The changes of ADI structure during high temperature annealing

A. Krzyńska a) M. Kaczorowski b)

a) Institute of Materials Processing b) Institute of Mechanics and Design,
Faculty of Production Engineering, Warsaw University of Technology,
ul. Narbutta 85, 02-524 Warszawa, POLAND

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Summary

The results of structure investigations of ADI during it was annealing at elevated temperature are presented. Ductile iron austempered at temperature 325°C was then isothermally annealed 360 minutes at temperature 400, 450, 500 and 550°C. The structure investigations showed that annealing at these temperatures caused substantial structure changes and thus essential hardness decrease, which is most useful property of ADI from point of view its practical application. Degradation advance of the structure depends mainly on annealing temperature, less on the time of the heat treatment. It was concluded that high temperature annealing caused precipitation of Fe₃C type carbides, which morphology and distribution depend on temperature. In case of 400°C annealing the carbides precipitates inside bainitic ferrite laths in specific crystallographic planes and partly at the grain boundaries. The annealing at the temperature 550°C caused disappearing of characteristic for ADI needle or lath – like morphology, which is replaced with equiaxed grains. In this case Fe₃C carbides take the form very fine precipitates with spheroidal geometry.

Key words: ADI, structure, mechanical properties

1. Introduction

Since isothermal quenching of ductile iron was discovered, leading to new grade of cast iron known generally at the name ADI [1,2], many attempts was undertaken to even more increase very attractive mechanical properties of this casting material. This exceptional mechanical properties follow from specific metallic matrix microstructure, which is the mixture of carbon stabilized austenite and bainitic ferrite. If negligible the nodular shape of graphite precipitates, ADI ductility depends mainly on proportion of austenite with FCC crystallographic lattice which relative content can reach even 40%.

On the other hand the microstructure of high strength and low ductility ADI produced by low-temperature austempering consists mainly of bainitic ferrite and some amount of martensite. Among many attractive properties of ADI the wear resistance looks to be most useful. In case of ADI wear resistance follows from very high hardness, which in turn results probably from hardening of matrix components. It looks that at least three mechanisms are responsible for high ADI hardness. The first is solid solution strengthening either ferrite or austenite which both is supersaturated with carbon atoms. The second are probably the residual stresses caused by rapid quenching from austenitizing to austempering temperature. The third one can be strengthening with phase boundaries. This mechanism is also very probable because dispersion of the microstructure which is the mixture of very fine needles or laths ferrite and austenite. The highly dispersed microstructure leads to many obstacles for dislocation movement and so to material strengthening. Finally, martensite mentioned above forming at low temperature austempering, which can not be neglected, because it’s very high influence on ADI strengthening.

The aim of this paper is mainly to show the influence of high temperature annealing on the ADI structure. For this the transmission electron microscopy (TEM) was applied.

2. Experimental procedure

Ductile iron grade EN-GJS-500-7, was chosen for investigations. It was melted in 3 ton induction furnace. Mg
treatment was carried with FeSiMg alloy using tundish-cover method. Liquid metal was then inoculated with FeSi75 put onto liquid metal stream while during pouring into pouring ladle.

From 25mm thickness YII type tests casting specimen were cut. Theses specimens were first austenitized for 90 minutes at temperature 900°C and then isothermally quenched 2h at temperature 275°C. After austempering these were subjected annealing for 360 minutes at temperature: 400, 450, 500 and 550°C. TEM observations were carried out using thin foil technique. First 3mm diameter and 100m long rods were cut from the heat treated specimens. These rods were then sliced into 0.1mm thin discs with load-less IF-07A wire saw. Disc were first dimpled and then subjected to ion-milling using Gatan equipment. This procedure allowed to get thin foils which were observed in transmission electron microscope Philips EM 300 working at accelerate voltage U = 100kV. During TEM observations thin foil were tilted in TEM holder to reach so called diffraction contrast.

3. Results

3.1. Metallography

An example of ADI microstructure austempered 2h at 325°C was show in fig.1. In photo the graphite nodules in the mixture of lath ferrite in austenite are visible. The laths of ferrite very often appear in form of packs – units – with very close crystallographic orientation each other. Although no quantitative evaluation was performed nevertheless it looks that austenite proportion not exceed 10-15%.

