Effect of Austenitizing Conditions on the Impact Properties of an Alloyed Austempered Ductile Iron of Initially Ferritic Matrix Structure

M. Delia, M. Alaalam, and M. Grech

The effect of austenitizing conditions on the microstructure and impact properties of an austempered ductile iron (ADI) containing 1.6% Cu and 1.6% Ni as the main alloying elements was investigated. Impact tests were carried out on samples of initially ferritic matrix structure and which had been first austenitized at 850, 900, 950, and 1000 °C for 15 to 360 min and austempered at 360 °C for 180 min.

Results showed that the austenitizing temperature, T_γ, and time, t_γ, have a significant effect on the impact properties of the alloy. This has been attributed to the influence of these variables on the carbon kinetics.

The impact energy is generally high after short t_γ, and it falls with further soaking. In samples austenitized at 850 and 900 °C, these trends correspond to the gradual disappearance of the pro-eutectoid ferrite and the attainment of fully developed ausferritic structures. In initially ferritic structures, the carbon diffusion distances involved during austenitization are large compared to those in pearlitic structures. This explains the relatively long soaking periods required to attain fully ausferritic structures, which in spite of the lower impact energy values, have a better combination of mechanical properties.

Microstructures of samples austenitized at 950 and 1000 °C contain no pro-eutectoid ferrite. The impact properties of the former structures are independent of t_γ, while those solution treated at 1000 °C are generally low and show wide variation over the range of soaking time investigated.

For fully ausferritic structures, impact properties fall with an increase in T_γ. This is particularly evident at 1000 °C. As the T_γ increases, the amount of carbon dissolved in the original austenite increases. This slows down the rate of austenite transformation and results in coarser structures with lower mechanical properties. Optimum impact properties are obtained following austenitizing between 900 and 950 °C for 120 to 180 min.

1. Introduction

The steadily increasing interest in the study and development of austempered ductile irons (ADI) can be attributed to the remarkable combination of mechanical properties (Ref 1-4) resulting from the unique microstructure (Ref 5, 6). The latter consists of acicular ferrite in a matrix of high carbon austenite and is frequently referred to as ausferrite (Ref 7).

In ADI, the properties attained are dependent on heat treatment parameters, initial matrix structure, and chemical composition. Work by Grech (Ref 8) has investigated the effect of austenitizing temperature, austempering temperature, and time on the mechanical properties of the alloy being studied.

Other work (Ref 9) has shown that the austenitizing conditions (T_γ and t_γ) have a significant influence on the microstructure and impact properties of the alloy with an initially pearlitic matrix structure.

The effect of the same variables on the microstructure and impact properties of samples with an initially ferritic matrix

structure have been studied. Also in progress is the study of the influence of these heat treatment parameters and matrix structure on other mechanical properties, namely, fatigue and tensile strength.

2. Experimental Procedure

The material under investigation and having chemical composition shown in Table 1 was produced from a 200 kg charge of low Mn good quality steel scrap, high purity graphite, ferrosilicon, copper, and nickel melted in a medium frequency induction furnace. The melt was superheated to 1520 °C to ensure homogenization and tapped into a ladle at 1450 °C onto 1.8% MgFeSi containing 75% silicon and some cerium. After

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Nomenclature	
T_γ	Austenitizing temperature
t_γ	Austenitizing time
T_A	Austempering temperature
t_A	Austempering time
D	Diffusion coefficient
x_{rms}	Diffusion distance

inoculation with 0.6% FeSi, the melt was cast at 1400°C into keel blocks of dimensions $300 \times 200 \times 30$ mm. The as-cast pearlitic matrix is shown in Fig. 1(a).
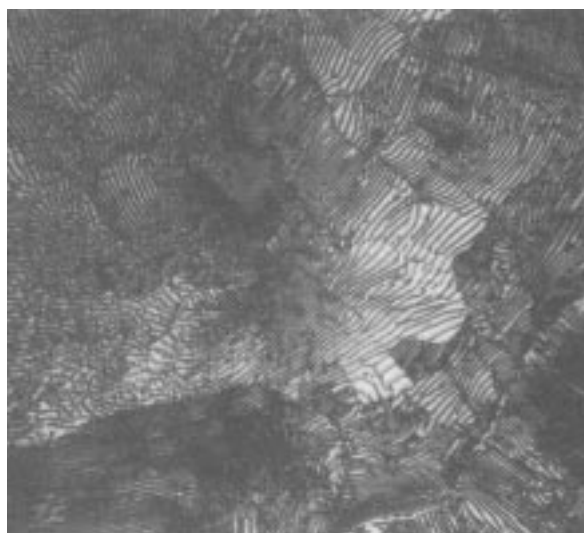
Prior to machining, keel blocks were annealed to produce the ferritic matrix structure. The resulting microstructure contained a ferritic matrix with some spheroidized carbides as shown in Fig. 1(b).

