The attempt at evaluation of the ADI microstructure with the use of the image analysis

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Abstract

In this work the attempt at quantitative evaluation of the microstructure of ADI cast iron matrix has been made. The automatic analysis of the image of the microstructure of austempered spheroidal cast iron has been used for the evaluation. The treatment variant was a two-phase austenitization. First phase was carried out at a temperature \( t_1 = 950^\circ C \) and after cooling to a temperature \( t_2 = 900^\circ C \). The isothermal process was carried out at a temperature \( t_{pi} = 300 \) and \( 400^\circ C \) for \( 8÷64 \) min. Ordinary cast iron was austempered. After it the microstructure images were recorded and subjected to automatic image analysis with the use of the NIS ELEMENTS 3,0 AR software.

Keywords: Image Analysis; Isothermal Process; Diffraction; ADI

1. Introduction

In the technology of high strength ADI cast iron grades the microstructure of matrix, consisting of austenite and ferrite [1÷3] is significant.

In ADI iron cast production the austempering is used in order to produce high carbon austenite and rich in carbon ferrite in the matrix. Such composition of microstructure is called ausferrite and the isothermal process of over-cooled austenite – ausferritzing.

Research works over ADI cast iron concentrate mostly on ausferritzing process [3, 8, 9÷13]. It is a rational approach, because shaping of strength, ductility and toughness as well as impact strength of the cast iron result mostly from the parameters of heat treatment. It is significant on account of the practical cast application in accountable machine parts.

Image analysis is considered to be significant in many fields of science connected to quantitative analysis of microstructure. Application of algorithms of automatic image analysis consists in processing the images of material microstructures in order to calculate statistic characteristics of the size and shape of the grains.

The microstructure which has been analysed in this way may be used to prepare a model of the material structure for simulation calculations with the use of Finite Element Method (FEM). In order to make the calculations reliable, the complex analysis of the input data basing on extraction of characteristic features like edges, inclusions and noise has to be carried out.

The microstructure image analysis is in many cases complex on account of the variety of shapes and sizes of grains, which in effect has an influence on the reliability of the results and the later calculations [4, 6÷9]. The majority of accessible algorithms serv-
ing to edges detection cannot cope with the problem of occurring
noise, treating it as a typical image element.

The aim of this work was to carry out a test of the ADI cast
iron components qualitative evaluation with the use of automatic
image analysis. The results of the quantitative microscopy have
been compared to the X-ray diffraction analysis.

2. Material, programme and research
methods

The researched cast iron has been smelted in a hot blast cupo-
la furnace. Balling of the cast iron was carried out with the use of
magnesium alloy ML5, applying rod method in the container of
the cupola. Cast iron has been cast to wet sand moulds. The casts
had a shape of ingots VII according to (PN-EN 1563:2000). On
the base of static tensile test the cast iron was qualified to the
grade EN-GJS-500-7. Cast iron had ferritic-pearlitic (10%) ma-
trix structure and proper nodular graphite form (share 11.5%, 112
rel./mm² microsection surface).

Chemical composition and properties in the initial state of the
ordinary cast iron are presented in the Table 1.

Table 1. The chemical composition and mechanical properties
in ductile iron

<table>
<thead>
<tr>
<th>Chemical element, % mas.</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>3.65</td>
<td>2.59</td>
<td>0.18</td>
<td>0.052</td>
<td>0.014</td>
<td>0.06</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Mechanical properties</th>
<th>R&lt;sub&gt;mp&lt;/sub&gt;</th>
<th>A&lt;sub&gt;5&lt;/sub&gt;</th>
<th>H&lt;sub&gt;HV10&lt;/sub&gt;</th>
<th>KCG&lt;sub&gt;x&lt;/sub&gt;</th>
<th>J/cm²</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>507</td>
<td>12.1</td>
<td>156</td>
<td>106</td>
<td></td>
</tr>
</tbody>
</table>

Before hardening the lower part of ingot VII were treated with
two-phase ferritizing annealing. The first phase at the temperature
of 1050°C for 4 hours and the second one after cooling to 680°C
held for 6 hours. Cooling down of the cut ingots was carried out
with the use of a furnace. After that the ingots were cut into three
flat bars. The flat bars were marked with the retaining of their
previous location in the ingots and the tensile test samples, whose
dimensions are depicted in the Figure 1, were made.

