DETERMINATION OF PROCESSING WINDOW FOR ADI MATERIALS ALLOYED WITH COPPER

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Abstract

Austempered ductile iron (ADI) materials belong to the group of new materials which are processed by isothermal transformation of nodular cast iron when the unique microstructure – ausferrite is obtained. The aim of the paper was to investigate the effect of austempering parameters (time and temperature) on the microstructure and mechanical properties of ADI alloyed with 0.45% Cu in order to establish the optimal processing window. Light microscopy (LM) and X-ray diffraction (XRD) analysis were performed for microstructural characterization, whereas hardness, tensile and impact tests were determined after different austempering temperatures. It was shown that strength, elongation and impact energy strongly depend on amounts of ausferritic ferrite and stable, high carbon enriched retained, reacted austenite. Processing window was established according to the results of kinetics of isothermal transformation. The processing window is open from 300 to 400°C and above this temperature is closed.
Key words: austempered ductile iron, processing window, ausferritic ferrite

Introduction

Austempered ductile iron (ADI) has received much publicity in recent years because of its excellent mechanical properties, i.e. the favorable combination of strength, toughness, fatigue strength and wear resistance [1-3]. ADI is produced by austempering heat treatment of ductile cast iron.

Silicon is the alloying element added (>2%) to ductile iron in order to suppress the formation of carbides normally associated with bainitic reactions, allowing carbon rejected by the formation of ferrite to enrich the carbon content of the retained austenite

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surrounding the ferrite platelets. Austenite is enriched to the extent it can become thermally stable to well below the room temperature. This initial transformation when the primary austenite (γ) decomposes into ferrite (α) and retained high-carbon stable austenite (γ_{HC}) is commonly known as the Stage I reaction [4], i.e.:

$$\gamma \rightarrow \alpha + \gamma_{HC} \tag{1}$$

If the castings are held at the austempering temperature for too long, then a second reaction (Stage II) sets in, where high-carbon austenite further decomposes into ferrite and carbide:

$$\gamma_{HC} \rightarrow \alpha + carbide \tag{2}$$

This reaction is undesirable since it causes the embrittlement of structure and degrades the mechanical properties of ADI [5].

Different terminologies corresponding to ADI microstructure may be found in the literature regarding the state of austenite and ferrite. For example, some authors apply the term (carbide free) bainitic ferrite (α) to the state of ferrite in ADI [6]. This is due to the fact that, similar to high silicon steels (>1.5%), austenite in ADI transforms to carbide-free ferrite.

Other researchers [7] follow a more unified definition for ADI microstructural consistituents. In this definition, ferrite in ADI microstructure is referred to either as acicular ferrite or bainitic ferrite. Acicular ferrite or ausferritic ferrite (as referred in the following text) is a product of the Stage I reaction and bainitic ferrite is a product of the Stage II reaction [8, 9]. The terminology suggested for the three types of austenite of different carbon content in ADI are: (i) un-reacted (metastable) austenite with less than 1.2% carbon, (ii) reacted metastable austenite with carbon content between 1.2%-1.7%, and (iii) reacted stable austenite with carbon content between 1.8%-2.2%, [7] .

The most important microstructural consistuents for this study should be considered as:(i) ausferrite which is a mixture of high carbon austenite and ausferritic ferrite, (ii) ausferritic (acicular) ferrite which is a ferrite phase saturated with carbon similar to carbide free bainitic ferrite in high Si steels [10], (iii) high carbon austenite with a carbon content from 1.2 to 2.2%, and (iv) unreacted austenite which contains less than 1.2% carbon and transforms to martensite at room temperature. The high carbon austenite which is stable at room temperature (retained austenite) is a mixture of reacted stabilized austenite(1.8-2.2%C) and reacted metastable austenite (1.2-1.7%C).

The optimum mechanical properties can be achieved upon completion of the first reaction, but before second reaction started. The time period between the end of Stage I and the start of Stage II is called processing window. In addition, processing window is the time interval over which the standard grades of ADI material can be produced [8]. Processing window defines the austempering time interval, ($t_2 - t_1$) over which the start of the processing window (time, t_1) has been defined as the austempering time at which the amount of unreacted austenite, low carbon austenite has been reduced to 3%. The end of the processing window (time, t_2) has been defined as the time at which the maximum retained austenite level has been reduced by 10% [8, 11].