Unnotched Izod impact test specimens of dimensions $11 \times 11 \times 126$ mm were machined from the ferritized keel blocks and austenitized in an electric furnace held at 850, 900, 950, and 1000 °C. For each solution treatment temperature, six soaking periods were employed, which varied from 15 to 360 min. Specimens were subsequently austempered for 180 min in a salt bath held at 360 °C. The various heat treatment conditions used are listed in Table 2. After austempering, test pieces were air cooled to room te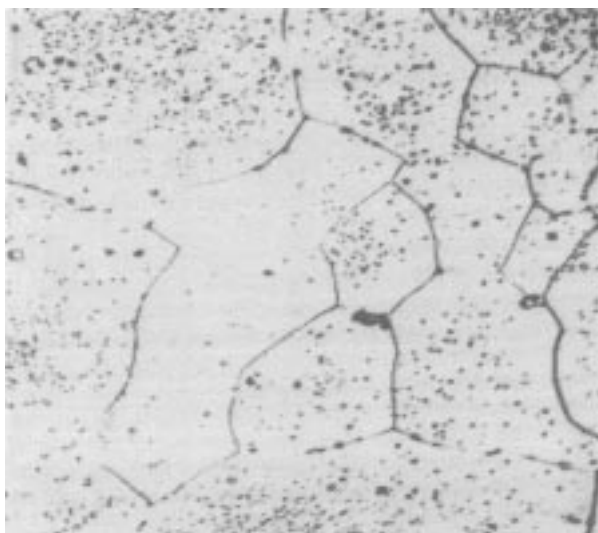mperature and ground to a 10×10 mm square section to remove any decarburized skin that may have formed during heat treatment. Care was taken during machining to avoid any stress-induced transformation of retained austenite to martensite.

Impact testing was carried out at room temperature using an Izod machine (Avery Ltd., Birmingham, England) with a 162 J capacity hammer and a striking velocity of $3.9/ms^{-1}$. At least three samples were tested for each heat treatment condition, yielding nine impact energy values.

Samples for metallographic study were taken from fractured impact specimens and polished using standard metallographic techniques. The volume fraction of graphite was measured on unetched polished specimens. Other samples were etched in 2% Nital and studied using an image analyzer. Some of the specimens were heat tinted (Ref 10) to help identify the various microstructural phases present.

Table 1   Composition of the keel blocks determined spectrographically

Element	Composition, %
C	3.3(a)
Si	2.6
Mn	0.35
S	0.008
P	0.01
Mg	0.04-0.05
Ni	1.6
Cu	1.6

(a) Assessed by the Leco method

Table 2   Heat treatment conditions

Austenitizing conditions	
Temperature, °C	Time, min
850	15
	30
	60
	120
	180
	360
900	15
	30
	60
	120
	180
	360
950	15
	30
	60
	120
	180
	360
1000	15
	30
	60
	120
	180
	360

Austempering conditions for all austenitizing conditions were 360 °C for 180 min.

(a)

(b)

Fig. 1   (a) As-cast fully pearlitic structure. 1000×. (b) Annealed ferritic structure. 1000×

3. Results

Annealed keel blocks have a ferritic matrix structure and contain some spheroidized carbides and graphite spheres. The latter have a nodularity in excess of 90% and an average diameter of 0.045 mm. The graphite nodules represent a volume fraction of 14% and have an average count of 75/mm^{-2}.

3.1 The Influence of Austenitizing Temperature

Figure 2 illustrates the influence of austenitizing temperature on the impact properties of samples solution treated between 850 and 1000 °C for 180 min and austempered at 360 °C for the same time period.

Impact energy values fall slightly as the solution treatment temperature increases from 850 to 950 °C and more rapidly with further increase in austenitizing temperature.

Specimens austenitized at 850 °C have microstructures containing a high volume fraction of pro-eutectoid ferrite, some acicular ferrite, and high carbon austenite (Fig. 3a). Increasing the T_γ to 900 °C and higher temperatures eliminates the pro-eutectoid ferrite and yields a structure containing ausferrite; see Fig. 3(b) and (c). Increasing the soaking temperature further to 1000 °C results in coarser and at times blocky austenite grains with martensite at their centers (Fig. 3d).

3.2 The Influence of Austenitizing Time

The effect of t_γ on the impact properties of specimens austenitized at 850, 900, 950, and 1000 °C is shown in Fig. 4. The impact properties are relatively high following short soaking periods and fall gradually with extended holding at temperature. The trend is more abrupt in the case of samples austenitized at 1000 °C.

