



Development of Austempered Ductile Iron for High Tensile and Fracture toughness by Two Step Austempering Process.

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ABSTRACT

During conventional austempering austempered ductile iron showed a decrease in fracture toughness with increasing austempering temperature, while the tensile toughness increased. Thus high fracture toughness was associated with low tensile toughness. A two step austempering treatment was then adopted where the samples were first austempered at 300°C for short periods varying from 10 minutes to 60 minutes, and then subsequently transferred to a second furnace at 400°C for further austempering for 2 hours. It was found that this resulted in fine ferrite grain size, high carbon content of the retained austenite together with increased stability of the austenite. Under such conditions it was possible to achieve an excellent combination of high fracture toughness and high tensile toughness.

Key words: austempered ductile iron, fracture toughness, tensile toughness, retained austenite, strain induced martensite.

INTRODUCTION

Austempered ductile irons (ADI) are an interesting class of materials because of their unique microstructure and interesting properties. When subjected to austempering treatment ductile iron transforms to a microstructure consisting of ferrite and retained austenite rather than ferrite and carbide as in austempered steels. Because of the presence of retained austenite ADI exhibits excellent combination of strength and ductility, together with good fatigue and wear properties¹⁻⁴. It has been reported^{5,6} that ADIs exhibit similar fracture toughness as quenched and tempered medium carbon steels and low alloy steels. It has been reported by several investigators⁷⁻¹⁰ that ADIs austempered at low temperatures show higher fracture toughness than those austempered at higher temperatures. Since strength decreases and ductility increases with increasing austempering temperature, we have the interesting phenomenon where fracture toughness increases with increasing strength and decreasing ductility. Tensile toughness, as measured from the area under the stress - strain diagram, decreases with decreasing austempering temperature while the fracture toughness

increases. Analysis of the fracture toughness behaviour has shown⁹⁻¹⁰ that the fracture toughness can be related to the yield strength and some microstructural features by the following relationship:

$$K_{Ic}^2 = \sigma_{YS}(X_y C_y)^{1/2} \quad (1)$$

In this equation K_{Ic} is the fracture toughness, σ_{YS} is the yield strength, X_y is the volume fraction of the retained austenite and C_y is the carbon content of the retained austenite. At low austempering temperatures σ_{YS} is large and $X_y C_y$ is low. At high austempering temperatures σ_{YS} is low and $X_y C_y$ is large. Therefore in order to simultaneously maximize K_{Ic} and tensile toughness, σ_{YS} and $X_y C_y$ must be maximised simultaneously. This cannot be achieved by conventional austempering treatment. Hence a two step austempering treatment is attempted where the ductile iron is first austempered at 300°C for short durations and then austempered at a higher temperature such as 400°C for two hours. This results in a microstructure characteristic of the two temperatures. In the present investigation microstructure, tensile properties and fracture toughness are characterized under two step austempering treatment and the results are compared with those of conventional austempering treatment carried out over a range of austempering temperatures.

EXPERIMENTAL WORK

The ductile iron of the chemical composition reported in Table 1 was cast in the form of slabs of dimension 200 mm x 150 mm x 30 mm. Round tensile samples of diameter 6 mm and gauge length 42 mm, as well as three point bend specimens of dimensions shown in Fig. 1 for fracture toughness were machined from the cast slabs. All the samples were austenitised at 900°C for 30 minutes in a muffle furnace. Austempering was carried out in salt baths. Conventional austempering was carried out at 280, 300, 320, 350, 380 and 400°C for 2 hours. In two step austempering, two salt bath furnaces were used, one at 300°C and the other at 400°C. The austenitised samples were first transferred to the salt bath at 300°C and held at that temperature for 10, 20, 30, 45 or 60

Table 1
Chemical composition of ADI used in the present investigation

Element	C	Si	Mn	S	P	Mg	Ni	Mo	Cu
Wt. %	3.5	2.8	0.3	0.022	0.02	0.04	1.5	0.3	0.5

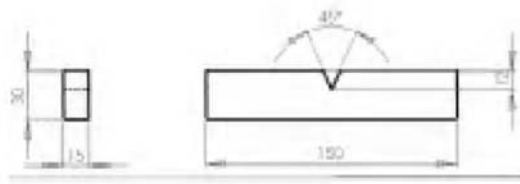


Fig. 1 : Dimensions of fracture toughness specimen in millimeter.

minutes. After the respective times, the samples were quickly transferred to the second furnace at 400°C and held at that temperature for 2 hours. The samples were then quenched in water.

