature of isothermal quenching was also determined by the DM-test method. The results of these measurements are presented in Fig. 8. From their analysis it follows that at the isothermal quench temperatures of 260 and 300°C for all specimens a small amount martensite (≤1%) is obtained following cryogenic treatment. On the other hand, when ADI is austempered at 360°C the amount of this phase is clearly greater.

There is also a difference in the amount of martensite measured on specimens of austempered ductile iron that was isothermally quenched in times of 60 and 120 minutes. In the case of 60 minutes of isothermal transformation, the martensite content is generally greater than in the case of 120 minutes. This is consistent with the theory of thermal stabilization of austenite by carbon during isothermal transformation, which causes the gradual lowering of the M_S temperature of austenite.

Based on this premise, a conclusion may be drawn that the extension of the time of isothermal transformation from 60 to 120 minutes for ADI at 360°C causes thermal stabilization of approx. 2% of austenite.

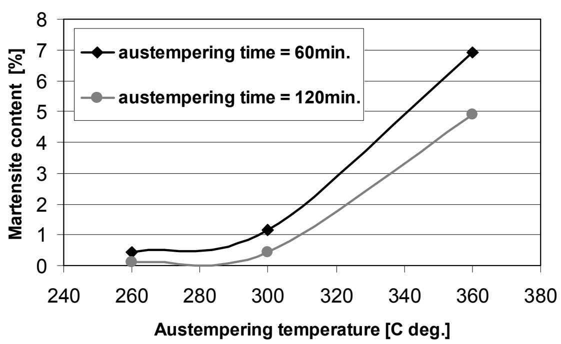
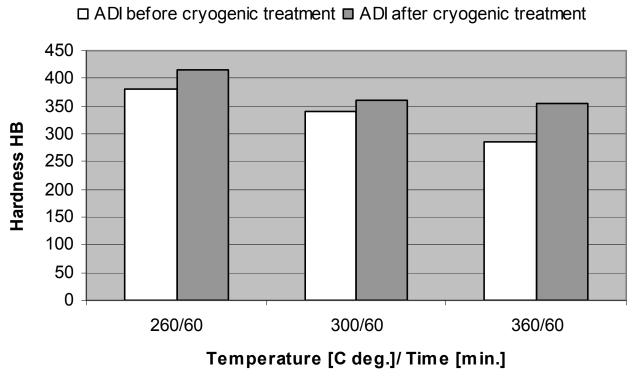


Fig. 8. Dependence of athermic austenite content after cryogenic treatment on the temperature of austempering.

Hardness measurements presented in Figs. 9 a and b also confirm the presence of martensite in the microstructure of ADI following cryogenic treatment. This is indicated by a rise in the hardness of all specimens subjected to cryogenic treatment. The presented results also show smaller differences in the hardness of ADI following longer times of isothermal transformation. This may be caused by the appearance of a minimum amount of martensite after austempering at temperatures of 260 and 300°C, as well as a greater amount of stable supercooled austenite in the matrix of ductile iron austempered at the temperature of 360°C following 120 minutes of transformation.

a)



b)

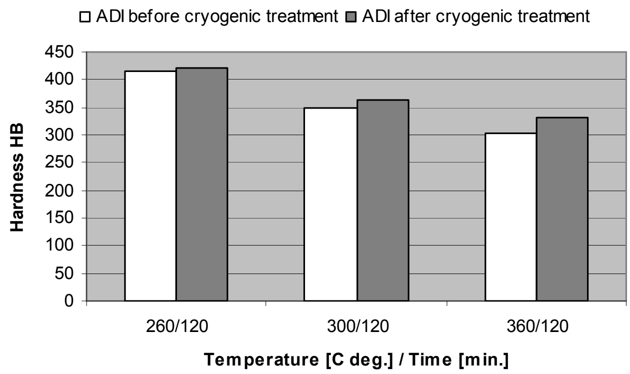


Fig. 9a,b. Results of hardness measurements of specimens that were cryogenically treated and those without that treatment for different austempering temperatures.

5 Conclusions

1. Austempered ductile iron is a structural material on the rise bearing a high potential for application in the automotive, railway, agricultural, defence and other branches of industry its full range of properties and applications remains still to be discovered.

2. What occurs in austempered ductile iron is the TRIP (Transformation Induced Plasticity) effect, as a result of which there comes about a transformation of metastable

austenite into deformational martensite, thus causing both positive and negative changes of properties of this material.

3. The identification of metastable austenite, as that of its proportion in the microstructure of austempered ductile iron may bring about a more precise control of properties by way of determining its positive and negative influence.

4. Cryogenic treatment of ADI in liquid nitrogen enables the transformation of thermally metastable austenite into martensite and, indirectly, the determination of its content.

5. Cryogenic treatment of ADI increases its hardness which varies depending on the amount of martensite created by this process.

References:

[1] Roedter H.: “ADI – Austempered Ductile Iron”, *Biuletyn Metals Minerals* 2004, nr 2;

[2] Hayrynen K.L., Keough J.R.: “Austempered Ductile Iron – The State of the Industry in 2003”, *Keith Millis Symposium on Ductile Cast Iron 2003*, Livonia, Michigan, USA;

[3] PN-EN1564, „Żeliwo sferoidalne hartowane z przemianą izotermiczną”, 2000;

[4] www.ductile.org

[5] Jeleńkowski J.: „Kształtowanie struktury stali Fe-26Ni-2Ti z wykorzystaniem przemiany martenzytycznej”, *Oficyna Wydawnicza PW*, Warszawa, 2005;

[6] Biernacki R, Kozłowski J., Myszka D., Perzyk M.: „Prediction of properties of ADI assisted by artificial neural network”, *Materials Science (Medziagotyra)*, 2006, vol.12, nr1, p.11-15;

[7] Zanardi F.: “The development of machinable ADI in Italy”, *Int. Scientific Conf. “Ductile Iron in 21st Century*”, Kraków, 2003;

[8] Santos H., Pinto A., Torres V.: “Żeliwo sferoidalne ADI z dodatkiem Cu i Mn”, *Przegląd Odlewnictwa*, 1992, nr3, p.113-119

[9] Dymski St.: „Kształtowanie struktury i właściwości mechanicznych żeliwa sferoidalnego podczas izotermicznej przemiany bainitycznej”, *Wyd. Uczelniane ATR*, Bydgoszcz, 1999;

[10] Garin J.L., Mannheim R.L.: „Strain-induced martensite In ADI alloys”, *Journal of Materials Processing Technology*, 2003, 143-144, p.347-351;

[11] Myszka D., Borowski T.: “Wymrażanie żeliwa sferoidalnego ausferrytycznego”, *Inżynieria Materiałowa*, nr3/2006, p.221-224;

[12] Myszka D.: Sposób pomiaru martenzytu w żeliwie sferoidalnym ausferrytycznym, *Zakład Odlewnictwa Politechniki Warszawskiej*, 2006.