STRUCTURAL AND MECHANICAL CHARACTERIZATION OF AUSTEMPERED Cu-Sn DUCTILE IRON ALLOYS

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Abstract The effect of heat treatment and alloying elements of copper and tin on the microstructure and mechanical properties of austempered ductile cast iron alloys is investigated. The austenitizing temperature of 890°C, the isothermal transformation (austempering) temperatures of 285, 335 and 375°C, and austempering times of 15, 30, 75 and 150 minutes are studied. The alloying elements of copper and tin are varied within the range of 0-1.5% and 0-0.4%, respectively. The results obtained indicate that the microstructure and mechanical properties of austempered ductileCu-Sn iron alloys are significantly influenced by the isothermal transformation time and temperature. The alloying elements of copper and tin have a pronounced effect on the matrix microstructure of as-cast and annealed ductile irons. These elements show no effect on the morphology of bainite in the austempered condition. However, the volume fraction of retained austenite in the austempered ductile iron alloys increass due to the addition of these elements. The mechanical properties of austempered ductile iron alloys are also significantly influenced by alloying elements. At austempering temperatures of 375 and 335°C, the 1% Cu-0.4%Sn ductile iron alloy austempered for 75 minutes shows the optimum combination of mechanical properties. Conversely, at austempering temperature of 285°C, the 1% Cu-0.2% Sn ducitile iron alloys austempered for 30 minutes represent the optimum combination of mechanical properties.

چکیده تأثیر عملیات حرارتی و عناصر آلیاژی مس و قلع، برروی ریزساختار و خواص مکانیکی چدنهای نشکن آستمپر شده تحقیق گردیده است. برای این منظور دمای آستنیته کردن ۸۹۰°c، دماهای آستمپرینگ ۲۸۵°c، ۳۳۵°c و ۳۷۵°c و زمانهای آستمپرینگ ۱۵، ۳۰، ۷۵ و ۱۵۰ دقیقه آزمایش شدند. عناصر آلیاژی مس و قلع به ترتیب در محدوده ۵/ ۰ - ۰ و ۴/ ۰ - ۰ درصد وزنی استفاده گردید. نتایج حاصل نشان داد که درجه حرارت و زمان آستمپرینگ تأثیر قابل ملاحظه‌ای بر روی ریزساختار و خواص مکانیکی چدنهای نشکن آستمپر شده حاوی مس و قلع دارد. همچنین عناصر آلیاژی مس و قلع تأثیر قابل توجهی برروی ریز ساختار و زمینهٔ چدنهای نشکن در شرایط ریختگی و بازپخت شده دارد. عناصر مزبور هیچگونه تأثیری برروی مورفولوژی بینیت در شرایط آستمپر شده از خود نشان نداد. مع هذا با افزایش عناصر مزبور کسر حجمی آستنیت باقیمانده در چدنهای نشکن افزایش یافت ازسوی دیگر عناصر آلیاژی قلع و مس تأثیر شدیدی برروی خواص مکانیکی چدنهای نشکن آستمپر شده داشت. در درجه حرارت آستمپرینگ ۳۷۵°c و ۳۳۵°c چدنهای نشکن حاوی ۱٪ مس و ۴/ ۰٪ قلع که به مدت زمان ۷۵ دقیقه آستمپر شده بودند مناسب‌ترین ترکیب خواص مکانیکی را از خود نشان دادند. از سوی دیگر در درجه حرارت آستمپرینگ ۲۸۵°c چدن نشکن حاوی ۱٪ مس و ۲/ ۰٪ قلع که به مدت ۳۰ دقیقه آستمپر شده بود مناسب‌ترین ترکیب خواص مکانیکی را داشت.

INTRODUCTION

Austempered Deuctile Irons (ADI) has been a subject of recent interest. This is due to a combination of mechanical properties coupled with relatively low production, i.e. about 30% less than steels with similar mechanical properties[1-3].

ADI exhibit an ultimate tensile strength in the order of 1000 [N/mm^2] [4], an elongation-to-fracture of up to 10% and with a favorable fracture toughness [5,6]. In addition, ADI respond to work hardening treatments at the surface and thus have exceptionally high bending fatigue strength and wear resistance [7]. These excellent properties of ADI are directly related to the unique microstructure consisting of bainitic ferrite and retained austenite. ADI are mainly used in highly stressed machine parts such as gears, chain-links, connecting-rods, load -arms and similar working condition components [8-10].

In ADI, graphite has a nodular form, thus the mechanical properties are mainly controlled by the metallic matrix, produced by the austempering transformation process.

Austempering Process

The proper matrix microstructure in ADI can be produced by a special heat-treatment cycle consisting of austenitizing

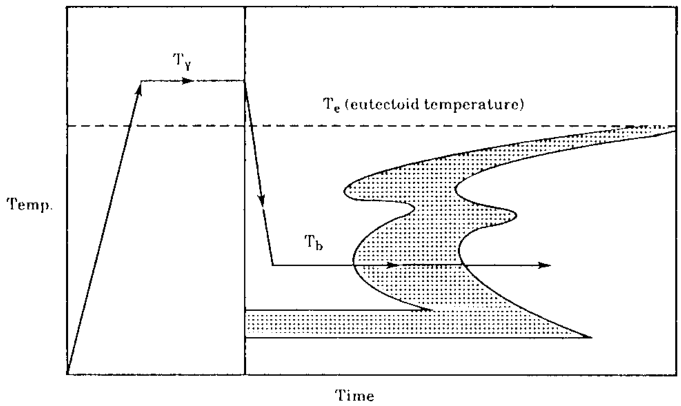

Figure. 1. Schematic illustration of the heat treatment for austempering ductile iron

at a temperature T_γ above 850°C, followed by rapid cooling (usually in a salt bath) to a temperature T_b between 250 and 450°C, holding for a predetermined period of time, and then air cooled to room temperature. This heat treatment is schematically illustrated in Figure 1. The isothermal transformation during austempering in ductile iron is quite different from that in steel. In most steels, austempering results in essentially simultaneous formation of ferrite and carbide from austenite (reaction 1).