For fracture toughness testing the samples were precracked in a laboratory designed cantilever loading machine. The precracked samples were then subjected to three point bending in a floor model Instron machine as per ASTM E399¹¹. It was confirmed in each case that valid K_{Ic} was obtained. Tensile tests were carried out as per ASTM E8¹² on the same Instron testing machine at a cross head speed of 1 mm per minute. Engineering stress strain curves were plotted and tensile toughness was estimated as the area under the stress strain curve. Both in fracture toughness and tensile test, three samples were tested for each heat treatment, and the results reported are an average of these three tests.

Qualitative microstructural studies were carried out through optical microscopy on both deformed and undeformed samples. To study the microstructures of undeformed samples, dummy samples 2 mm x 15 mm x 15 mm were heat treated along with the tensile samples, and their microstructures were studied. To study the effect of deformation, small samples were cut from the fractured samples at the fracture end. The fracture surface was polished, etched and studied under the optical microscope. X-ray diffraction studies were also carried out on samples similar to those of optical microscopy to elicit quantitative information such as volume fraction of retained austenite and its carbon content. These followed the well defined procedure as detailed in literature¹³.

RESULTS AND DISCUSSION

Typical microstructures of samples subjected to conventional austempering are shown in Fig. 2. Retained austenite content and its carbon content are presented in Fig. 3 as a function of austempering temperature. These match well with the results

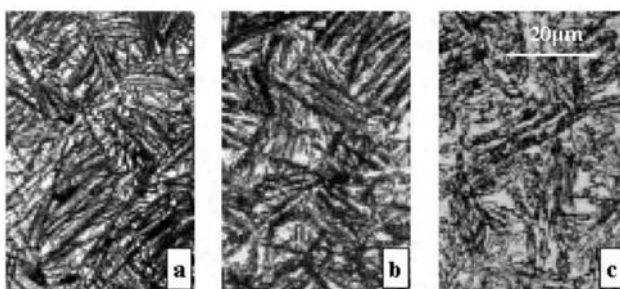


Fig. 2 : Microstructure of the samples austempered at a) 300°C, b) 350°C and c) 400°C

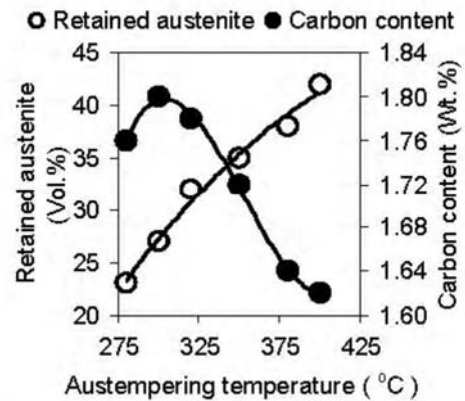


Fig. 3 : Retained austenite and carbon content for conventional austempering

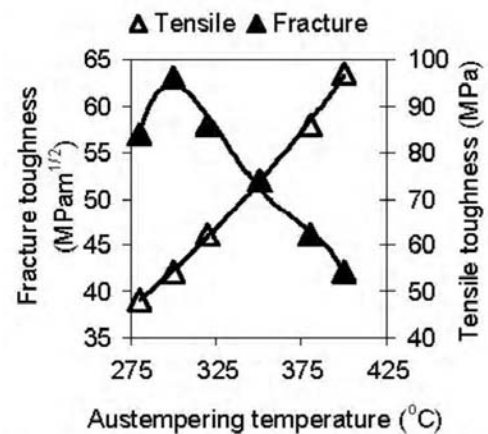


Fig. 4 : Tensile and fracture toughness for conventional austempering

of other investigators^{13,14} and can be explained on the same basis. Fig. 4 shows the influence of austempering temperature on the fracture toughness as well as the tensile toughness as measured from the area under the stress - strain curve. As explained by previous investigators⁷⁻¹⁰ it was found that the fracture toughness was high at low austempering temperatures while the tensile toughness was low. At high austempering temperatures the fracture toughness was low while the tensile toughness was high. The results of the conventional austempering with regard to microstructure and fracture toughness and tensile toughness in the present investigation are thus in conformity with those of the previous investigators⁷⁻¹⁰.