The aim of this paper was to determine the effect of austempering parameters (time and temperature) on the microstructure and mechanical properties of ADI alloyed with 0.45% Cu in order to establish processing window in which ADI will satisfy optimal mechanical properties prescribed by the ASTM A897 M-03 and EN 1564:2005 standards.

Experimental

The ductile iron alloyed with 0.45% Cu has been examined in as-cast and austempered condition. The chemical compositions of as cast materials are given in Table 1.

An alloyed ADI (designated as CuSG) with the chemical composition given in Table 1 was produced by a sandwich spheroidization treatment. The melt was poured from about 1420°C into standard 25.4mm Y block sand molds (ASTM A-395), which ensured sound castings. Ductile iron keel blocks were produced in a commercial electro-induction foundry furnace.

Tensile specimens of 6 mm diameter and 30 mm gauge length and un-notched Charpy specimens (55 x 55 x 10 mm) were machined from the Y-blocks.

Specimens austenitized in a protective argon atmosphere at 900°C for 2h were rapidly transferred to a salt bath at the austempereing temperature 300, 350 and 400°C, held for 1, 2, 3, 4 and 6 h and then air-cooled to room temperature.

Standard metallographic preparation technique (mechanical grinding and polishing followed by etching with Nital) was applied prior to light microscopy (LM) examinations of samples cut from Charpy impact specimens.

For microstructural characterization, a "Leitz-Orthoplan" metallographic microscope was used.

The volume fraction unreacted austenite in ADI material (start of the processing window (t_1) was determined using the "Leitz-Orthoplan" light microscope and image analysis methods "Qwin Lite Plus Leica Imaging Systems".

The volume fraction of unreacted austenite was measured by a technique of heat tinting [5]. Specimens etched by the standard procedure were heated in a furnace without a protective atmosphere at a temperature of 260°C for a period of 330 min and then air cooled to room temperature. After this heat treatment the volume fraction of primary austenite appears as light blue, high-carbon austenite as purple red, ausferritic ferrite as light brown (beige), eutectic carbide as white, while the martensite turns dark blue.

The change in the volume fraction of retained austenite during austempering was determined by X-ray diffraction (XRD) analysis using a Siemens D-500 diffractometer with nickel filtered Cu-K_α radiation. The diffractograms were analyzed applying the direct comparison method [12].

Tensile tests of austempered specimens were performed on 50 kN hydraulic machine with a constant cross-beam travel speed of 1 mm/min. For all samples mechanical properties, namely, tensile properties (R_m-ultimate tensile strength, $R_{p0,2}$-proof strength, A_5-elongation, EN 10002), Charpy impact energy (K_0, EN 10045) and Vickers hardness (HV_{10}, ISO 6507) were determined at room temperature. At least three specimens were tested for each heat treatment.

Results and discussion

As-cast microstructure

The chemical composition of the experimental ductile iron is listed in Table 1.

Table 1. Chemical composition of as-cast material (mass %)

Material	Chemical composition, (mass %)						
	C	Si	Mn	P	S	Cu	
DI Cu	3.64	2.49	0.3	0.014	0.014	0.46	

The spheroidisation of graphite in as-cast ductile iron alloyed with Cu was higher than 90%. Microstructure is dominantly pearlitic with 10% ferrite and 13% graphite. The morphology of graphite nodules in the microstructure is fully spherical, with the nodule count distribution varying from 60 to 80 nodules/mm^2. Spheroidization is evident (>90%) with an average nodule size ranging between 40 and 55 μm (Fig. 1).
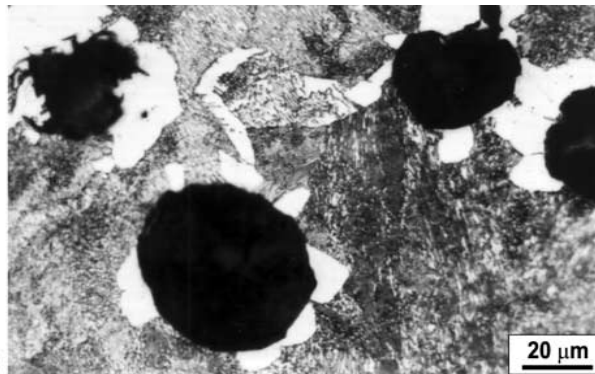

Fig. 1 LM. Microstructure of as-cast ADI specimen

Results of quantitative analysis of microstructures together with the volume fraction of phases present in as-cast CuSG ductile iron are given in Table 2.