Figure 5(a) shows that the microstructure of specimens austenitized for 15 min at 850 °C contains some acicular ferrite, high carbon austenite, and a large volume fraction of pro-eutectoid ferrite. As shown in Fig. 5(b), the amount of pro-eutectoid ferrite decreases with soaking time. Impact energy values of samples austenitized at 850 °C lie between 100 and 120 J.

Specimens soaked at 900 °C for periods between 15 and 30 min have microstructures containing acicular ferrite, high carbon austenite, and relatively large volumes of pro-eutectoid ferrite (Fig. 6a). Further holding at temperature yields fully ausferritic structures (Fig. 6b). Corresponding impact energy values are between 95 and 120 J.

Samples soaked at 950 °C for different t_γ have impact energy values that lie between 95 and 100 J. The resulting microstructure is shown in Fig. 3(c) and is relatively independent of t_γ.

Specimens austenitized at 1000 °C and over the entire range of soaking periods investigated have generally low impact energy values and exhibit different trends compared to those soaked at lower temperatures. The impact energy values fall from 85 to 30 J as t_γ increases from 15 to 60 min. Further holding increases the toughness to 65 J. The microstructure of specimens soaked for a short duration, namely 15 min, is shown in Fig. 7(a) and contains acicular ferrite in a high carbon austenite matrix. Microstructures of samples austenitized for longer periods are relatively coarse and contain martensite (Fig.7b).

4. Discussion

4.1 Effect of Austenitizing Temperature

Samples austenitized between 850 and 1000 °C for 180 min and austempered at 360 °C for the same period were selected to demonstrate the effect of T_γ on the microstructure and impact properties of the alloy under investigation.

Generally, trends are somewhat similar to those reported for samples of an initially pearlitic matrix structure (Ref 9). There are, however, some significant differences, particularly at 850 °C.

Microstructure. In a nodular iron with an as-cast pearlitic structure, the graphite spheroids and pearlite both contribute to the carbon enrichment of the mother austenite (Ref 11). In a fully ferritic matrix structure, however, the graphite nodules are the only source of carbon, and consequently, the carbon diffusion distances involved during solution treatment may be relatively large. This is nearly true because very little carbon can be attained from the small quantities of spheroidized carbides present. Consequently, full austenitization requires either very long solution treatment cycles or a very high carbon diffusion rate, which in turn, calls for a high austenitizing temperature. At 850 °C the carbon mobility is rather slow, and the soaking time selected is not sufficient for complete austenitization to take place. Consequently as shown in Fig. 3(a), these structures contain large volumes of pro-eutectoid ferrite.
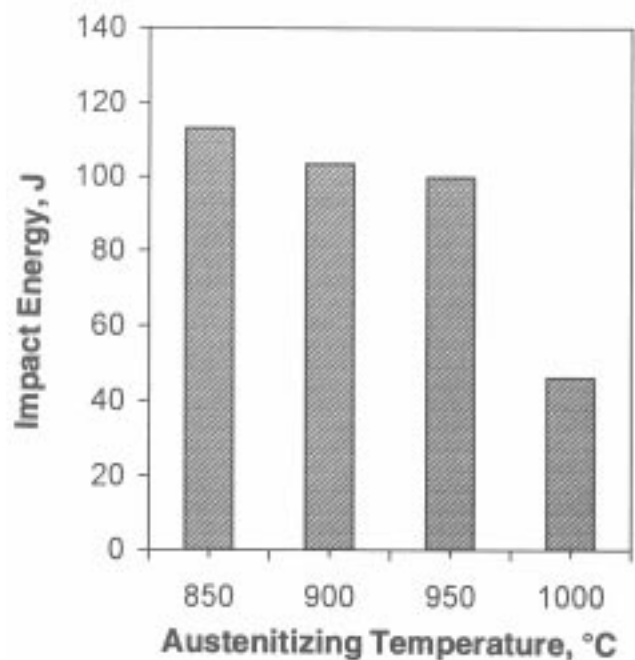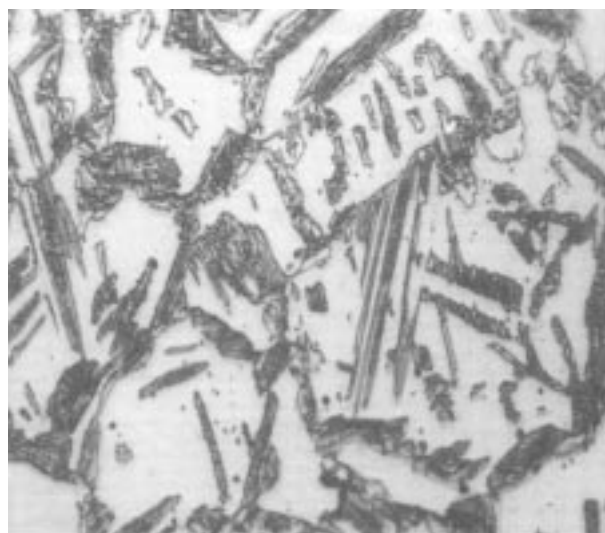

Fig. 2 Effect of austenitizing temperature on the impact properties of specimens austenitized for 180 min

Figures 3(b) and (c) show that samples austenitized at 900 and 950 °C contain acicular ferrite surrounded by high carbon austenite. The absence of pro-eutectoid ferrite and martensite can respectively be attributed to full austenitization and to the resulting stable high carbon austenite.