![Fig. 1. Dimensions of the tensile test samples](image)

Tensile test samples have been austempered according to the
schemes shown by the Figure 2. For every treatment condition a
set consisting of 3 samples, originating from 1 ingot YII, has been
prepared.

From the gripping part of the samples after heat treatment the
11-millimetre-long specimens were taken, on which metallo-
graphic microsections were made. They served for the assessment
of their microstructure under an optic microscope. The record of
the microstructure images has been made with the use of scanning
electron microscope (SEM) type JSM-50A made by company
JEOL with the acceleration voltage 20 kV and absorptive current
of the sample ca. 0.2·10⁻¹⁰ A. The images have been registered on
the enlargement scale 1000x3000x.

![Fig. 2. Scheme depicting austempering of the ductile cast iron](image)

Automatic image analysis has been made with the use of NIS
ELEMENTS 3.0 AR software. Its task was to calculate specially
marked pixels of the image which was calibrated according to the
enlargement. Halftone screen image received from the scanning
microscope has been converted to the binary image, which explicit-
lly determined the evaluated microstructure components. With
the use of computer software the share of austenite and sizes of
the ferrite needles have been determined.

On the metallographic specimens also the X-ray diffraction
analysis has been carried out. The research has been made on the
diffractometer, using CoKα radiation and iron filtering. The follow-
ing parameters of diffractometer's work have been assumed:
- lamp voltage 36 kV;
- lamp current 6 mA;
- the arrangement of collimating slits I – 2, II – 2, III – 2
  mm, the Soller slits on the primary beam and on the dif-
  fracted beam;
- scintillation counter detector operated at 700 V.

Diffraction patterns have been recorded within the angular
range 2θ 49,5°-53,5°. Within this angular range, using step 0,01°,
diffraction lines (110) phases α and (111) and phases γ have been
recorded. Intensity of each interference line has been determined
three times by planimetric of the field under the curve to the
background level. Share of austenite (V<sub>y</sub>) from the formula 1 [17]:

![Graph](image)
\[
V_\gamma = \frac{1}{1 + \alpha \cdot I_\gamma \cdot R} \cdot 100 \%
\]

(1)

where:
\( V_\gamma \) - share of austenite in the volume fraction,
\( I_\alpha \) - relative intensity of diffraction line (110) phase \( a \), planimetred on the X-ray photograph,
\( I_\gamma \) - relative intensity of diffraction line (111) phase \( \gamma \), planimetred on the X-ray photograph,
\( R \) - value of the constant accepted to these measurements 0,85.

Averaging size of ferrite needles has been determined from the formula 2 [18]:
\[
d = \frac{0.9 \cdot \lambda}{B \cdot \cos \Theta}
\]

(2)

where:
\( d \) - average diameter of the crystal particle,
\( \lambda \) - wavelength in nm,
\( B \) - diffraction line broadening, measured in the middle of its maximal intensity in rad,
\( \Theta \) - the angle of the interference line determined from the diffraction pattern in °.

3. The results of the research and their analysis

The quantitative evaluation of the components of the austempered cast matrix microstructure has been carried out on 5 random chosen images, registered for each specimen which has been enlarged 2000x. The demonstration microstructures of the cast iron have been depicted in the Figures 3-6.