$$\gamma \rightarrow \alpha + \text{Carbide} \qquad (1)$$

The ferrite and carbide phases nucleate and grow cooperatively over a relatively short period of time[11-13]. Conversely, the austempering reaction in ductile cast iron is significantly slower than that in steel and is known as a multi-stage reaction [11-13]. During austempering of ducitle iron, bainitic ferrite nucleates and grows into the austenite. At the same time, carbon is rejected from the growing ferrite into the surrounding austenite. The high silicon content in ductile iron suppresses the formation of carbide in the austenite phase. Consequently, the remaining austenite continues to absorb carbon as the reaction proceeds. As the austenite is enriched with carbon, it becomes increasingly stable with respect to $\gamma \rightarrow \alpha$ reaction. Thus, growth of the bainitic ferrite is inhibited and the reaction is temporarily arrested [14].

The remaining austenite can have a carbon content as high as 2% and an Ms temperature below -120°C [14]. However, this high carbon austenite is not stable. With sufficient time the austenite eventually decomposes to ferrite and carbide. Therefore, the austempering transformation in ADI can be described as a two stage reaction, as follows;

$$\text{Stage one:} \qquad \gamma_{\text{matrix}} \rightarrow \alpha + \gamma_{\text{high carbon}} \qquad (2a)$$

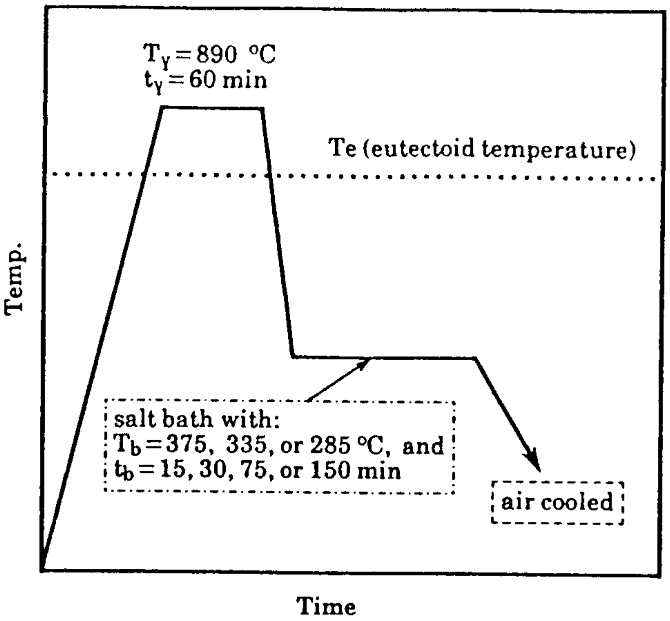

Figure. 2. Schematic presentation of austempering treatment used.

$$\text{Stage two:} \qquad \gamma_{\text{high carbon}} \rightarrow \alpha + \text{Carbide} \qquad (2b)$$

The remarkable combination of mechanical properties attributed to ADI stems from the unique bainitic ferrite-retained austenite microstructure produced by the first reaction (2a), while the second reaction (2b) is known to be undesirable.

The most significant variables that influence the extent of reactions, the resultant microstructure and hence the mechanical properties of ADI are chemical composition, austenitizing time and temperature and austempering time and temperature[15-28]. These variables are briefly discussed here.

Composition

The addition of certain alloying elements is known to increase the hardenability and cause austempering time to be less critical. It has been shown [15-22] that the individual or combined addition of some elements such as copper, nickel, manganese, molybdenum and tin are effective. The maximum recommended percent of elements such as molybdenum, manganese and copper are reported to be 1.3, 1.25 and 2 respectively [24].

Austenitizing Time AND Temperature

The temperature and time of austenitization determine the amount of carbon dissolved in the matrix as well as its distribution. Since the rate of dissolution of nodular graphite is relatively low at the austenitizing temperature, it is important to control the amount of pearlite in the as-cast material. This leads to having the proper amount of carbon

TABLE 1. The composition of Sorel metal used

Element	C	Si	Mn	S	P
Percent	3.8-4.3	1.5	.02-.08	.02-.08	.02-.04

dissolved homogeneously in austenite.

Regarding the two stage reaction proposed, it is believed that time at the austempering can be quite critical. Quenching from the austempering temperature too early produces marteniste as a result of insufficient carbon enrichment of the remaining austenite. The martensite reduces ductility. When the holding time is too long, the second stage of austenite decomposition (carbide precipitation) can occur, which results in a marked reduction in ductility and toughness [25,26]. Conversely, the morphology of bainitic ferrite depends on the transformation temperature[27]. In ductile iron austempered in the range of 350-450°C, the ferrite is relatively coarse and distinct individual units or platelets are produced, while much finer ferrite, which resembles more the typical upper bainite observed in steels, is obtained in the range of 250-330°C. This type of bainite consists of alternate platelets of ferrite and stabilized austenite.

Similar to steels, the two different types of bainite obtained in ADI in the upper and lower ranges of austempering temperatures are sometimes called upper and lower bainite, respectively [28]. It has been shown that [17,27] the mechanical properties, especially the tensile strength, of ADI are significantly influenced by the nature, size and spacing of bainitic ferrite units. For instance, the tensile strength increases with decreasing ferrite-austenite spacing.

EXPERIMENTAL PROCEDURE

Melting was carried out in a 60 kg crucible furnace. The composition of the base cast iron used (sorel metal) is shown in Table 1.

