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Design and optimization of stepped austempered ductile iron using characterization techniques

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ARTICLE DATA

Article history:
Received 24 January 2013
Received in revised form 3 May 2013
Accepted 7 June 2013

Keywords:
Metals
Heat treatment
Optical metallography
Phase transformations

ABSTRACT

Conventional characterization techniques such as dilatometry, X-ray diffraction and metallography were used to select and optimize temperatures and times for conventional and stepped austempering. Austenitization and conventional austempering time was selected when the dilatometry graphs showed a constant expansion value. A special heat color-etching technique was applied to distinguish between the untransformed austenite and high carbon stabilized austenite which had formed during the treatments. Finally, it was found that carbide precipitation was absent during the stepped austempering in contrast to conventional austempering, on which carbide evidence was found.

1. Introduction

Austempered ductile iron (ADI) has been used to replace forged steel components in several applications such as crankshafts, gears, bit of drill and rolls and also in some structural applications [1,2] due to its relatively low production costs and excellent combination of good ductility at high tensile strength, high fatigue strength, and superior wear resistance [3]. These outstanding ADI properties are related to its microstructure that consists of a mixture of ferrite needles and high carbon stabilized austenite (HCSA), aggregate that is commonly referred as ausferrite.

During conventional austempering, two-stage phase transformation occurs in ADI [4]. First, austenite decomposes into ferrite and HCSA. A second reaction takes place if the casting is held at the austempering temperature for longer time, which causes decomposition of the HCSA into ferrite and carbides (commonly transition carbides). The presence of carbides makes the material more brittle and that is why this second reaction must be avoided. On the other hand, it could occur that some austenite remains as untransformed austenite (UA) in the microstructure and then it transforms into martensite when ADI is cooled to room temperature before first reaction completion. The period between the end of the first reaction and the onset of the second is termed as processing window [5]. It is well established that the best combination of mechanical properties is obtained in ADI when it is processed at this processing window [5].

Bayati et al. [5] and Hsun and Chuang [6] have reported that process window can be expanded if a stepped austempering is applied. This variant consists of using a lower temperature after the first one in order to transform the UA that remains in the intercellular regions. On the other hand, conventional characterization techniques such as dilatometry, X-ray diffraction and metallography offer the advantages of being simple and easy to implement to study phase transformations. The information that can be obtained by using those techniques includes: intercritical region temperatures, austenitization temperature

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http://dx.doi.org/10.1016/j.matchar.2013.06.004

and time, martensite start temperature and austempering time. In addition, it is an important advantage that the material consumption for these tests is negligible.

The current work provides results on the austenitization and conventional austempering behavior of a non alloyed ductile iron through the use of the characterization techniques that have been mentioned. Furthermore, stepped austempering temperature and time has been determined in order to establish the processing window for the composition employed.

2. Experimental Procedure

The samples for all tests were obtained from the bottom part of "keel" blocks in order to avoid defects such as porosity and segregation which commonly appear in the top area. Chemical composition of the ADI is presented in Table 1. The as-cast microstructure characterization was done taking as a reference the standard ASTM A-247 [7]. Volume fraction of the constituents was determined according to ASTM E-562 [8]. Samples for dilatometry were machined with a diameter of 2 mm and 12 mm long. A dilatometer Adamel-Lhomargy was used to perform these experiments. The heating and cooling rate for all tests was 1 °C/s. The austenitization temperature was 900 °C for a maximum time of 30 min. This temperature was selected according to a previous test by which the intercritical region was determined. Cycles of the conventional treatment were performed at 300 and a 370 °C for a maximum time of 110 min. The number of samples tested on the dilatometry was 3 on each run. An additional experiment was done to determine the martensite start temperature because this value was important for establishing the second transformation temperature of the stepped austempering in order to avoid martensite precipitation. The value found was 182 °C. This test was also useful for evaluating the C content dissolved in the austenite (C_0) during austenitization which was calculated by using the following expression proposed by Darwish and Elliot [9]:

$$C_\gamma^0 = \lambda\left(18/\left(5\sin^2\theta_{112} - 2\sin^2\theta_{211}\right)\right)^{1/2} - 5.722/_{0.232} \quad (1)$$

where θ_{112} and θ_{211} are the martensite peaks position measured in an X-ray diffractogram and λ is the wavelength of the radiation used. On the other hand, stepped austempering was applied using a transformation temperature of 370 °C for 30 min in the first step, and for the second step the temperatures were 210, 250 and 280 °C for several times.

Metallographic preparation consisted of grinding with SiC paper using the following mesh sizes: 120, 320, 400, 600 and 1000. At least 0.5 mm was eliminated by this operation in order to avoid any presence of superficial corrosion or decarburization. Then, samples were polished with a lap and a diamond suspension of 3 and 1 μm size. Etching was done using 2% Nital.

Table 1 – Chemical composition of the ADI used in this work.

Element	C	Si	Mn	P	S	Sn	Cu	Cr	Mg
wt.%	3.68	2.46	.47	0.019	0.005	0.005	0.31	0.04	0.039

Table 2 – Color that different phases exhibit in ADI after especial heat color-etching [11].

Phase	Segregation carbides	HCSA	UA	Ferrite
Color displayed	White	Pink and purple	Light blue	Brown and green

Selected samples were subjected to special heat color-etching in order to distinguish the HCSA from UA. The color that each phase showed after this procedure is presented in Table 2. Microstructural observations were carried out in an SEM Phillips XL-30 equipped with an energy dispersive spectroscopy unit. In a previous report of our group [10] we have calculated and discussed HCSA volume fraction and its C content by two quantification methods: MAUD software (MAUD) and direct comparison method (CD). These values are taken into account in the present work for some calculations.

3. Results and Discussion

3.1. *As-cast Microstructure*

Table 3 shows the as-cast microstructure characterization results. The nodularity was close to the limit (80%) recommended in the literature for an adequate response to the austempering treatment [12]. On the other hand, nodule count was lower than the 100 nodules/mm^2 which is also suggested in the same reference. Micrographs that show the appearance of ductile iron with and without etching are presented in Fig. 1A) and B), respectively. As can be seen in those micrographs, the highest volume fraction consisted of pearlite while the amount of ferrite was lower.

Segregation of carbides and micropores were found close to the intercellular region (Fig. 1C) and D)). It was evidenced by chemical analysis that the composition of such carbides contained Cr and Mn. These elements probably come from the scrap used during the primary ductile iron production. On the other hand, it was found a severe Si and Mn segregation to different areas of the microstructure. For example, Si segregated preferentially to regions close to the nodule reaching a value of 3.3 wt.% while close to the intercellular region the content was 1%.

The Mn content in those areas was 0.5% and 2.7%, respectively. The segregation level which was found would exert an important influence in the subsequent austenitization and austempering steps. These processes will be discussed in next paragraphs.

Table 3 – Graphite nodules characterization and phase volume fraction in the as-cast ductile iron.

Nodularity (%)	Nodule count (nodules/mm^2)	% pearlite volume fraction	% ferrite volume fraction	% graphite volume fraction
85 ± 4	94 ± 5	55 ± 2.2	33 ± 2.2	12 ± 1.8