J Mater Sci (2008) 43:4929–4937
DOI 10.1007/s10853-008-2717-8

Influence of austenitising temperature on the formation of strain induced martensite in austempered ductile iron

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Received: 15 July 2007 / Accepted: 12 May 2008 / Published online: 30 May 2008

Abstract The present work was taken up to study the influence of austenitising temperature on the formation of strain-induced martensite in austempered ductile iron. Ductile iron containing 1.5 wt.% nickel, 0.3 wt.% molybdenum and 0.5 wt.% copper was subjected to austempering treatments which consisted of three austenitising temperatures, namely 850, 900 and 950 °C, and three austempering temperatures, namely 300, 350 and 400 °C. Tensile tests were carried out under all the heat-treatment conditions and strain-hardening behaviour was studied by applying Hollomon equation. Microstructures were studied by optical microscopy and X-ray diffraction. It was found that increasing austenitising temperature increased the tendency for the formation of strain-induced martensite at all the austempering temperatures.

Introduction

Austempered ductile iron (ADI) is an interesting engineering material because of its unique microstructure and unusual combination of high strength and high ductility. Unlike in the case of steels, the austempering treatment in ductile iron results in a microstructure consisting of ferrite and austenite rather than ferrite and carbide because of the high silicon content. The excellent properties, such as high tensile and fracture toughness, excellent wear resistance and fatigue properties are related to the presence of large volume fraction of austenite [1–3]. In general it is believed that austenite exhibits high-strain hardening tendency. It is also believed that austenite can transform to martensite under strain, and that a TRIP like phenomenon may be responsible for some of these properties [4, 5]. The ability of austenite to transform to martensite under strain depends on several factors [6]. Some of these are the carbon content of the austenite, its size and morphology, and its distribution within the microstructure. All these parameters can be varied by varying the heat treatment parameters such as austenitising temperature and time as well as the austempering time and temperature. It has been shown [7] that high austempering temperature generally results in a microstructure consisting of retained austenite with low stability. Such a retained austenite will undergo transformation to martensite relatively easily when strained as compared to austenite in an ADI austempered at low temperatures. The importance of the microstructural features in determining the stability of the austenite has been shown [7] by subjecting the ADI to a two-step austempering. This results in a microstructure consisting of a combination of lower and upper bainites. Greater the amount of upper bainite in the microstructure, higher will be its tendency to undergo transformation to strain-induced martensite. The present work is taken up to study the influence of austenitising temperature on the stability of retained austenite and on the formation of strain-induced martensite.

Experimental work

Ductile iron having a composition shown in Table 1 was cast as slabs of dimension 200 mm × 150 mm × 75 mm. Coupons of 2 mm thick and 20 mm × 20 mm dimension

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Table 1 Chemical composition of ADI used in the present investigation

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

were cut from the slabs for metallographic and diffraction work. Round tensile bars of diameter 6 mm and gauge length 30 mm were also machined from the cast blocks. The dimensions of the tensile specimen are shown in Fig. 1.

Austenitising treatment was carried out at 850, 900 or 950 °C for half an hour in a muffle furnace. The samples were then quickly transferred to a salt bath maintained at 300, 350 and 400 °C. After austempering for 2 h at these temperatures, the samples were cooled in air to room temperature.

The microstructures of the samples were studied by optical microscopy as well as X-ray diffraction. While optical microscopy gave qualitative information on the morphological aspect of the microstructure, the X-ray diffraction studies revealed quantitative information such as volume fraction of retained austenite and its carbon content. For optical microscopy, the samples were etched with nital. X-ray diffraction studies were carried out on JEOL JDX 8P diffractometer using copper K alpha radiation. Scanning was done over the 2θ range of 40–50° at a scan speed of 1° min^{-1}. The volume fraction of retained austenite was determined by the direct comparison method of Cullity [8] using integrated intensities of (111) peak of austenite, and the (200) peak of ferrite. The carbon content of retained austenite was determined from the lattice parameter of austenite using the following relationship due to Roberts [9]:

$$a_\gamma = 0.3548 + 0.0044C_\gamma \tag{1}$$

where a_γ is the lattice parameter of austenite in nanometers and C_γ is the carbon content in weight percent. The Bragg angle obtained from the (111) peak of austenite was used in estimating the lattice parameter. The carbon content was then estimated using this value of the lattice parameter in the above equation.