3.2. TEM observations

Preparing of thin foils from multiphase materials for TEM is very difficult. The ductile iron or ADI is such experimentally difficult material because two quite different phases which are graphite and metallic matrix. The problem is even more if the specimens are ferromagnetic as is the case of ADI. Tilting of the specimen in TEM causes the interaction between ferromagnetic specimen and electron beam. This interaction leads to escaping of electron beam which position has to be continuously corrected. Because of these difficulties the authors decided to carry out TEM for the ADI specimen annealed at the temperature 400 and 550°C.

3.2.1. TEM observations of starting material

In fig.2 the results of ductile observations after 120 minutes austempering at the temperature 325°C were shown. In the first micrograph (fig.2a) numerous ferrite with many dislocations can be observed.

On the other hand fig.2b shows austenite grain with specific contrast looking as parallel dark and white lines. This contrast can be interpreted or as caused by stacking fault or microtwins [3, 4]. The austenite grain is surrounded by ferrite grains where, similar as in fig.2a, many dislocations lines inside are visible.

Both kind defects, stacking faults and/or microtwins in austenite and dislocations in ferrite grains were caused by thermal stresses caused by rapid cooling from austenitizing temperature.

Fig. 1. The microstructure of ductile iron after 120 minutes austempering at temperature 325°C

Fig. 2. TEM micrographs of ADI after 120 minutes austempering at the temperature 325oC: a – x 15.000 and b – x 37.500
3.1.1. The TEM observation of annealed ADI

The aim of these observations was identification of structure changes proceeding during high temperature annealing. The main goal was to conclude if at all and when the process of aus ferrite decomposition starts at given annealing temperature. To achieve this, besides conventional microscopic structure observations, many selected area diffraction (SAD) patterns were taken. Resolving of them (indexing) is the way to get information needed for identification of precipitates forming when the system tends to the thermodynamic equilibrium.

In fig.3 the results of ADI structure observations after its 6h annealing at the temperature 400°C were shown. Whereas in the first (fig.3a) no precipitates and if at all they are quite weak visible these are very clear observable in fig.3b and c, having mainly spherical (fig.3c) or slightly elongated shapes, distributed at the grain boundaries (fig.3b).

In the last photos the structure of ADI 6h isothermally annealed at the temperature 550°C (fig.4) was shown. The first (fig.4a) shows electron micrograph taken at relative small magnification where the total lack lath or needle-like structure characteristic for ADI is visible. This specific structure was replaced with almost equiaxed grains. Fig.4b illustrates selected area diffraction pattern from the area in fig.4a. In spite of the diffraction spots are distributed in form concentric diffraction rings it can be identified that some preferred crystallographic orientation of grain reflecting <111> zone axis appears. The strongest diffraction spots located close to the 000 central spot represent diffraction from 110 type ferrite planes [5, 6]. More careful inspection of SAD pattern reveal very weak spots located closer to 000 central spot then very strong {110} type diffraction spots from ferrite. The next photo (fig.4c) shows so called dark-field electron micrograph taken using one of such weak diffraction spot. In this dark-field electron micrograph a large number of shiny small areas are visible. This dark - field electron micrograph (fig.4c) is the proof that weak diffraction spots discovered in SAD picture (fig.4b) were caused by small coherent diffracting areas visible in fig.4c. Moreover, fig.4c is explicit confirmation of precipitates formation in ADI annealed 6h at the temperature 550°C.

Fig.3. TEM micrographs of ADI structure after 6h isothermal heating at the temperature 400°C: a - x 17.000, b – x 26.000 and c – x 90.000
4. Summary and conclusions

The results of structure investigations, especially using TEM technique were presented. To complement the information’s obtained by TEM method the hardness measurements of ADI just after austempering and after 6h annealing at the temperature 400, 450, 500 and 550°C were carried out. The results of hardness testing were given in fig.5 and in table 1.