It is apparent from Fig. 8 (Ref 12) that, as the austenitizing temperature increases from T_1 to T_2, the percentage of carbon dissolved in the austenite also increases. This, in turn, lowers the driving force controlling the transformation of austenite to ausferrite from a′b′ to ab. The driving force reduction is responsible for the decrease in the number of ferrite nuclei formed and the slower growth along the ferrite platelet. Therefore, as shown in Fig. 3(d), increasing the austenitizing temperature to 1000 °C leads to structures containing a high percentage of large austenite grains. The center of these regions is low in car-

bon content and is therefore relatively unstable. It transforms to martensite as the specimens cool to room temperature or upon the application of mechanical stress.

Image analysis measurements on specimens austenitized at 1000 °C show austenite grains with diameters up to 28 µm. Conversely, in specimens austenitized at lower temperatures, the distance between adjacent ferrite plates may be less than 2 µm. In the former case, therefore, the carbon rejected from the growing ferrite needles has longer diffusion distances to travel during the austempering reaction for all the austenite matrix to be fully enriched in carbon. The rate of the competing processes of ferrite nucleation and growth and carbon enrichment of the austenite may not allow enough time for the austenite to attain a sufficient percentage of carbon to be stable.
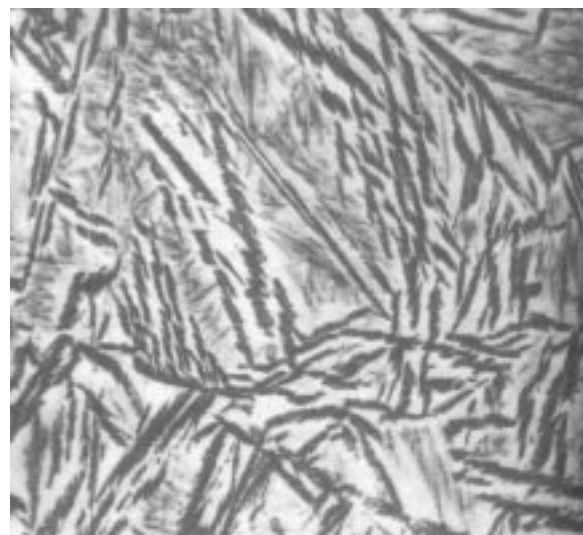

(a)
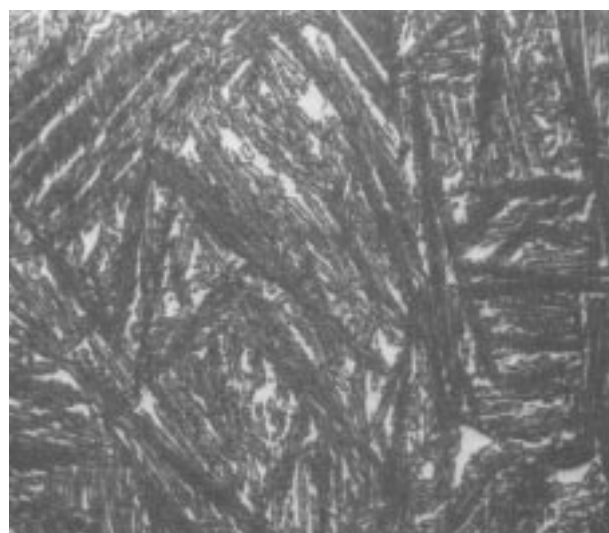
(b)
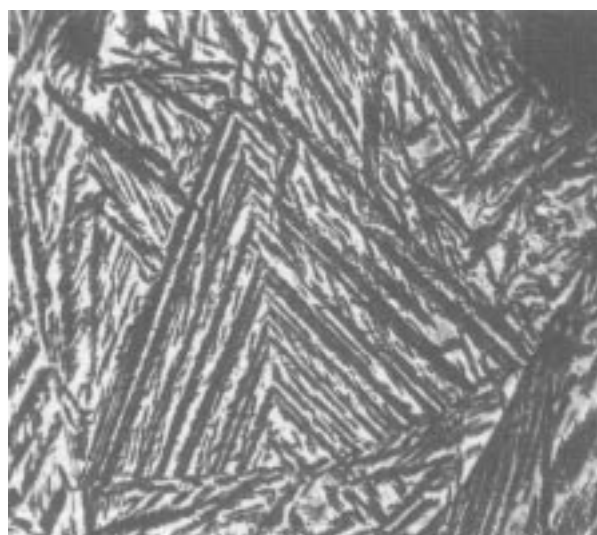
(c)