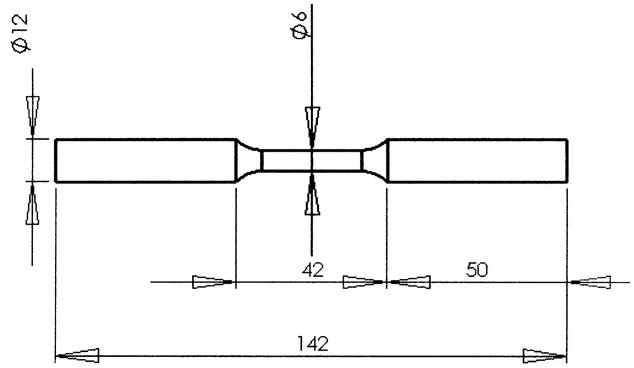

Fig. 1 Dimensions of tensile sample in mm

Tensile tests were carried out on a floor model 10T Instron machine (model 4206) as per ASTM E8 [10] at a cross head speed of 1 mm min^{-1}. Elongation was obtained from the cross head movement. Load elongation plots were obtained on an X–Y recorder and these were converted into stress–strain diagrams. 0.2% offset yield strength, ultimate tensile strength and percent elongation were obtained from these. Three tests were carried out for each heat treatment, and the results reported are an average of these three tests.

Investigations were carried out on the microstructure at the point of fracture. For this purpose the fracture surface was ground and polished. It was ground just sufficient to obtain a flat surface that was etched with nital and examined under the optical microscope. Since no sever grinding was carried out, a few depressions of the fracture surface remained, but did not detract from the quality of the microstructure. X-ray diffraction studies were also carried out on these surfaces to estimate the amount of retained austenite.

Results and discussion

Microstructural studies

The microstructure of ADI consists of bainitic ferrite and retained austenite. Retained austenite is present rather than carbides as in austempered steels due to the presence of large amount of silicon. However, several investigators [11, 12] have reported the presence of carbides in bainitic ferrite austempered at low temperatures such as 300 °C. This is attributed to the low diffusion rate of carbon at these low temperatures. All the carbon from the region transforming to ferrite is not able to diffuse into the surrounding austenite. The carbon that gets trapped will precipitate as ε carbide ($Fe_{2.4}C$). This tendency will decrease as the austempering temperature is increased. At 400 °C, the bainitic ferrite is free of such epsilon carbides. The microstructure of ADI which essentially consists of ferrite and austenite is called as ausferrite to distinguish it from the microstructure of austempered steel. As the austenitising temperature is increased, two changes occur in the microstructure: the carbon content of the austenite increases, and the austenite grain size increases. These bring about perceptible changes in the microstructure of the ADI. As the carbon content of the austenite is increased the driving force for the bainitic reaction decreases. There will be fewer nucleation of ferrite laths. These will grow to a larger extent. Hence, for a given austempering temperature, as the austenitising temperature is raised the microstructure appears coarser. Figure 2a, b