The microstructures of two step austempering are presented in Fig. 5. At first step time of 30 minutes the microstructure was predominantly upper bainitic with a small amount of lower bainite constituent as shown in Fig. 5a. At the first step time of 60 minutes the microstructure was predominantly lower bainitic as shown in Fig. 5c. At the first step time of 30 minutes the microstructure consisted of both upper and lower bainitic constituents as shown in Fig. 5b. Thus it was found that the upper bainitic constituent decreased in volume fraction while the lower bainitic constituent increased, as the first step time was increased from 10 minutes to 60

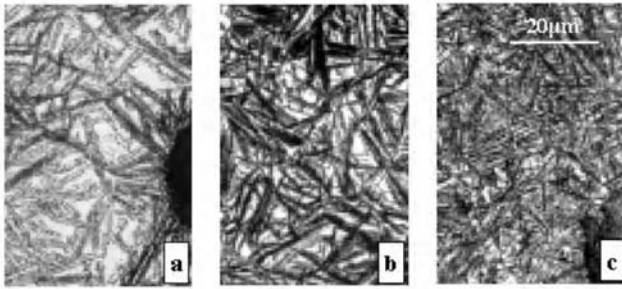


Fig. 5 : Microstructures of the samples subjected to two step austempering at the first step time of a) 10 min, b) 30 min and c) 60 min

minutes. At the first step time of 10 minutes only a small fraction of the austenite underwent bainitic transformation at 300°C. The remaining untransformed austenite underwent the bainitic transformation at the upper temperature of 400°C. Thus we observe a large fraction of upper bainite and only a small fraction of lower bainite. At first step time of 60 minutes, sufficient time was available at 300°C for substantial amount of austenite to transform to bainite. Only a small fraction of the original austenite which remained untransformed underwent bainitic transformation at the higher temperature. Thus we observe a large volume fraction of lower bainite and only a small volume fraction of upper bainite. Thus the two step austempering results in a microstructure which is a mixture of microstructures of conventional annealing at the two temperatures. Relative volume fraction of each depends on the first step time.

The quantitative information on the microstructures is also quite interesting. It is presented in Fig. 6, which shows the influence of the first step time on the volume fraction of the retained austenite as well as its carbon content. Retained austenite content was found to drop from about 38 vol. % at 10 minutes to about 21 vol. % at 60 minutes. The carbon content of the retained austenite was about 0.65 wt. % at 10 minutes and about 0.43 wt. % at 60 minutes. Comparison of these with Fig. 3 for conventional austempering corroborates the earlier statement that the microstructure at first step time of 10m in utes was close to that of conventional

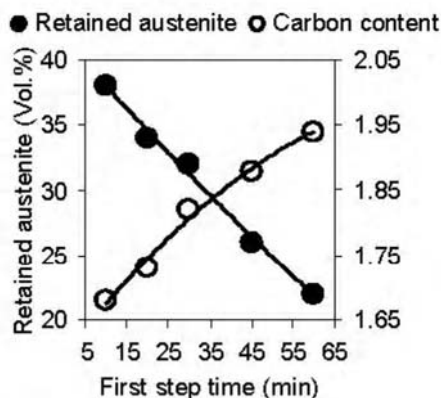


Fig. 6 : Retained austenite and carbon content for two step austempering.

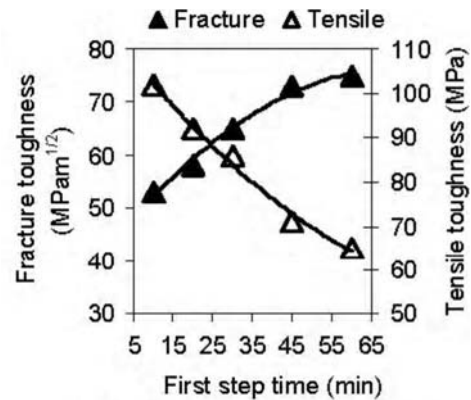


Fig. 7 : Tensile and fracture toughness for two step austempering.

austempering, while that of first step time of 60 minutes was similar to that of conventional austempering at 400°C.

The results of fracture toughness testing and tensile testing are shown in Fig. 7. Fracture toughness was found to increase with increasing first step time from 52 MPa√m at 10 minutes to 78 MPa√m at 60 minutes. Simultaneously, the tensile toughness dropped from 105 MPa to 65 MPa. On comparing these results with those of conventional austempering presented in Fig. 4, remarkable improvement in tensile toughness can be observed at similar levels of fracture toughness. For example, during conventional austempering the maximum fracture toughness obtained was 63 MPa√m, at a corresponding tensile toughness of 53 MPa. In two step austempering the same fracture toughness would correspond to a considerably higher tensile toughness of 87 MPa.

Tensile properties of conventional austempering and two step austempering are presented in Tables 2 and 3 respectively, along with their fracture toughness values. For similar fracture toughness values, two step austempering results in better combination of tensile strength and ductility.