Ductile iron was produced using 75% Fe-Si and 5% Mg-Fe-Si. Other alloying elements such as copper and tin were also added in the form of pure elements. The spheroidizing treatment was carried out by plunging technique. Pouring temperature was maintained around 1400°C. Altogether, six sets of ductile iron melts containg various percentages of copper and tin were prepared. The final composition of experimental ductile cast irons is shown in Table 2.

Each melt was cast in the form of Y-blocks (ASTM-A536-80 standard) in silicate bonded sand molds. All Y-blocks, apart from one from each melt, were annealed at 890°C for 1 hour. Two different types of specimens were then prepared from as cast and annealed Y-blocks, tensile specimens, according to ASTM-A536-80 standard, and 10x10x50 mm V-notch impact specimens.

Six specimens, three V-notch and three tensile, were kept and others were packed in cast iron chips and austenitizied in a muffle furnace at 890°C for 1 hour. The specimens were then removed from the furnace and transferred very quickly to a fused NaOH bath with temperatures of 285, 335 and 375°C and held for 15, 30, 75, and 150 min, and then air cooled to room temperature (Figure 2). The temperatures and transformation times were selected on the basis of previous experiments [29,30].

Tensile and charpy impact tests were carried out on the as cast, annealed and austempered specimens. The brinnell hardness of all specimens was also measured using 187.5 [Kg] load and 2.5 [mm] diameter ball indenter. Optical and

TABLE 2. Chemical composition of experimental ductile cast iron alloys.

Alloys	Composition, Weight Percent								
	Cu*	Sn*	C	Si	Mn	S	P	Mg	Ni	Cu
1	0	0	3.48	2.45	.015	.015	.019	.057	.012	0
2	.5	.2	3.52	2.46	.012	.018	.02	.064	.114	.46
3	.5	.4	3.57	2.6	.013	.016	.017	.056	.117	.45
4	1	.2	3.51	2.55	.015	.017	.014	0.06	.124	.94
5	1	.4	3.51	2.57	.014	.016	.02	.059	.12	.96
6	1.5	.2	3.56	2.57	.012	.019	.016	.054	.119	1.4

* The values of two columns are the nominal composition of copper and tin

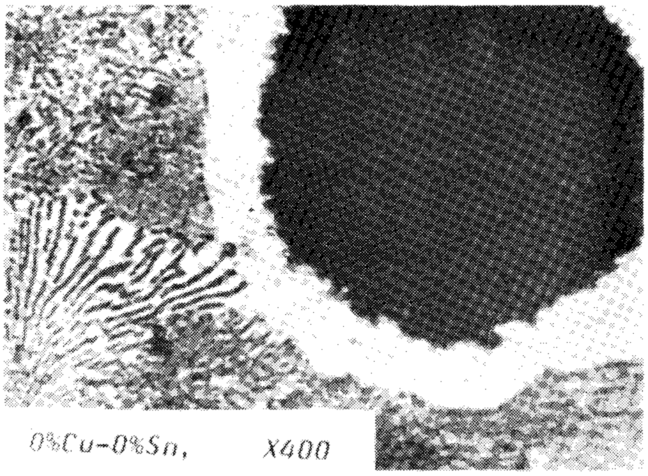

Figure. 3. Optical microstructure of as-cast unalloy ductile iron.

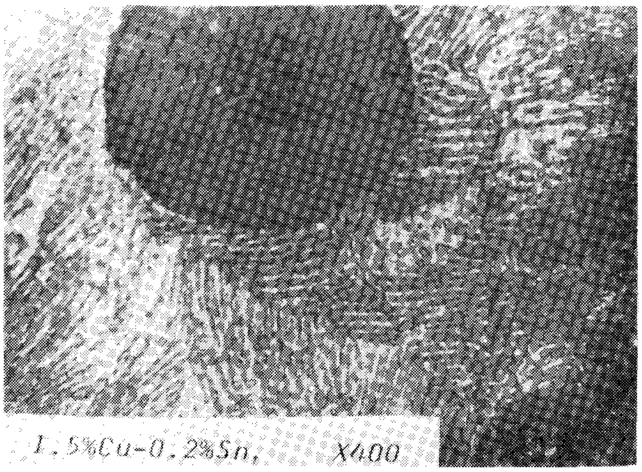

Figure. 4. Optical microstructure of as-cast ductile iron alloy containing copper and tin.

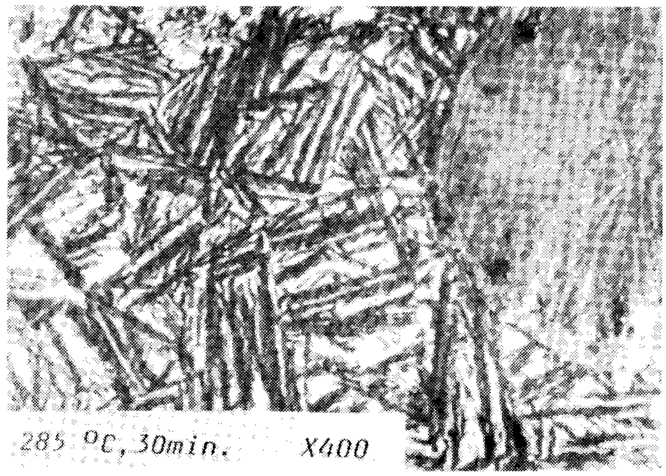

Figure 5. Optical microstructure of 1% Cu-0.2% Sn austempered ductile iron alloy.