(d)

Fig. 3 Effect of austenitizing temperature on the microstructure of specimens austenitized for 180 min at (a) 850, (b) 900, (c) 950, and (d) 1000 °C. 1000×

Assuming that the diffusion coefficient, D, is given by the relationship:

$$D = 0.25 \exp -34500/RT_A$$

and that the diffusion distance, x_{rms}, is given by:

$$x_{rms} = (2Dt_A)^{1/2}$$

and taking the austempering temperature, T_A, to be 360 °C (633 K) and the austempering time, t_A, as 180 min, then:

$$x_{rms} = 0.73 \ \mu m$$

Austenite grains with diameters of $< 1.5 \ \mu m$ should theoretically contain the equilibrium carbon content; conversely, the center of larger austenite grains would contain less carbon and may therefore be unstable. Some equiaxed austenite grains in samples austenitized at 1000 °C have diameters up to 20 times larger than the diffusion distance calculated above. These regions may, upon cooling to room temperature, transform to martensite. The segregation of manganese to the intercellular regions may slow the diffusion of carbon and hinder further the attainment of an equilibrium carbon content in the austenite.

To summarize, increasing T_γ from 850 to 1000 °C eliminates the pro-eutectoid ferrite and increases the austenite volume fraction. The latter, however, has a lower carbon content, is less stable, and may transform to martensite on cooling to room temperature or upon the application of stress.

Impact Properties. Figure 2 shows the effect of T_γ on the impact properties of the ductile iron under investigation. Specimens austenitized at 850 °C have the highest impact energy value, namely 113 J. This has been attributed to the large volume fraction of the pro-eutectoid ferrite and the morphology of the acicular ferrite. These results support findings claimed by Shih et al. (Ref 13) but contradict those by Kovacs (Ref 14). Results derived here may lead to the erroneous conclusion that pro-eutectoid ferrite is a beneficial phase. Hughes (Ref 15) has shown that ausferrite has a considerably better combination of mechanical properties than ferrite or any other matrix structure found in ductile iron. Thus, although the structure shown in Fig. 3(a) has a very high impact energy, its tensile strength may be as low as half that of the corresponding ausferritic structure.
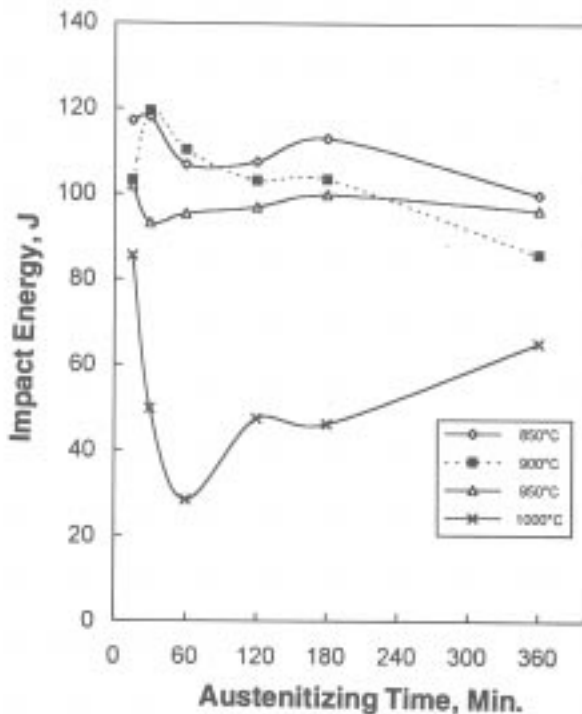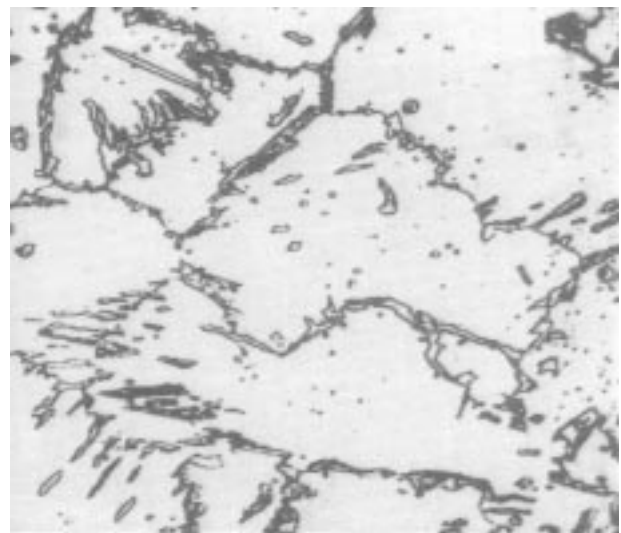