Table 2. The results of quantitative analysis of CuSG ductile iron microstructures

Material	Shape factor,	Nodule size (μm)	Nodule count, (mm^{-2})	Ferrite (Vol.%)	Pearlite, (Vol.%)	Graphite, (Vol.%)	Carbide, (Vol.%)
DI Cu	0.90	40-55	60-80	10	90	13	-

The volume fraction of unreacted austenite

The variation of volume fraction of unreacted austenite with austempering time at 300, 350 and 400°C is given in Fig. 2.
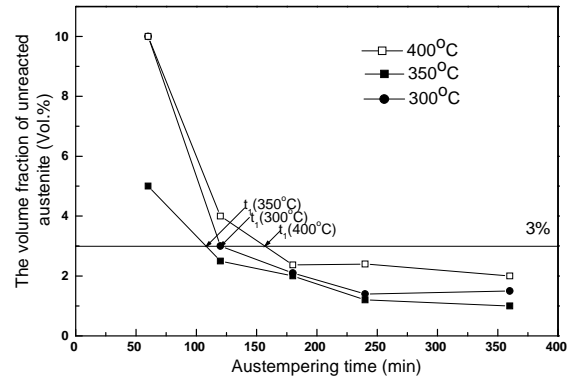
Fig.2 The varation of volume fraction of unreacted austenite with austempering time at austempering temperatures 300, 350 and 400°C

The end of the Stage I reaction, distinguished by the appearance of intercellular boundaries occurring after 2h of austempering, corresponds to a time when the volume fraction of unreacted austenite reaches the critical value of 3%. According to Elliot [13] with longer time of austempering the Stage I reaction becomes more present in the intercellular area. The results of other authors [14,15] showed that the Stage I reaction in intercellular area is manifested by a decreased volume fraction of unreacted austenite, an increased volume fraction of retained austenite and a decreased values of hardness.

The time when the Stage I reaction is terminated at 300°C is shortest compared to other of the austempering temperatures. This phenomenon, noted in this paper, was also described by other authors [12-15]. Generally, this behaviour is related to the fact that during cooling to 300°C Stage I reaction occurs at highest rate than at other austempering temperatures. Also, at Stage I a finer distribution of ferrite needles in austenite matrix should be expected.
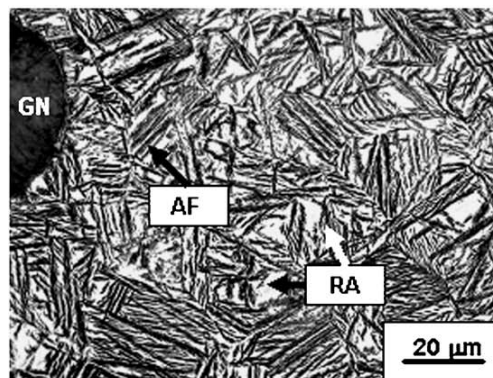

Fig. 3 LM. Microstructure austempered CuSG after austempering at 350°C/2h (tinting technique); GN-graphite nodule, AF-ausferrite, RA-retained austenite.

The volume fraction of retained austenite

        The representative microstructure of the ADI alloyed with Cu austempered at 350°C/2h is shown in Fig. 3. The microstructure is fully ausferritic consisting of a mixture of ausferritic ferrite and carbon enriched retained austenite. The microstructures for other austempering conditions are discussed in detail elsewhere [14-19].
        The variation of volume fraction of retained austenite with austempering time at 300, 350 and 400°C is given in Fig. 4.
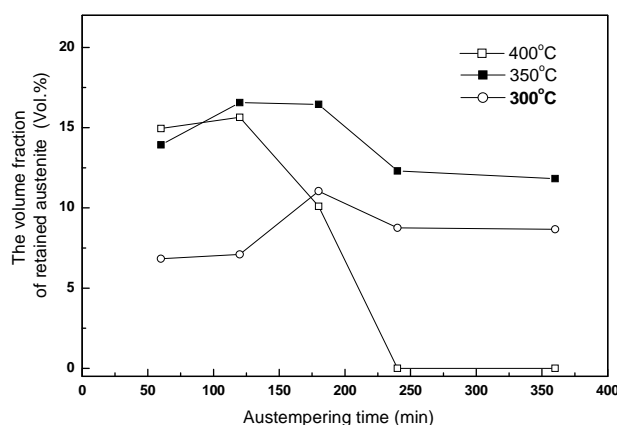