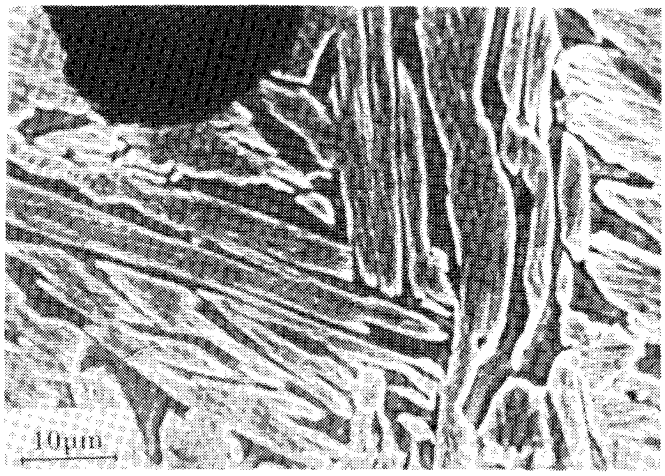

Figure. 6. Scanning electron micrograph of the same specimen shown in Figure 5.

scanning electron microscopy were used to study the microstructure of all the specimens. The scanning electron microscope employed was Jeol model JSM-6400.

RESULTS AND DISCUSSION

As-Cast and Annealed Conditions

Mechanical properties of ductile cast irons produced in the as cast and annealed conditions are presented in Tables 3 and 4.

Examples of the microstructures of these alloys are shown in Figures 3 and 4. The microstructure of the base cast iron consisted of graphite nodules distributed in a pearlite matrix. The graphite nodules were surrounded by a layer of ferrite, i.e., bulleye microstructure (Figure 3). Individually, either 0.4% Sn or 2% Cu was sufficient to eliminate the ferrite layer. Some effect was obtained through simultaneous addition of 1.5% Cu and 0.2% Sn as shown in Figure 4.

Microstructural studies of as-cast and annealed specimens of unalloyed and alloyed ductile irons revealed that additions of copper and/or tin increased the uniformity of matrix microstructure produced under annealed conditions, i.e., the pearlite interlamellar distance and pearlite lamella distribution in the matrix of annealed pearlite interlamellar distance and pearlite lamella distribution in the matrix of annealed alloy ductile iron were much more uniform than in the unalloyed and/or as-cast condition.

Regarding the graphite nodules, the as cast and annealed microstructures of all irons were similar; i.e. they contained mainly the same nodule counts with the same size in the same section of Y-blocks for each composition.

In ductile iron alloys 3, 5 and 6 some intercellular carbides were seen in the as cast microstructures. However, these massive carbides were not present in the heat

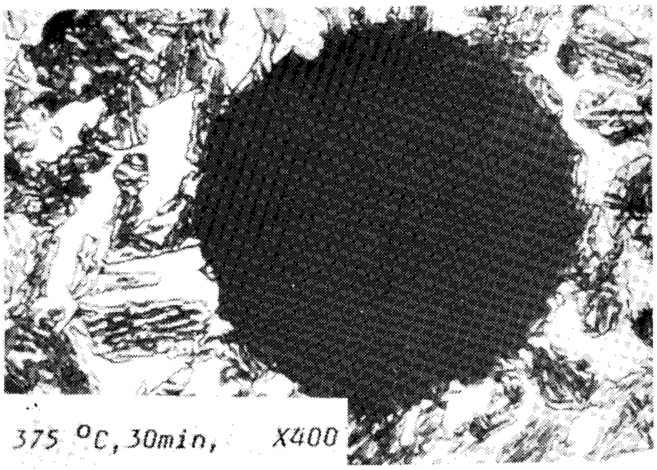

Figure 7. Optical microstructure of 1% Cu-0.4% Sn austempered ductile iron alloy.

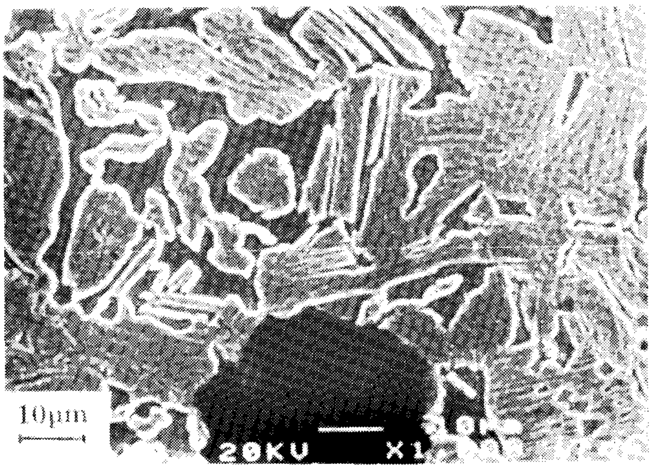

Figure. 8. Scanning electron micrograph of the same specimen shown in Figure 7.

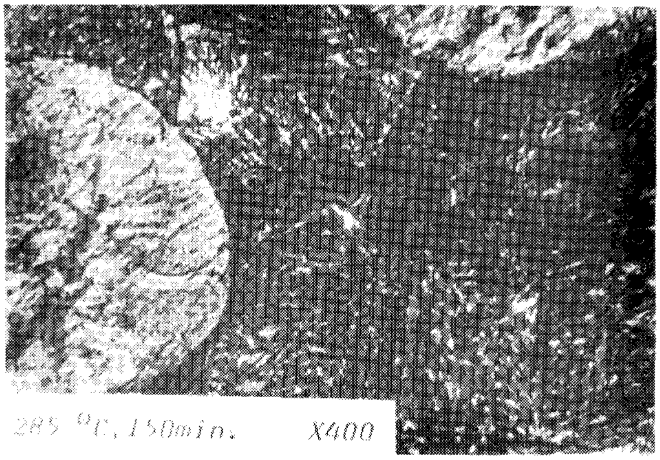

Figure. 9. Optical microstructure of 1% Cu-0.2% sn austempered ductile iron alloy.