(a)

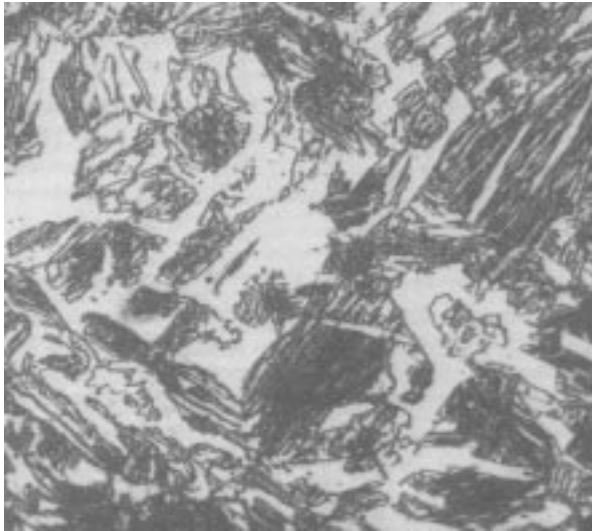**(b)**

Fig. 5 Effect of austenitizing time on specimens austenitized at 850 °C for (a) 15 and (b) 360 min. 1000×

Fig. 4 Effect of austenitizing time on impact energy of specimens austenitized at 850, 900, 950, and 1000 °C
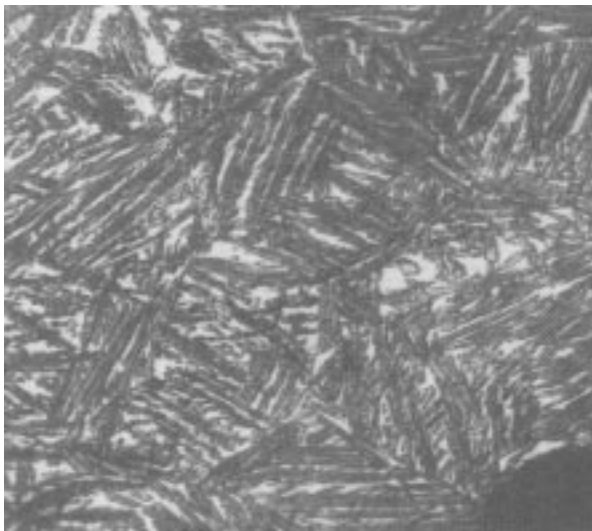
(a)

(a)

(b)

(b)

Fig. 6 Effect of austenitizing time on specimens austenitized at 900 °C for (a) 15 and (b) 60 min. 1000×

Fig. 7 Effect of austenitizing time on specimens austenitized at 1000 °C for (a) 15 and (b) 60 min. 1000×
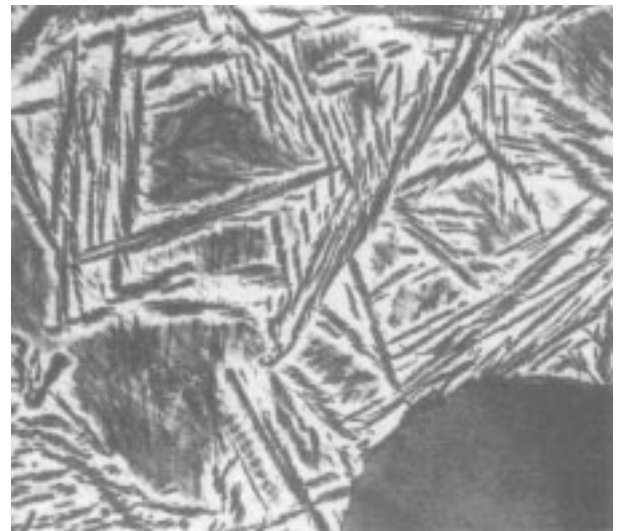
Specimens solution heat treated at 900 and 950 °C have a fully austempered structure and relatively high impact energy values. These structures have marginally lower toughness values compared to those shown in Fig. 3(a) but a better combination of mechanical properties.

In contrast, specimens austenitized at 1000 °C, have a much lower impact energy value, namely 46 J. This is attributed to the higher percentage of low carbon austenite and martensite (Fig. 3d).