Improved toughness results from reduced ferrite grain size, increased carbon content of the retained austenite and its stability. Ferrite grain size was determined through x-ray diffraction using Scherrer formula ¹⁵:

$$t = 0.9\lambda/B.\cos\theta_s \quad (2)$$

where t is the ferrite particle size, λ is the wavelength of the x-ray diffraction, B is the width of the ferrite peak at half intensity and θ_s is the Bragg angle. Values of the ferrite particle sizes so determined under different heat treatment conditions are presented in Tables 4 and 5 for the conventional and two step treatments respectively. The fracture toughness was found to increase with decreasing ferrite particle size under both types of heat treatments. Under conventional treatment, ferrite particle size was generally less than 0.2 μm for fracture toughness greater than 60 MPa√m. However, in the case of two step treatment, much coarser ferrite particle size of 0.62 μm resulted in fracture toughness values better than 60 MPa√m.

Carbon content of the retained austenite is an important parameter affecting the toughness. Under conventional

**Table 2**

Tensile properties of the samples for conventional austempering.

Austempering temperature (°C)	Yield strength (MPa)	Tensile strength (MPa)	Elongation (%)	Tensile toughness (MPa)	Fracture toughness (MPa ^{1/2})
280	1277	1468	1.5	48	56
300	1266	1390	2.3	54	63
320	1245	1320	3.3	62	58
350	1136	986	5.6	74	52
380	669	964	8.8	86	46
400	488	865	10.7	97	42

Table 3

Tensile properties of the samples for two step austempering.

First step time (min)	Yield strength (MPa)	Tensile strength (MPa)	Elongation (%)	Tensile toughness (MPa)	Fracture toughness (MPa ^{1/2})
10	662	1090	10.6	102	53
20	794	1209	8.5	92	58
30	846	1256	5.4	86	65
45	934	1389	3.9	71	73
60	1008	1418	2.9	65	75

treatment this had a maximum value of nearly 1.8 wt. % at the austempering temperature of 300°C. At other temperatures, the carbon content was lower, and fracture toughness values were all less than 60 MPa^{1/2}m. Under two step treatment, as seen from Fig. 6, it was greater than 1.8 wt. % at short durations of first step time upto 30 minutes. Under all these conditions, fracture toughness was greater than 60 MPa^{1/2}m, and increased with increasing carbon content. At the maximum fracture toughness of 74 MPa^{1/2}m, the carbon

content was 1.94 wt. %. The carbon content is important as it increases the strain hardening ability of the austenite. Strain hardening coefficient increases with increasing carbon content of the austenite. This increases the load bearing capacity of the material as the cross sectional area is reducing during plastic deformation, and delays the onset of necking. Thus toughness can increase with increasing carbon content of the retained austenite. Increased carbon content during two step austempering can be attributed to the long holding at the higher temperature of 400°C after the transformation at the lower temperature for short durations. It is believed that ferrite formed at the first step temperature traps the carbon in the transforming austenite region. This carbon may be present within the ferrite or more likely at the ferrite/austenite interface. Epsilon and other transition carbides often occur in lower bainite in high carbon high silicon steels^{16, 17} as well as in AD1^{18,19}. Detection of these carbides in lower bainite is important since it indicates that part of the carbon is not available for partitioning into the residual austenite. The presence of transition carbides also implies that high carbon concentration is retained in solution in ferrite. Roberts²⁰ has reported a stable dissolved carbon to a level of nearly 0.25 wt. % during precipitation of E carbide. In situ study of precipitation of carbides within ferrite using hot stage electron microscopy has indicated²¹ a supersaturation of carbon to the extent of 0.3 wt.% in ferrite in high silicon steels during bainite transformation at 310°C. Holding at the second step temperature allows this carbon

Table 4

Ferrite particle size for conventional austempering.

Austempering temperature(°C)	280	300	320	350	380	400
Ferrite particle size (µm)	0.1	0.18	0.28	0.45	0.8	1.2

Table 5

Ferrite particle size for two step austempering.

First step Austempering time (min)	10	20	30	45	50
Ferrite particle size (µm)	1.15	0.81	0.32	0.23	0.12

to diffuse into the surrounding austenite. Thus the carbon content of the retained austenite is generally higher under the two step treatment.

Stability of the retained austenite is the third important parameter which controls the toughness of the ADI. Formation of strain induced martensite is known to enhance the toughness in TRIP steels. Since the retained austenite in ADI has high carbon content, and the martensite formed in it will be brittle, it is debatable if formation of strain induced martensite in these can improve the toughness. It should be noted that TRIP steels are designed to have a carbon content of not more than 0.2 wt. % so that the martensite formed is not brittle. The contribution of strain induced martensite to the toughness of ADI has been the subject of a recent investigation²². It has been argued that the martensite containing ADI can be visualized as fiber reinforced ductile material. Even if the fiber is brittle, it will not make the composite brittle, as long as the fiber length is less than the critical length, since then, it will not be loaded to its maximum strength, and therefore will not fracture. According to this argument if the retained austenite regions are not massive, then, only very short lengths of thin martensite will form, and will improve the toughness of the ADI.