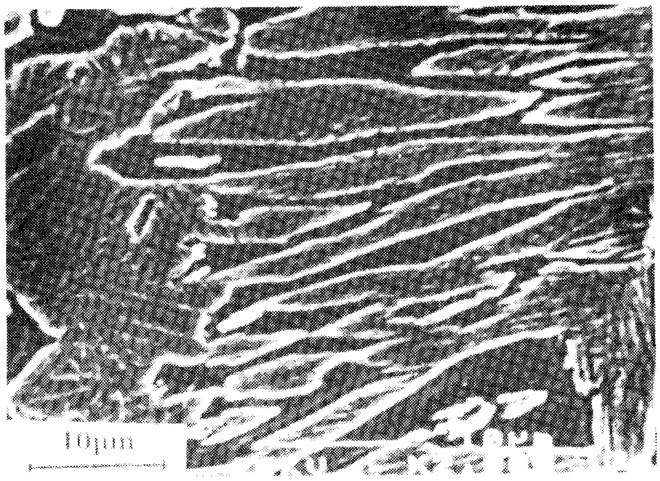

Figure. 10 Scanning electron micrograph of the same specimen shown in Figure 9.

treated annealed condition, indicating that during the austenitizing treatment these carbides dissolved in austenite.

Austempered Condition, the Micro-structure

The investigation of microstructure of ADI specimens by optical and scanning electron microscopy revealed that the time and temperature at which the isothermal transformation of austenite to bainite takes place have a pronounced effect on the volume fraction, morphology and characteristic of various products. With increasing isothermal temperature, a change in the bainite morphology is observed.

At 285°C the bainite has a distinct acicular shape morphology. Figure 5 is an optical micrograph of the alloy containing 1% copper and 0.2% tin isothermally treated at 285°C for 30 minutes. The dark constituent which is the bainite and consists of a fine mixture of ferrite and carbide is clearly shown in the scanning electron micrograph of Figure 6. The stabilized austenite can be seen as bright small regions with almost angular shapes in Figure 5 and as smooth gray areas between the baintic ferrite zones in Figure 6. In this figure, the packets of ferrite and carbide lamella which are the main characteristics of bainite are quite clear.

At higher austempering temperature such as 375°C, the bainite has a more granular morphology as shown in Figure 7, which represents the ductile iron alloy containing 1% copper and 0.4% tin austempered for 30 minutes. This is demonstrated at higher magnification in Figure 8.

The effect of isothermal transformation time on the microstructure of austempered ductile iron depends on the nature of the reaction that occurs, i.e. either first stage (reaction 2a) or second stage (reaction 2b). Within the range of austempering time employed during the course of this research work, it was observed that increasing the

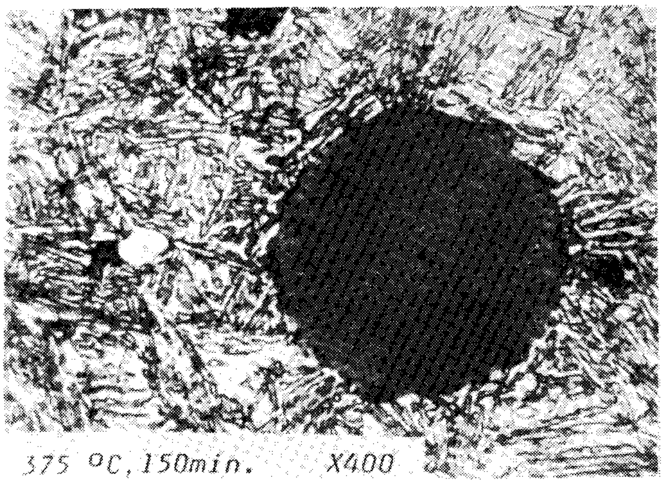

Figure. 11. Optical microstructure of 1% cu-0.4% Sn austempered ductile iron alloy.

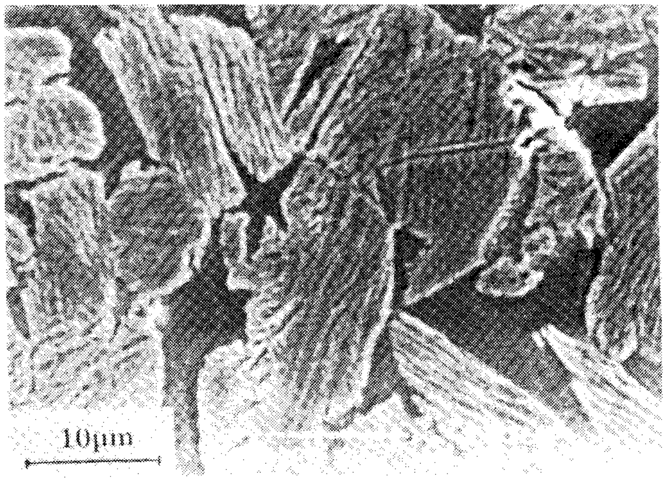

Figure. 12. Scanning electron micrograph of the same specimen shown in Figure 11.

isothermal transformation time at a given austempering temperature resulted in an increase in the volume fraction of bainite. This corresponded to a decrease in the volume fraction of stabilized austenite.

This is clearly seen by comparing Figures 9 and 11 with Figures 5 and 7. The microstructural characteristics of austempering products produced after 150 minutes at low (285°C) and high (375°C) temperatures are demonstrated at higher magnification in scanning electron micrographs of Figures 10 and 12. Using scanning electron microscopy, the precise role of isothermal transformation time and temperature on the two stage austempering reaction (reactions 2a and 2b) is under investigation.