Fig. 3. The microstructure of the cast iron austempered at a temperature \( t_{\text{pi}} = 300^\circ\text{C}, \tau_{\text{pi}} = 8 \) min. Etched in nital, micr. surf. 2000x

Fig. 4. The microstructure of the cast iron austempered at a temperature \( t_{\text{pi}} = 400^\circ\text{C}, \tau_{\text{pi}} = 8 \) min. Etched in nital, micr. surf. 2000x

Fig. 5. Binary image of the cast iron microstructure austempered at a temperature \( t_{\text{pi}} = 300^\circ\text{C}, \tau_{\text{pi}} = 64 \) min. Etched in nital, micr. surf. 2000x

Fig. 6. Binary image of the cast iron microstructure austempered at a temperature \( t_{\text{pi}} = 400^\circ\text{C}, \tau_{\text{pi}} = 8 \) min. Etched in nital, micr. surf. 2000x
After austempering at temperature $t_{pi} = 300^\circ$C the matrix consisted of the lower ausferrite (Fig. 3, 5). While at a temperature $t_{pi} = 400^\circ$C upper ausferrite (Fig. 4, 6). The morphology of the components of ADI cast iron microstructure has been changing in the holding time function. For comparison the microstructure registered on (SEM) for the specimen austempered at a temperature $t_{pi} = 300^\circ$C for 8 minutes (Fig. 3) as well as the binary image of the specimen austempered at the same temperature for 64 minutes (Fig. 5). With the elongation of the conversion time the ferrite needles have undergone the length change and the austenite plates have been evenly distributed. The conversion time affects the microstructure arrangement.

![Image Analysis vs X-ray Diffraction](image.png)

**Fig. 7.** Impact of the temperature and austempering time on the austenite share ($V_\gamma$) in the ADI cast iron matrix

On the base of image analysis the share of austenite in the microstructure has been determined. The microstructure of the ADI cast iron austempered at a temperature $t_{pi} = 400^\circ$C contained austenite in the range $32,1\pm26,4%$ while at a temperature $t_{pi} = 300^\circ$C - $15,6\pm11,0%$. The share of phase $\gamma$ determined by diffraction was bigger for both temperatures (apart from $\tau = 16$ min for $t_{pi} = 400^\circ$C). The character of the chart lines was the same for both research methods. The growth of conversion time caused a decrease of the austenite share. The differences of the received results depicting the austenite share in the cast iron matrix were bigger for the lower temperature of austempering. It results from finer microstructure of the matrix.

The diameter of the ferrite needles (Fig. 8) measured by image analysis is almost ten times, and with the use of diffraction thirteen times smaller at $300^\circ$C than at $400^\circ$C. The little transverse size of the ferrite needles in lower ausferrite has contributed to registering low contrasted images. The result of it has been depicted in the Figure 8 between the diameters at – values determined from the image analysis and the diameters at – values received from the rtg diffraction for $t_{pi} = 300^\circ$C. Bigger diameters at – sizes received from the image analysis result from binary images which have been enlarged by noise and shadows. The change of the transverse sizes of the ferrite needles is connected to the conversion $\gamma \rightarrow \alpha$ mechanism.

The length of the ferrite needles in ADI cast iron under the influence of time decreased (Fig. 9). This relation is stronger in cast iron which has been hardened from the temperature $t_{pi} = 300^\circ$C than at – $400^\circ$C.
Fig. 8. Impact of the temperature and isothermal process time on the average diameter of the ferrite needles (d) in ADI cast iron matrix

![Diameter vs Time](chart1.png)

Fig. 9. Impact of the temperature and isothermal process time on the length of the ferrite needles determined with the use image analysis software

![Length vs Time](chart2.png)
4. Conclusion

On the base of the research carried out and its results the following statements can be made:
1. The image analysis software NIS ELEMENTS 3.0 AR can be used to the quantitative evaluation of the components of ADI cast iron matrix microstructure.
2. From the comparison of methods to the evaluation of the percentage share of austenite in ADI it results that the computer image analysis can be used in the upper ausferrite examination. However, in the range of the lower ausferrite the measurement discrepancy between the methods rises to over 2%.
3. The evaluation of the sizes (diameters and length) of the ferrite needles in ausferrite has been treated as a pre-operation to the next research, although during the analysis of the received results the meaningful impact of the temperature and austempering time on these sizes has been noticed.

References