Thus, ADI with lower bainite in which the microstructure basically consists of thin ferrite and austenite regions should benefit from the martensite formation, while that with upper bainite consisting of massive retained austenite together with coarse ferrite will not. The study of the formation of strain induced martensite in ADI has shown²³ that retained austenite in upper bainite can easily transform to martensite. Too low stability of austenite is detrimental to the toughness of steel.

The amount of retained austenite was estimated in the samples which have undergone deformation. For this purpose, the samples cut from the fracture surface of tensile samples were studied under optical microscope as well as through x-ray diffraction. The results of optical microscopy are shown in Figs. 8 and 9, while those of x-ray diffraction are shown

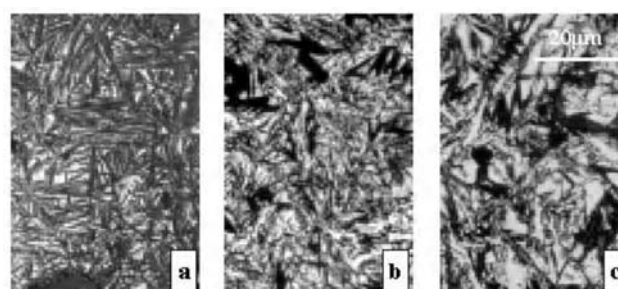


Fig. 8 : Microstructures on the fracture surfaces of the samples austempered at a) 300°C, b) 350°C and c) 400°C

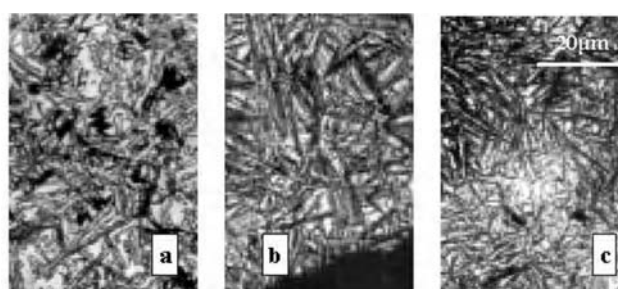


Fig. 9 : Microstructures on the fracture surfaces of the samples subjected to two step austempering at the first step times of a) 10 min, b) 30 min and c) 60 min.

in Tables 6 and 7. These indicate that during conventional austempering, copious formation of martensite occurs in samples austempered at higher temperatures such as 350°C and above. Similarly, in the case of two step austempering, considerable martensite formation is observed at short durations of first step austempering, like 10 and 20 minutes.

All the three parameters cited above, namely, fine ferrite grain size, high carbon content of retained austenite, and high stability of retained austenite are achieved in two step austempering at longer first step times of 30 minutes and more. As a result, high fracture toughness together with high tensile toughness is achieved in these cases. The best fracture toughness obtained in two step austempering is about 24% higher than that obtained through conventional austempering, together with an improvement in tensile toughness of 47%.

CONCLUSIONS

1. Two step austempering results in a microstructure consisting of a mixture of upper and lower bainite, the relative proportions of which depends on the first step time. The retained austenite content decreases with increasing first step time, while the carbon content of the retained austenite increases.
2. The tensile and yield strengths increase with increasing first step time, while the ductility and tensile toughness decrease.
3. The fracture toughness increases with increasing first step time.
4. The best fracture toughness of 78 Mpa√m obtained

Table 6

Retained austenite after deformation for conventional austempering.

Austempering temperature(°C)	280	300	320	350	380	400
Retained austenite (Vol.%)	22	24	28	27	27	26

Table 7

Retained austenite after deformation for two step austempering.

First step Austempering time (min)	10	20	30	45	50
Retained austenite (Vol.%)	1.15	0.81	0.32	0.23	0.12



through twostep austempering is 24% higher than that obtained through conventional austempering.

5. Strain induced martensite forms during deformation of ADI if the microstructure primarily consists of bulky austenite with low carbon content.
6. The improved fracture toughness obtained through two step austempering can be attributed to fine ferrite particle size, increased carbon content of retained austenite, and the increased stability of retained austenite.
7. A combination of high fracture toughness and high tensile toughness can be achieved by adopting two step austempering process.

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