For all transformation temperatures, no sign of pearlite was observed on the fracture surfaces of charpy and tensile specimens made of ADI alloys containing copper > 0.5 and tin > 0.2. However the base ductile iron samples (0% Cu-0% Sn) showed a small dark etching pearlitic area near the center of the sections for the austempering temperature of 375°C. Conversely, at the lower transformation temperature of 285°C there was no sign of pearlite.

The above observation, is simply explained as being due to the hardenability effect of copper and tin, and also due to the increased quench severity with lower austempering transformation temperature. It is worth mentioning that, no sign of any effect of copper and tin alloying elements was seen on the microstructural characteristic and morphology of austempering products, i.e. bainitic ferrite and retained austenite.

From microstructural evolution, it can also be concluded that the maximum obtainable amount of retained austenite depends on transformation temperature and concentration of copper and tin. Lower transformation temperatures result in a smaller amount of retained austenite. Additions of copper and tin enlarge the amount of retained austenite, particularly at high transformation temperature. The exact role of such alloying elements is under study.

TABLE 3. Mechanical properties of as-cast ductile iron

Alloy	Tensile strength (MPa)	Elongation-to-fracture (%)	Hardness (BH)	Impact strength (J)
1	631	4.8	232	3.1
2	738	1.4	275	3.4
3	700	1.2	255	2.6
4	670	2.9	280	3.1
5	480	0.3	360	2 5
6	700	1.3	290	3.9

TABLE 4. The mechanical properties in annealed conditions

Alloy	Tensile strength (MPa)	Elongation-to-fracture (%)	Hardness (BH)	Impact strength (J)
1	510	12	130	3.8
2	538	5	199	3.4
3	430	4.2	166	2.5
4	630	5.6	230	3.7
5	580	2.2	255	3.2
6	620	5.9	230	5.4

Austempered Condition, and the Mechanical Properties

Hardness

The influence of the isothermal time and temperature on the hardness is illustrated in Figure 13. In general, for all ductile iron alloys used, the hardness for the specimens austempered at 285°C is higher than those austempered at 335°C. In other words, the maximum hardness belongs to alloys austempered at lower temperatures.

For most alloys investigated, the hardness generally decreases with increasing austempering time. However, exceptions can be seen, especially at the higher austempering temperature of 375°C. This is most pronounced for the base ductile iron used, i.e., 0% Cu-0% Sn. The results obtained (Figure 13) indicate that the maximum hardness obtained depends on the austempering time and temperature as well as the alloying elements of copper and tin. These are summarized in Table 5.

Tensile Strength

Apart from a few isolated discrepancies, the tensile strength versus austempering temperature virtually follows a similar appearance as the hardness curve (Figure 14). The effect of isothermal transformation time is as follows: for most of the alloys investigated, the tensile strength increases as the austempering time is increased up to 30-75 minutes, and then either remains constant or decreases again. However, a few exceptions were observed.

The results obtained indicated that compared with hardness, the tensile strength is far more affected by the alloying elements of copper and tin. This is more pronounced at higher austempering temperatures, especially 335°C. The maximum tensile strength obtained for the investigated alloys austempered under various conditions is summarized in Table 6.

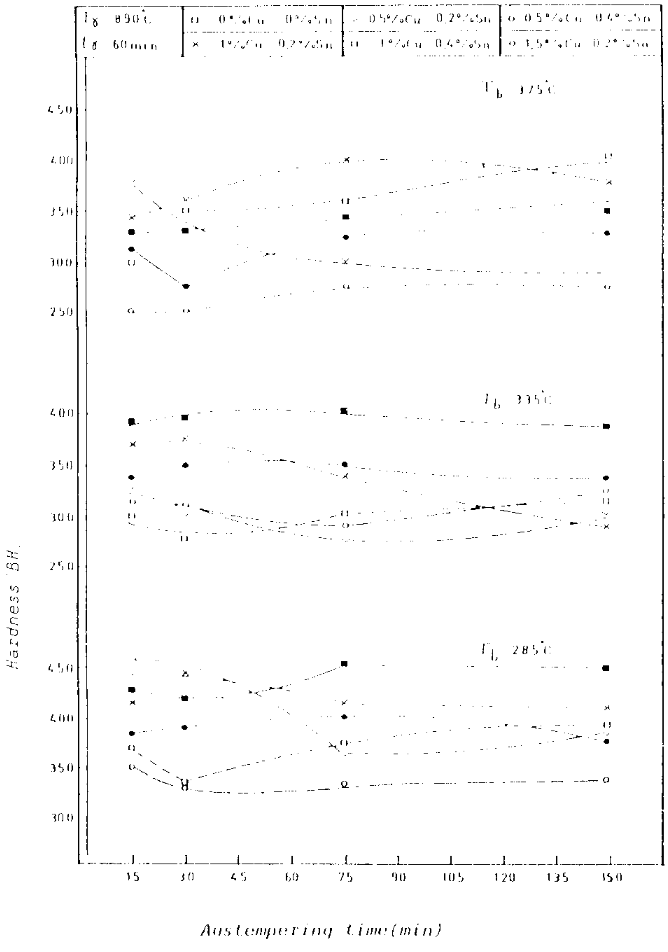

Figure. 13. Hardness as a function of isothermal transformation time for various temperatures.