Work carried out by Grech (Ref 8) shows that the percentage of austenite may have a major beneficial influence on the impact energy and ductility values. However, caution is required as this assumes that the austenite contains a high carbon content and is stable. In this respect, it is more appropriate to use the product of austenite volume and carbon content within the austenite as an indication of mechanical properties, rather than just the austenite volume fraction. Work by H. Bayati and R. Elliott (Ref 16) confirms this and shows that irons austempered at 375 and 400 °C and having the same austenite content, that is 40%, had widely varying elongations, namely 5.7 and 2.8%, respectively.

When comparing structures, the morphology of the resulting austenite is also important (Ref 14); in fact, that formed between the acicular ferrite plates is relatively more stable and beneficial than the blocky type counterpart (Fig 3). It follows that the low impact energy values of the specimens austenitized at 1000 °C are due to the high volume fraction of low carbon austenite. The resulting austenite can easily transform to martensite on cooling to room temperature or upon the application of stress.

4.2 Effect of Austenitizing Time

Austenitizing time is the other heat treatment parameter investigated. It determines the percentage of carbon dissolved in the austenite, which in turn, affects the rate of austenite transformation during austempering.

Microstructure. Microstructures of specimens austenitized at the four temperatures being studied for durations between 15 and 360 min consist of acicular ferrite with varying proportions of retained austenite and in some cases pro-eutectoid ferrite or martensite. Figures 5(a) and 6(a) demonstrate that the microstructures of samples austenitized for short periods at 850 and 900 °C contain a considerable volume of pro-eutectoid ferrite. This phase is replaced by ausferrite as the soaking period extends to 360 min at 850 °C (Fig. 5b) or to 60 min at 900 °C (Fig. 6b).

In contrast, the microstructures of specimens austenitized at 950 °C for durations between 15 and 360 min consist generally of acicular ferrite and high carbon austenite. There are only marginal differences between microstructures of specimens soaked for different periods.

The microstructures of samples austenitized for 15 min at 1000 °C are fully ausferritic (Fig 7a). Increasing the soaking period to 60 min and further results in structures containing martensite, Fig. 7(b).

Impact Properties. Figure 4 shows the effect of t_γ on the impact properties of samples solution treated between 850 and 1000 °C. High impact properties in excess of 118 J are attained following austenitizing at 850 °C for 30 min. The Fe-C-2%Si equilibrium diagram in Fig. 8 shows that an austenitizing temperature of 850 °C is insufficient for full austenitization. In fact, pro-eutectoid ferrite occurred even in samples solution treated for as long as 180 min. It is apparent that the high impact energy values are due to the pro-eutectoid ferrite and not the ausferrite. Structures containing this type of ferrite would, however, have a low tensile strength compared to those with a fully ausferritic structure.

Other work by the present authors (Ref 9) shows that specimens with an initially pearlitic matrix and austenitized at 850 and 900 °C attain near optimum properties after 60 min at temperature. In contrast, in a ferritic matrix structure, significantly more time is required to reach an equilibrium carbon content in the mother austenite. This is attributed to the larger carbon diffusion distances and the presence of copper, which segregates to the graphite-metal interface and creates a carbon diffusion barrier.

As shown in Fig. 4, the impact energy values of samples solution treated at 900 °C fall from 120 to 103 J as t_γ increases to 180 min and change only marginally with further increases in the soaking time. The higher toughness values correspond to structures containing some pro-eutectoid ferrite. The fully austempered structures have impact values of ~100 J. In specimens austenitized at 950 °C, the impact energy values are relatively constant at ~100 J indicating that a high T_γ increases the carbon diffusion rate and leads to a rapid austenitization. This agrees with work done by Shea and Ryntz on nodular iron containing 2.88%Si (Ref 17). The slightly lower impact energy values as compared with test samples austenitized at lower T_γ can be attributed to the elimination of the pro-eutectoid ferrite phase as well as the lower rate of austenite transformation.
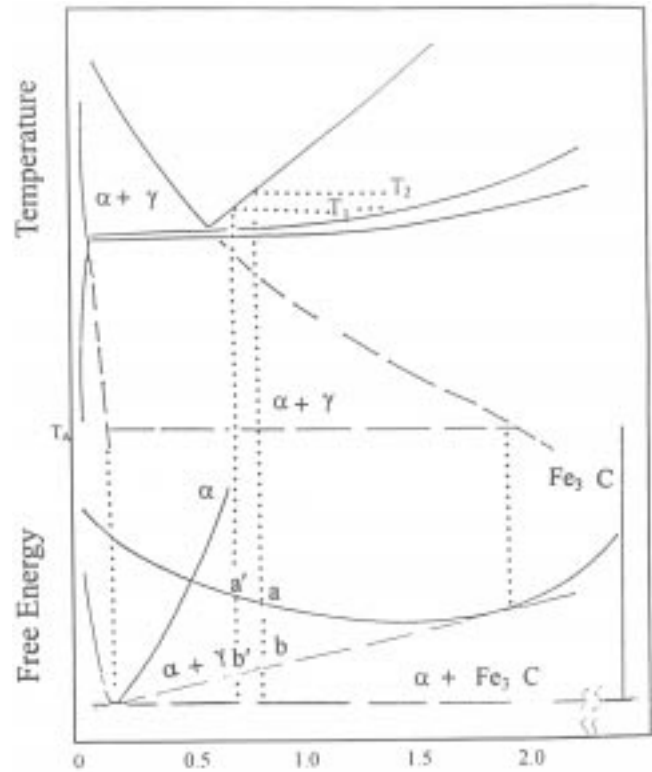

