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Calorimetric examinations of austempered ductile iron ADI

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Abstract

The study presents the results of calorimetric examinations during heating and cooling of austempered ductile iron ADI after austempering at temperatures of 280, 330 and 380°C. The samples for examinations were taken from cast rods of 20 and 60 mm diameter. Examinations were carried out on a differential scanning calorimeter, type Multi HTC S60. During heating, on a DSC curve one strong exothermic effect has been noted to occur (it does not occur in the case of common-grade cast iron), accompanied by two endothermic effects. The exothermic effect occurs within the range of about 20°C. Depending on the temperature of austempering treatment, its beginning falls to the temperatures from 469 to 490°C. The heat of this effect is proportional to the austenite content in ADI matrix after austempering. The endothermic effects are related with decomposition of pearlite (or bainite) and with phase transformation $\alpha \rightarrow \gamma$ (ferrite as a constituent of ausferritic matrix.

Keywords: Scanning calorimeter, Temperature, Heating, Cooling, Heat of phase transformations, Pearlite, Graphite

1. Introduction

ADI (Austempered Ductile Iron) is a material of extraordinary combination of tensile strength, toughness and abrasion wear resistance as well as fatigue resistance. This combination enables reducing the weight and cost of casting manufacture. These unique properties are the result of formation of a matrix composed of a mixture of austenite and ferrite, produced during the heat treatment commonly known as austempering [1-3]. The mechanical properties of ADI usually depend on the parameters of this treatment, i.e. on the temperature and time of austenitising and on the temperature and time of austempering. By proper monitoring of these parameters, it is possible to control and „forge" in proper way the microstructure of the metallic matrix, and hence to obtain the required complex of mechanical and plastic properties. The ADI mechanical properties depend to a great extent on the chemical composition of base cast iron and on the features of the metallic matrix and graphite morphology (the content and size) [4].

Though the effect of cast iron austempering is well-known, until now the thermal effects that accompany this transformation and result in formation of ausferritic matrix have not been fully explained. These are the effects difficult to investigate. Basing on the results of calorimetric examinations of grey and nodular graphite cast irons, it has been concluded that thermal effects during cooling of samples take place in a sequence inverse to that which is observed during heating; their character also differs: it is endothermic on heating and exothermic on cooling [5, 6]. Therefore, the authors of this study have decided that the knowledge of the thermal effects that occur during heating of ADI will help in understanding the course of transformation during austempering in the temperature range from 280 to 380°C.

2. Materials and methods of investigation

The investigations were made on nodular graphite cast iron containing: 3,75 % C, 2,55% Si, 0,19% Mn, 0,08% Mg, 0,62 % Cu, 1,42% Ni and 0,08% S as well as 0,03% P. Melting was carried out in acid-lined, crucible induction furnace of medium frequency, a power of 100kV, and a capacity of 50 kg. The nodularising treatment and inoculation were carried out in furnace crucible using FeNi Mg18 (18%Mg) master alloy in an amount of 1,2% and FeSi75T ferrosilicon in an amount of 1% respective of the melt weight. The temperature of the nodularising treatment was 1400^{0}C, that of pouring - 1370^{o}C. Moulds were prepared from a mixture based on silica sand with bentonite binder. An addition of coal dust was also used. Rods of 10, 20, 40 and 60 mm diameter were cast. From these rods specimens for the heat treatment were prepared. The process of austenitising was carried out at a temperature of 900^{o}C for 2h. Austempering was made in salt bath (50% KNO_3 and 50%$NaNO_3$) at temperatures of 280, 330 and 380^{o}C. After this process, specimens were taken for microstructural examinations. The selected morphological features of microstructural constituents after austempering were examined by means of a computer program called Lucia v. 4.82. The examinations were done at the Department of Iron Alloys in Foundry Research Institute, Krakow

The thermal effects related with phase transformations in the investigated cast iron were examined on a differential scanning calorimeter, type Multi HTC S60. Its construction and operation was described in [5-7] on account of the examinations of phase transformations in grey and nodular graphite cast irons. The samples for calorimetric examinations were taken from castings of 20 and 60 mm diameter. The samples had similar weight from 310 to 340 mg. Tests were carried out under argon protective atmosphere at the heating and cooling rate of 10^{o}C/min. The samples were preheated to a temperature of 880^{o}C, and were held at that temperature for about 20 minutes.