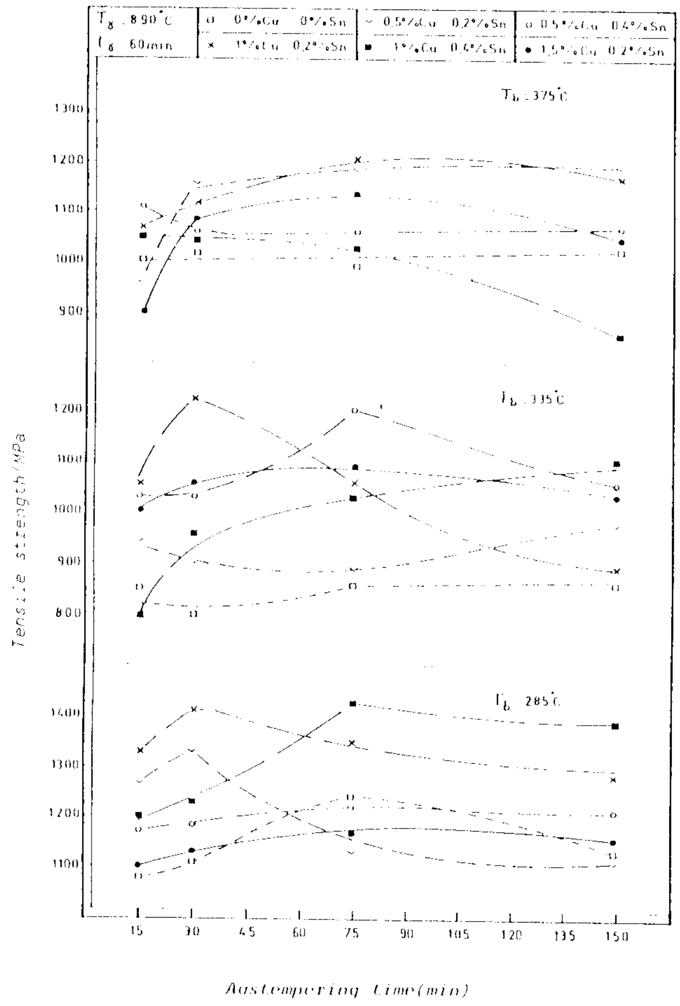

Figure. 14. Tensile strength as a function of isothermal transformation time for various temperatures.

Elongation-to-Fracture

The results obtained from elongation-to-fracture measurements are illustrated in Figure 15. As can be seen, regarding the isothermal transformation time, the variation of the elongation-to-fracture of most of the investigated alloys is somehow similar to the tensile strength behavior. However unlike hardness and tensile strength, when the isothermal transformation temperature is raised, the elongation-to-fracture tends to increase. As can be seen in Figure 15, regardless of austempering time and temperature, the maximum elongation-to-fracture belongs to ductile iron alloy containing 1.5% Cu-0.2% Sn. The maximum values obtained are summarized in Table 7.

The Impact Strength

The impact energy versus austempering time for various

TABLE 5. Maximum hardness obtained for different alloys austempered at various temperatures

Austempering condition		Alloying element		Max. hardness
Temp. (°C)	Time (min)	%Cu	%Sn	(BH)
285	15	0.5	0.2	455
	30	0.5	0.2	455
	75	1	0.4	452
	150	1	0.4	448
335	15	1	0.4	390
	30	1	0.4	396
	75	1	0.4	400
	150	1	0.4	385
375	15	0.5	0.2	375
	30	1	0.2	360
	75	1	0.2	400
	150	0	0	400

TABLE 6. Maximum tensile strength obtained for different alloys austempered at various times and temperatures

Austempering condition		Alloying element		Max.tensile strength
Temp. (°C)	Time (min)	%Cu	%Sn	(MPa)
285	15	1	0.2	1330
	30	1	0.2	1410
	75	1	0.4	1420
	150	1	0.4	1390
335	15	1	0.2	1060
	30	1	0.2	1230
	75	0.5	0.4	1200
	150	1	0.4	1100
375	15	0.5	0.4	1110
	30	0.5	0.2	1150
	75	1	0.2	1200
	150	0	0.2	1190

TABLE 7. Maximum elongation-to-fracture obtained for different alloys austempered at various time and temperatures

Austempering condition		Alloying element		Max. elong-to-fracture
Temp. (°C)	Time (min)	%Cu	%Sn	(%)
285	15	1.5	0.2	5.6
	30			7.4
	75			5.6
	150			4.4
335	15	1.5	0.2	7.4
	30			9.0
	75			9.4
	150			3.4
375	15	1.5	0.2	4.2
	30			9.0
	75			8.0
	150			7.5

TABLE 8. Maximum impact energy obtained under various austempering conditions.

Austempering condition		Alloying element		Impact energy
Temp. (°C)	Time (min)	%Cu	%Sn	(J)
285	15	1.5	0.2	8
	30		0.2	10
	75		0	10.5
	150		0	8
335	15	1.5	0.2	10
	30			10.5
	75			10.5
	150			8.5
375	15	0.5	0.4	10.5
	30	1.5	0.2	12.5
	75	0.5	0.2	13
	150	1.5	0.2	12