Fig. 4 The variation of the volume fraction of retained austenite with austempering time at austempering temperatures 300, 350 and 400°C

        Time and temperature of isothermal transformation during austempering treatment have a marked influence on the relative amount of retained austenite (Fig.4). It should be noted that from the shape of curves it is apparent that two stages are involved in the isothermal transformation. In the Stage I reaction (time less than 2h) the amount of retained austenite increases with time. According to some authors [15, 16] this may be explained taking into account that the transformation to bainite was not completed. It is well documented [17] that austenitic regions having low silicon and high carbon concentration, i.e. regions between graphite nodules, will not undergo transformation (to bainitic ferrite and retained austenite) during short time of austempering. Also, during the subsequent cooling from austempering to room temperature the formation of martensite cannot be prevented. However, after 2h of austempering the amount of retained austenite decreases, indicating the start of the Stage II of austempering reaction when retained austenite decomposes to bainitic ferrite and carbide. At 400°C this decrease is more pronounced and is associated with the decomposition of austenite to ferrite and carbide [18].

XRD analysis

        XRD patterns of specimens austempered at different temperatures are shown in Fig. 5(a-c).

The volume fraction of the retained austenite was determined from the integrated area under austenite and ferrite peaks. The shift in the volume fraction of retained austenite during austenite is clearly visible in Fig. 5.
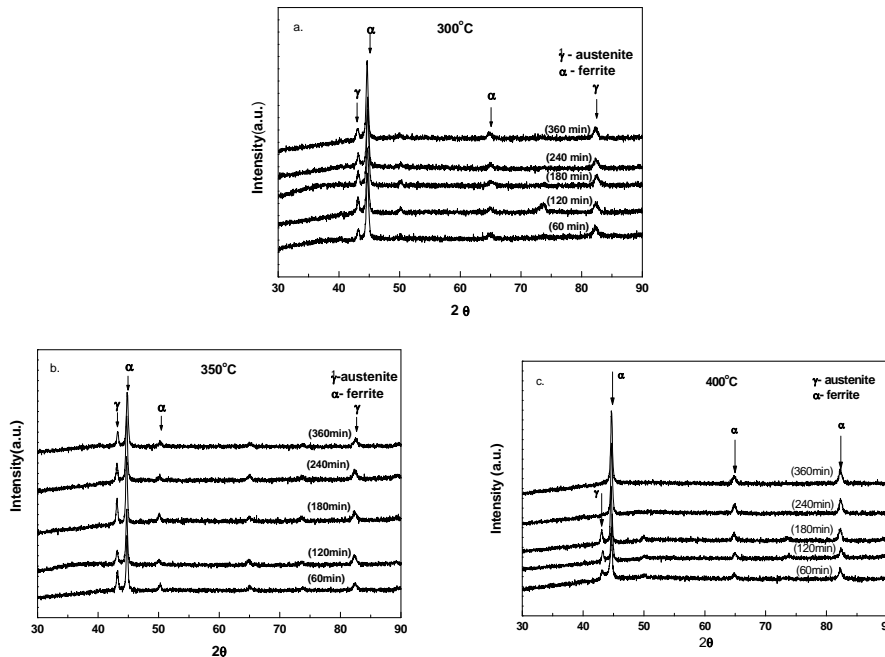

Fig. 5 XRD patterns at different austempering temperatures.
(a) 300°C; (b) 350°C; (c) 400°C

The result which illustrates a very pronounced decrease of retained austenite after 4h at 400C is agreement with result shown in Fig. 4 [18].

Austempering kinetics

The change of hardness with austempering time for austempering temperatures 300, 350 and 400^{0}C is demonstrated in Fig. 6.

Hardness shows minimum values approximately after 180 min of austempering, the value roughly corresponding to maximum volume fraction of retained austenite after austempering at 300 and 350°C, whereas this maximum at 400°C was reached much earlier (see Fig. 4).

The variation of hardness, volume fractions of retained austenite and unreacted austenite with austempering time can be used to gain information concerning the kinetics on the Stages I and II reactions in the austempering process.