3. The discussion of results

Examples of microstructures in specimens of nodular graphite cast iron after austempering (casting of Φ60 mm) are shown in Figures 1 to 2. The results of measurements of the graphite content and precipitates number per 1 mm^2 of the surface area done by the Lucia program are shown in Figures 3 and 4. Figure 5, on the other hand, shows the results of analysis of an effect that the austempering temperature is expected to have on austenite content in the metallic matrix.

As cast, the content of graphite increases with decreasing cooling rate. Most probably, it is the result of longer time of eutectic transformation, and hence of more favourable conditions for carbon diffusion and graphite precipitates growth. The number of graphite precipitates (Fig. 5) as cast increases with increasing cooling rate. This is the result of the well-known effect of the cooling rate on the value of undercooling and on the number of graphite nuclei during crystallisation. Of much more complex nature is the effect of heat treatment conditions on the discussed parameters of graphite. Both fraction and number of the graphite precipitates decrease with increasing temperature of austempering. This result is quite surprising. The content of carbon dissolved in austenite during austenitising depends on the temperature and time of holding, and both these parameters remain the same for all the examined specimens. It seems quite obvious that the fraction and number of graphite precipitates decreases after this operation, since austenite is saturated with carbon up to the content of about 1,8% (this is, among others, due to the Fe-C phase equilibrium diagram), while the amount of carbon dissolved in austenite and originating from the decomposition of pearlite does not exceed 0,8%. As can be observed, the process of graphite dissolving in austenite is proceeding much more easily in specimens with higher rate of cooling. The number of graphite precipitates in specimens is large, and therefore the path of carbon diffusion is relatively small. Explaining the effect of higher austempering temperature on the reduced fraction and number of graphite precipitates is, at this stage of research, impossible.

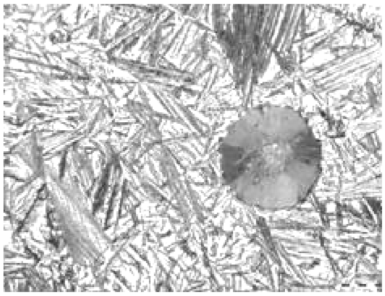

Fig. 1. Microstructure of nodular graphite cast iron after austempering at 330^{o}C

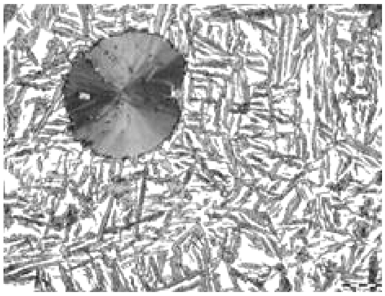

Fig. 2. Microstructure of nodular graphite cast iron after austempering at 380^{o}C

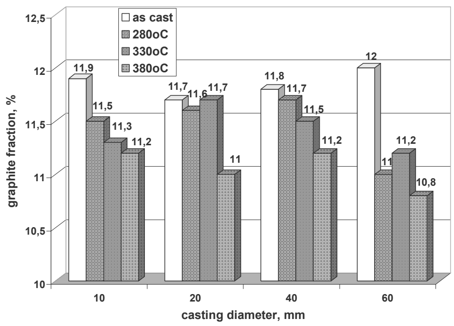

Fig. 3. Effect of austempering temperature on graphite fraction in function of casting cooling rate