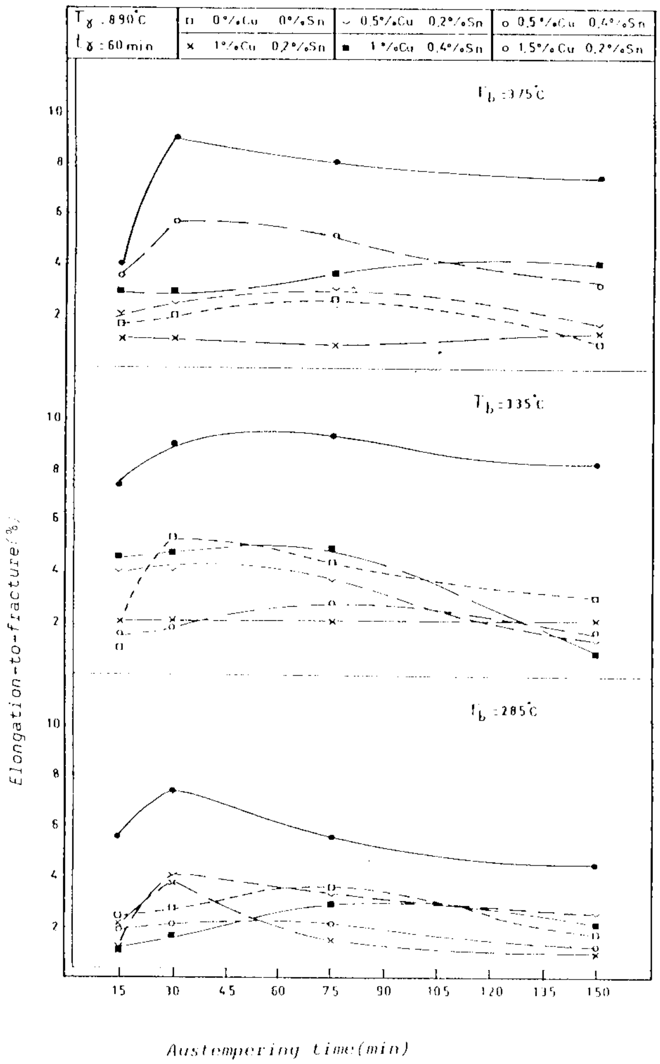

Figure. 15. Elongation-to-fracture as a function of isothermal transformation time.

isothermal transformation temperatures is shown in Figure 16. As can be seen, the impact energy of all ductile iron alloys austempered under various conditions were in a closed spread. However, there is a general trend towards higher impact strength with increasing austempering temperature.

Although a general rule describing the effect of time on the impact energy was not observed, the results obtained indicate that, in most cases, especially at the higher isothermal transformation temperature of 375°C, the impact energy versus austempering time curve goes through a maximum. No obvious relationship exists between the time required to achieve maximum impact energy and the concentration of alloying elements. The maximum values obtained under various conditions are summarized in Table 8.

CONCLUSION

In this research work the microstructure and mechanical

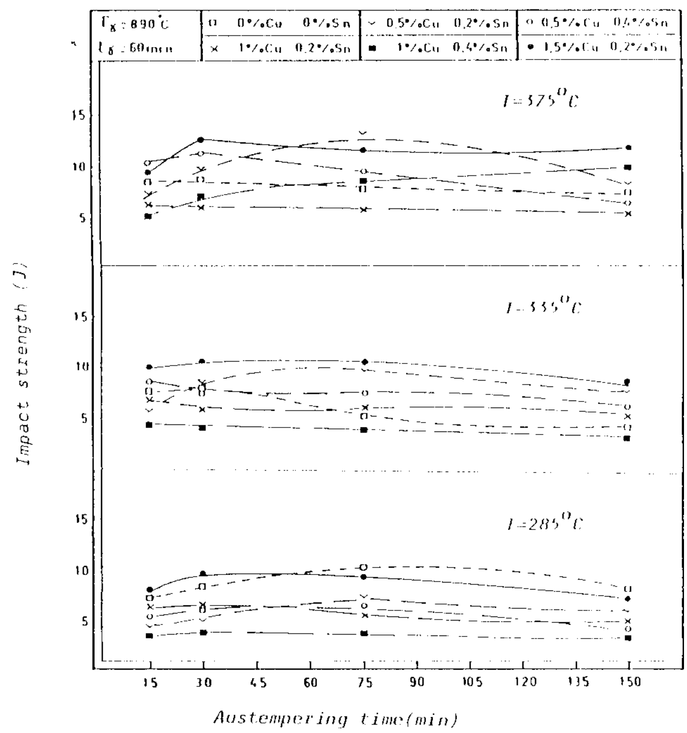

Figure. 16. Impact strength as a function of isothermal transformation time.

properties of austempered ductile iron alloys containing various percentages of copper (0, 0.5, 1 and 1.5) and tin (0, 0.2 and 0.4) have been investigated. The isothermal transformation temperatures used were 285, 335 and 375°C. The austempering times employed were 15, 30, 75 and 150 minutes. The results obtained can be summarized as follows:

1. Addition of alloying elements of copper and tin to ductile iron increased the volume fraction of peartile and the uniformity of pearlite distribution in the as cast and annealed condition. It was also found that, the presence of these alloying elements in ductile irons increases the hardenability during the austempering process.

2. The microstructure of all austempered ductile iron alloys produced consisted of bainite with various amounts of retained austenite. The amount of retained austenite decreased as the austempering time and temperature increased. It was also found that the morphology of bainite changed from acicular shape, at the low austempering temperature of 285°C, to a more granular shape at the high austempering temperature of 375°C.

3. In general, the alloying elements of copper and tin enlarge the amount of stabilized austenite, especially at the high austempering temperature. Conversely, no effect was observed on the nature and morphology of bainite.

4. Combined additions of alloying elements of copper and tin to ductile irons showed a pronounced effect on the mechanical properties. For instance, combined addition of 1% Cu-0.4% Sn to ductile iron increased the tensile strength from 1130 to 1390 [MPa] after 150 minutes austempering at 285°C. In another instance, it can be seen that, addition of 1% Cu-0.2% Sn to ductile iron increased the tensile strength from just over 800 [MPa] to more than 1200 [MPa] after austempering for only 15 minutes at 335°C. Although a general rule cannot be established, alloying elements of copper and tin showed a significant improvement in the hardness of the specimens austempered at lower temperatures of 335°C and 285°C, for a short period of time. Regardless of austempering time and temperature used, the highest elongation-to-fracture among the 6 ductile iron alloys investigated, belongs to the alloy containing 1.5% Cu-0.2% Sn.

5. In some cases, the addition of alloying elements of copper and tin to base ductile iron has improved the impact strength to a limited degree. However, within the ranges of austempering time and temperature employed, the impact energy for all alloys was quite low. This became more pronounced as the tin content was increased.

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