Fig. 8 A section of the Fe-C-Si phase equilibrium diagram with a free energy schematic of the austempering process (Ref 12)
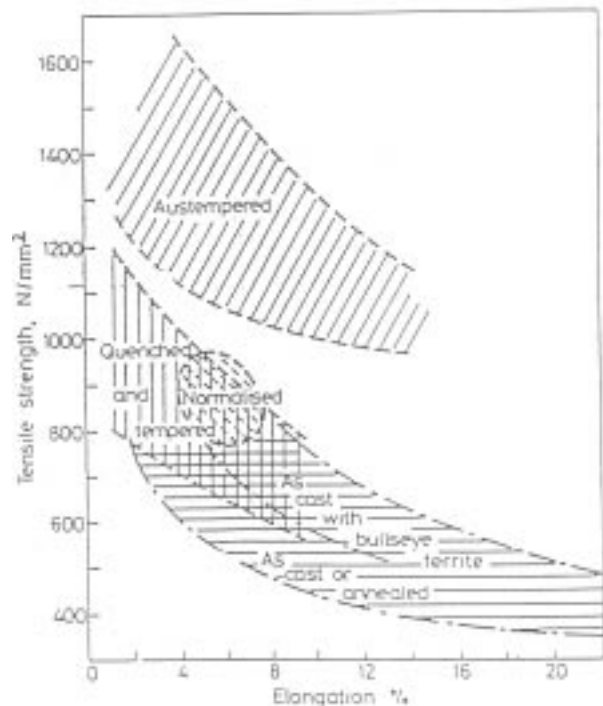

Fig. 9 Comparison of mechanical properties of ADI with other ductile irons (Ref 15)

Samples austenitized at 1000 °C for very short periods have impact energy values of approximately 90 J. The toughness falls rapidly as the solution treatment time increases. The high impact energy values can be attributed to the low content of dissolved carbon in the austenite and, consequently, the relatively high driving force controlling the austempering reaction. Increasing the soaking period to 60 min increases the dissolved carbon and results in a coarser structure containing martensite. It is not clear, however, why soaking for more than 60 min gives rise to a recovery in impact energy values.

5. Conclusions

The effect of austenitizing temperature and time on the impact properties of an austempered ductile iron alloyed with Cu and Ni and having an initially ferritic matrix structure has been studied. The following conclusions can be drawn:

- At 850 °C, complete austenitization is difficult to achieve. In fact, irons austenitized for up to 180 min still contain pro-eutectoid ferrite in the austempered structure.
- Increasing T_γ from 900 to 1000 °C increases the percentage of carbon dissolved in the original austenite, which in turn, decreases the free energy controlling the transformation of austenite to ferrite and high carbon austenite. Less ferrite nuclei are formed at 1000 °C, and the resulting structure is coarser and contains more blocky type austenite.
- The larger austenite grains contain less carbon, are less stable, and may transform to martensite upon cooling to room temperature. This has a negative influence on the impact properties.
- The austenitizing time determines the percentage carbon dissolved in the mother austenite and therefore, has an influence on the impact energy values attained.
- Specimens austenitized at 1000 °C for different periods have generally low impact energy values. This has been attributed to the large amount of low carbon austenite and the associated martensite.
- It has been shown that, for the Cu-Ni alloy investigated, optimum impact energy values are attained when austenitizing is carried out between 900 and 950 °C for 120 to 180 min. These austenitizing conditions are such as to eliminate the pro-eutectoid phase but at the same time do not reduce substantially the rate of austenite transformation; consequently, these optimum conditions do not promote the formation of martensite.
- For complete austenitization, initially ferritic structures require longer soaking periods than corresponding as-cast pearlitic structures. This is due to the larger carbon diffusion distances involved in the former structure. Furthermore, the diffusion of Cu to the graphite boundaries reduces the rate of diffusion of carbon into the surrounding matrix. Since graphite is the main source of carbon, this considerably impedes the austenitizing process.

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