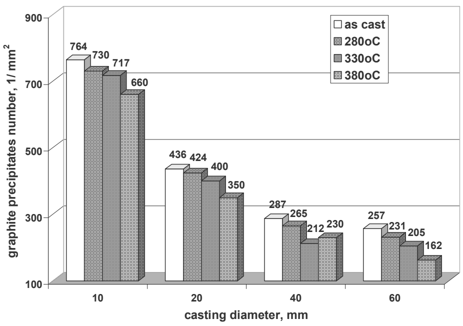

Fig. 4. Effect of austempering temperature on graphite number in function of casting cooling rate

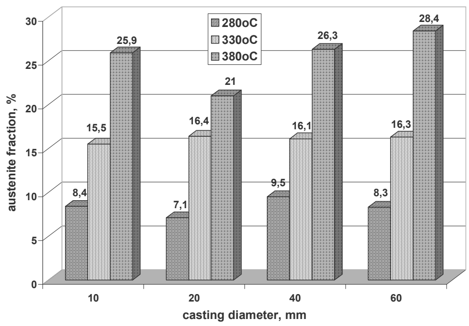

Fig. 5. Effect of austempering temperature on austenite fraction in function of casting cooling rate

An example of diagram plotted from the calorimetric analysis of heating and cooling of 40 mm diameter casting specimen after austempering at a temperature of 330°C is shown in Figure 6.

In these studies it has been assumed that thermal effects accompanying transformations during austempering should appear on the heating diagram as thermal effects of reverse nature. For example, the endothermic effect of eutectoid transformation during heating and cooling shows on the curve as an exothermic effect, etc.

During heating of specimens after austempering, a very strong exothermic effect appears on DSC diagrams.

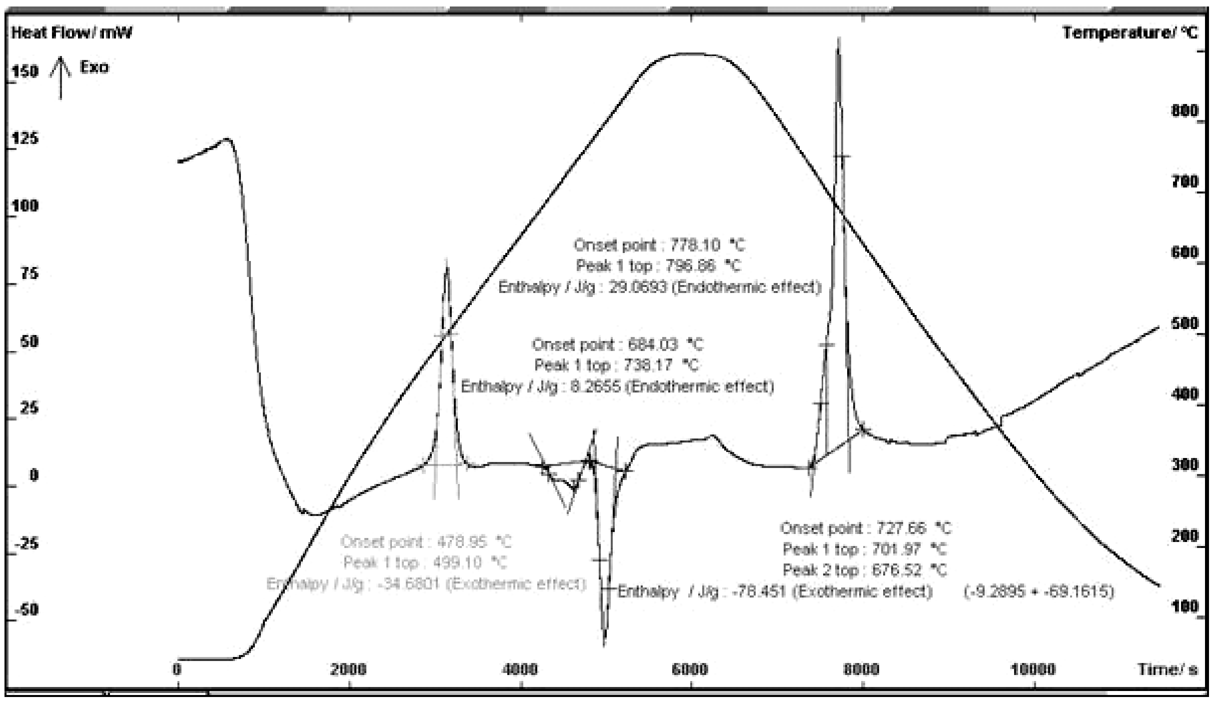

Fig. 6. The DSC diagram of nodular cast iron heating and cooling. Casting specimen of 20 mm diameter after austempering at 330°C