The progress of the Stage I reaction is accompanied by an increase in retained austenite content and by a decrease in hardness and volume fraction of unreacted austenite. The Stage II reaction produces an increase in hardness and a decrease in retained austenite volume fraction. The change in these parameters with austempering

time for different austempering temperatures, which is in accordance with the results of other authors [14-16], is shown in Figs. 4-6.
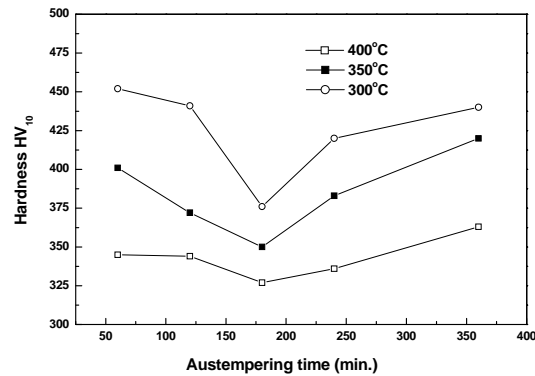

Fig. 6 The variation of hardness with austempering time at austempering temperatures 300, 350 and 400^{0}C

Austempered microstructure within and outside processing window is illustrated in Fig. 7.

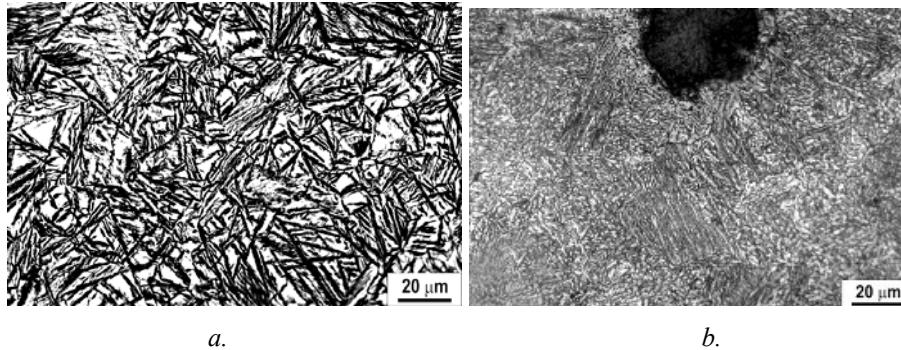a.   b.

Fig. 7 LM. Austempered microstructure. (a) within processing window (350°C/3h); (b) outside processing window (400°C/4h)

The microstructure after 3h of isothermal transformation at 350°C is distinguished by a plate-like morphology of ausferritic ferrite and a higher amount of retained austenite (Fig. 7a). However, after 4h of austempering at 400°C the amount of retained austenite decreases, indicating the full activity of the Stage II reaction when retained austenite decomposes to a mixture of ferrite and precipitated carbides corresponding to a microstructure of a feathery bainite (Fig. 7b). This reaction is undesirable since it degrades the mechanical properties of ADI [19-23]. The result which shows a disappearance of retained austenite after 4h at 400°C is clearly shown by XRD results (see Fig. 4c).

The mechanical properties

The results of ultimate tensile strength, proof strength, elongation, impact energy and Vickers hardness at different austempering temperatures are given in Table 3 [14].

Table 3 Mechanical properties of ADI alloyed with Cu [14]

Austempering		R_m [MPa]	$R_{po.2\%}$ [MPa]	A_s [%]	K_O [J]	HV_{10}	V_Y[%]	ASTM A897M-03	SRPS EN 1564:2005
Temp.	Time								
300°C	1h	1427	1315	1.4[A]	52.6	452	6.8	-	EN-GJS-1400-1
	2h	1428	1346	3.4	59.7	440	7.1	1400/1100/02 (4)	EN-GJS-1400-1
	3h	1445	1342	3.7	76.9	375	11.0	1400/1100/02 (4)	EN-GJS-1400-1
	4h	1481	1391	3.1	71.6	420	8.7	1400/1100/02 (4)	EN-GJS-1400-1
	6h	1412	1345	2.7	2.7	436	8.7	1400/1100/02 (4)	EN-GJS-1400-1
350°C	1h	1158	1026	4.7[AEI]	85.6	402	13.9	-	-
	2h	1112	998	7.9	106.1	373	16.6	1050/750/07(2)	EN-GJS-1000-5
	3h	1109	993	7.1	105.1	350	16.4	1050/750/07(2)	EN-GJS-1000-5
	4h	1205	1121	5.9	100.8	380	12.3	1200/850/04(3)	EN-GJS-1000-5
	6h	1160	1066	5.3[AI]	91.6	420	11.8		EN-GJS-1000-5
400°C	1h	977	760	6.3[AEI]	86.3[A]	345	14.9	-	-
	2h	984	834	7.1[AEI]	89.4[A]	344	15.5	-	-
	3h	987	820	6.2[AEI]	56.5[A]	327	13.7	-	-
	4h	1007	804	4.5[AEI]	23.0[A]	332	0.0	-	-
	6h	1019	884	2.0[AEI]	20.4[A]	364	0.0	-	-