It follows from fig.5 that 6h annealing at different temperature value causes hardness decrease from 370HB at starting point to approximately 340HB, 325HB, 295HB and 240HB for annealing temperature: 400, 450, 500 and 550°C respectively. It means that annealing at temperature 400°C leads to 10% hardness decrease while the annealing at temperature 550°C even up to 35%, with respect ADI hardness after austempering (table 1)

Because of limited space the authors concentrated only on more deep discussion the results of TEM observations, especially those concerning ADI specimens annealed at the temperature 550°C. It should be mentioned that during 6h annealing at this temperature lath morphology of metallic matrix disappears what is visible if the TEM micrographs showed in fig.3a and 4a were compared. In ADI annealed 6h either in temperature 400 and 550°C precede the precipitation processes. However, the precipitates formed during annealing in lower temperature are much lower and locates preferentially in given crystallographic planes (fig.3b), seldom at grain boundaries (fig.3c).

![Fig.4. The ADI structure after 6h isothermal annealing at temperature 550°C: a – TEM micrograph (x 17.000), b – SAD and c – dark-field micrograph (x 90.000)](image)

![Fig.5. Brinell hardness as a function of annealing temperature](image)
Table 1. Hardness of ADI annealed at temperature 400 and 550°C.

<table>
<thead>
<tr>
<th>Starting T  = 400°C</th>
<th>T  = 550°C</th>
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<tr>
<td>370</td>
<td>340.4 ± 5.0</td>
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The most interesting results obtained in this elaboration are diffraction patterns given in fig.6 and 7. In fig.6 SAD the pattern from specimen annealed at temperature 400°C is shown. In fig.6a where the whole diffraction pattern is presented, relative weak diffraction spots forming concentric rings are located to 000 central spot closer then the diffraction spots from ferrite. Fact that weak diffraction spots are located closer than the spots 110 type from ferrite is the immediate proof of phase appearance with the lattice parameter larger then ferrite. The phases which can appear in ADI could be carbides [7]. Specific distribution of these weak spots forming make impossible to index them and identify the kind of carbides. The one more information can be taken off from diffraction pattern if deep analysis of fig. 6 will be carried out.

The result is shown in fig.6b being the magnified part of fig.6a. In this photo two very close located diffraction spots can be identified. One of them located closer to 000 central spot represents diffraction from 111 type plane in austenite with FCC lattice while the second (further) 101 type planes in BCC ferrite. It means that 6h annealing at the temperature 400°C causes precipitation of carbides (fig.6a) but some amount of austenite remain in metallic matrix (fig.6b).

In fig.7 the selected area diffraction pattern from ADI annealed at the temperature 550°C is depicted.

Fig.6. Selected area diffraction pattern (SAD) from the specimen annealed 6h at the temperature 400°C: a – the whole diffraction pattern indexed for ferrite, b – the magnified part of diffraction pattern revealing two very close laying spots (222) type from austenite (γ) and (022) from ferrite (α)

Fig.7. Selected area diffraction pattern (SAD) from the specimen annealed 6h at the temperature 550°C showing the spots from ferrite (α) and iron carbide Fe3C

In this picture, beside strong, arched diffraction spots from ferrite, distributed in the corners of regular hexahedron, much weaker spots located closer to 000 central reflex then the strong ones are visible. Using so called interior standard, whose role play diffraction spots from ferrite, microscope constant 2λL was calculated, where λ is the electron wavelength for acceleration voltage 100kV and L - camera length. Thanks to this precisely calculated microscope constant interplanar distances responsible for the weak diffraction spots were calculated. The values of interplanar distances were collected. The comparison of distances calculated from electron diffraction pattern fit the values for Fe3C given by Andrews and coworkers in [7]. The fact that diffraction spots are distributed in form of rectangular allowed the verification indexing of electron diffraction pattern according with the formula, that the Miller indices h, k and l fulfill the dependence:

\[
h_3 = h_1 + h_2, \\
k_3 = k_1 + k_2, \\
l_3 = l_1 + l_2,
\]

where: h_i, k_i i l_i are the Miller indices for the spots representing diffraction from given crystallographic planes.

The experimental results and their analysis given above have enabled the authors the following conclusions to propose:
1. The 6h isothermal annealing at the temperature 400°C and higher causes the decomposition of ausferrite matrix which is replaced with the mixture ferrite and carbides.

2. In spite of carbides precipitation during 6h annealing at temperature 400°C some amount of austenite remains in metallic matrix.

3. The carbides which precipitate during isothermal high temperature (550°C) annealing are Fe₃C type carbides.

4. Ausferrite decomposition cases substantial ADI hardness decrease which value depends on annealing temperature and change from 10 to 25% of starting value for 400 and 550°C respectively.

References