4. Analysis of the results

Figure 7 shows relationship between the temperatures of the beginning and end of exothermic effect, the heat of transformation, and austenite content in the metallic matrix in function of the austempering temperature. The diagram was plotted for samples taken from casting of 20 mm diameter.

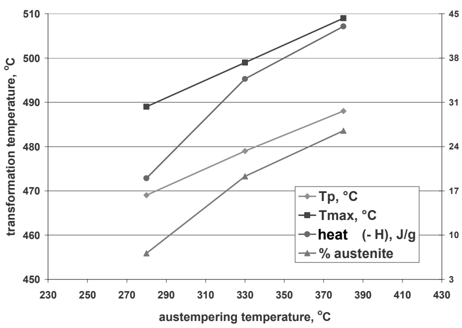

Fig. 7. Relationship between austenite content and range and heat of exothermic effect in function of austempering temperature

As results from Figure 7, the exothermic effect takes place within the range of about 20^{o}C. Its beginning, depending on the austempering temperature, falls to the temperatures from a range of 469 to 490^{o}C. Similar results were obtained on samples taken from casting of 60 mm diameter.

Special attention deserves, first of all, an almost proportional relationship between the heat of this effect and austenite content. Therefore it has been assumed that it results from the presence of austenite in the sample as an effect of transformations during austempering. Consequently, a question arises - is during austempering an endothermic effect present? If so, then what accounts for its presence? It is the fact well-known that during cast iron cooling all effects are of an exothermic nature as proved by the DSC curve shown in Figure 6 and by the results of investigations obtained so far [5, 6].

Besides the above mentioned exothermic effect present on the DSC curve during heating, two endothermic effects also take place. The first of them is due to the decomposition of pearlite or bainite, following the sequence given below:

$$(\alpha + Fe_3C) \rightarrow \gamma_1(C_1)$$

The second effect is probably due to ferrite transformation (present in base cast iron sample) into austenite, following the sequence given below:

$$\alpha \rightarrow \gamma_2(C_2)$$

The third effect related with the process of carbon dissolution in austenite occurs no longer on the DSC curve. Austenite in this cast iron already contains carbon in an amount approaching the state of saturation at a temperature close to the point of eutectoid transformation.

The processes proceeding during cooling are of a nature similar to the case of common grey and nodular graphite cast irons.

At certain temperature, two consecutive exothermic effects take place. The first effect is related with transformation of austenite into ferrite, following the sequence given below:

$$\gamma \rightarrow \alpha + \gamma_1$$

The remaining austenite undergoes eutectoid transformation, following the sequence given below:

$$\gamma_1 \rightarrow (\alpha + Fe_3C)$$

After the process of heating and cooling, the cast iron of base ausferritic microstructure acquires the traditional microstructure of nodular graphite cast iron characterised by pearlitic-ferritic matrix.

5. Conclusions

Although the technology of making austempered ductile iron ADI has been mastered in practice, the phenomena that accompany the formation of ausferritic matrix have not been as yet fully explained. This refers, first of all, to the mechanism of transformation (or several transformations) taking place during austempering.

The results of calorimetric analysis made for ADI samples after preheating have revealed the presence of one very strong exothermic effect, which does not occur in cast iron of common grades. So, the question is: does during the process of austempering an endothermic effect occur? Can the „forced" retention of carbon in austenite be this process? The explanation requires very precise calorimetric investigations of the process of austempering. The studies are at the stage of being prepared.

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