A-označena mehanička karakteristika koja ne ispunjava minimalne uslove prema standardu ASTM A897M-03

E-označena mehanička karakteristika koja ne ispunjava minimalne uslove prema standardu SRPS EN 1564:2005

I-označena mehanička karakteristika koja ne ispunjava minimalne uslove prema standardu ISO17804:2005

The results from Table 3 suggest that the high strength at low austempering temperature is associated with an acicular appearance of ausferritic ferrite with some martensite and retained austenite. Fine structure of ferrite platelets and the low amount of retained austenite contribute to this high strength. As austempering temperature increases the amount of retained austenite decreases. These changes result in reduced strength. Values of elongation and impact energy show maximum at 350°C which coincides with the highest amount of retained austenite (see Figs. 4 and 5).

The optimal processing window

The optimal processing window for ADI material was introduced by Elliott [24] as the time interval in which optimum of mechanical properties, especially ductility, is achieved.

Processing window at 300, 350 and 400°C in the time interval from 1 to 6h of isothermal transformation has been determined based on the calculated values of t_1 and t_2 (Fig.8).
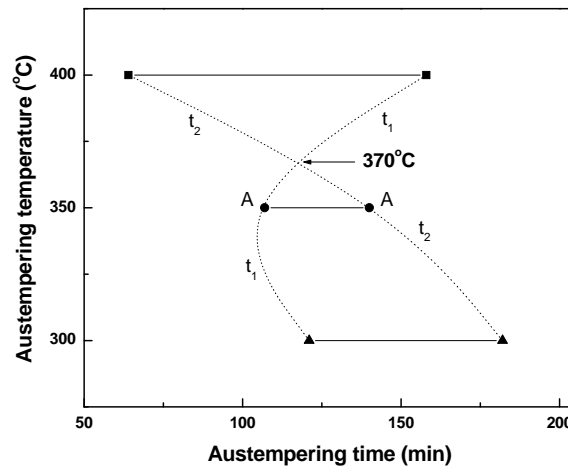

Fig. 8 Austempering window for ADI alloyed with 0.45% Cu

The processing window was determined comparing obtained mechanical properties with minimal values required by standard ASTM A897M-03 and EN 1564:2005 [14]. The processing window is narrower in case of ASTM, as it requires higher values of mechanical properties, especially ductility. On the other hand, the EN standard provides larger processing windows, as its requirements are not so severe. Alloying ductile iron with Cu affects the mechanical properties and the processing window, i.e. ADI alloyed with Cu has a narrower processing window and achieved standard grades are of higher strength and lower ductility.

Results of Fig. 8 show the changes of t_1 and t_2 calculated on the basis of the above procedures (austempering temperature as a function austempering time). Narrower processing window was observed at 300°C. The processing window is open at 300 and closed at 400°C. The area surrounded by curves t_1 and t_2 and positioned below 370°C corresponds to the region where Stage I is dominant. The width of the line A-A inside Stage I indicates the optimal range of processing at 350°C for ADI alloyed with Cu. Intersection of t_1 and t_2 at 370°C denotes overlapping of Stages I and II and above this temperature Stage II reaction is only active. At temperature above 400°C when processing window is closed it is not possible to obtain microstructure that would correspond to ADI material.

Conclusions

Microstructural and mechanical properties of ADI alloyed with 0.45% Cu were studied by means of light microscopy, X-ray diffraction analysis, hardness, tensile and impact tests. It was shown that:

- As-cast microstructure was a predominantly pearlitic with 90% spheroidization of graphite nodules.
- Strength, elongation and impact energy strongly depend on amounts of ausferritic ferrite and retained austenite.
- Fine structure of ferrite platelets and the low amount of retained austenite contribute to this high strength. As austempering temperature increases the amount of retained austenite decreases. These changes result in reduced strength. Values of elongation and impact energy show maximum at 350°C which coincides with the highest amount of retained austenite
- The optimal processing window is open at 300 and closed at 400°C.

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