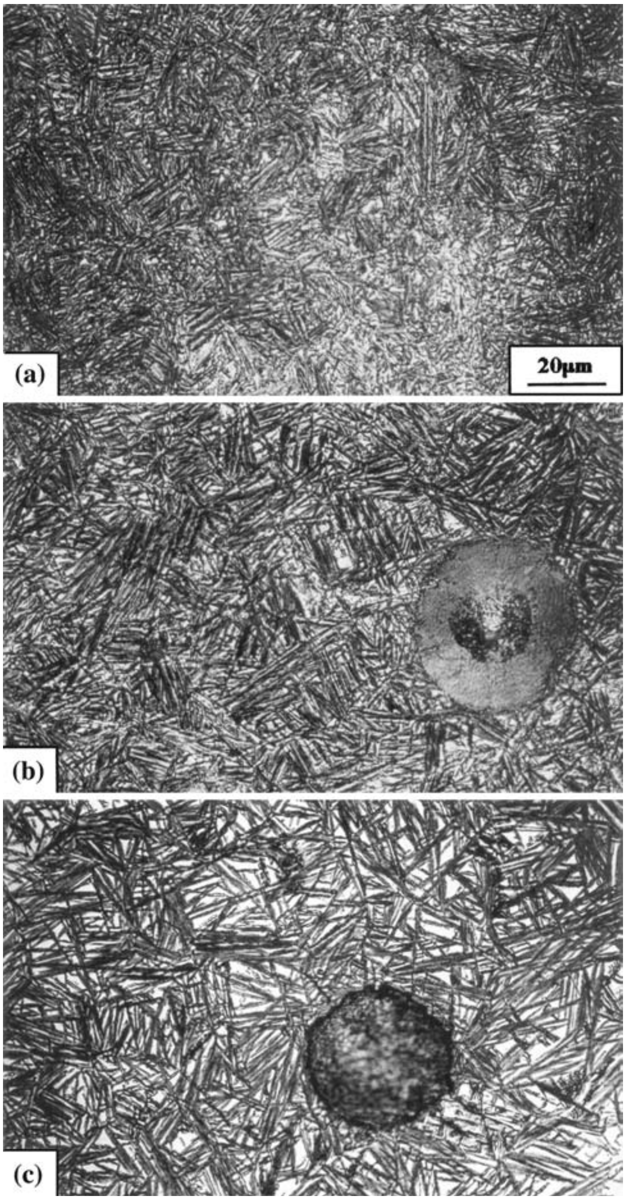

Fig. 2 Microstructures of samples subjected to austempering at 300 °C after austenitising at (**a**) 850 °C, (**b**) 900 °C, (**c**) 950 °C

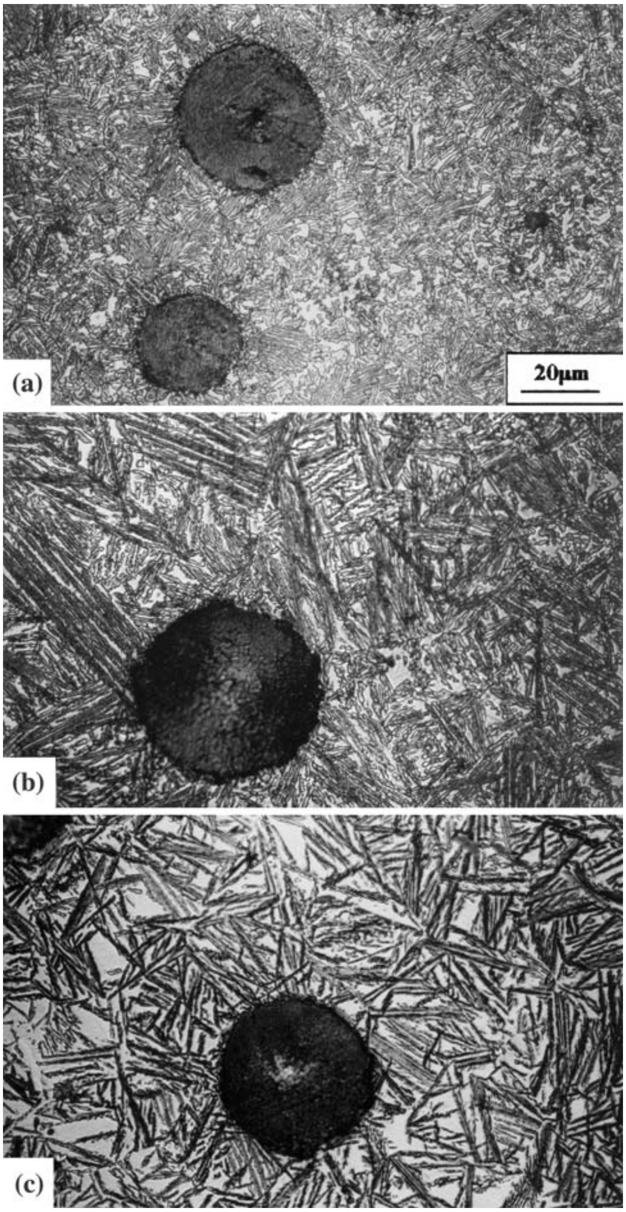

Fig. 3 Microstructures of samples subjected to austempering at 350 °C after austenitising at (**a**) 850 °C, (**b**) 900 °C, (**c**) 950 °C

and c show the microstructures of samples austempered at 300 °C after austenitising at the three temperatures of 850, 900 and 950 °C, respectively. A systematic change in the microstructure can be observed as the austenitising temperature is raised. At 850 °C the ferrite sheaths are small in size, numerous in number, and are closely spaced. At the temperature of 950 °C, they are long in length, and fewer in number. At 900 °C, the microstructural features are in between those of the above two. Similar observations can be made at the other two-austempering temperatures of 350 and 400 °C as shown in Figs. 3 and 4, respectively.

The quantitative features of the microstructure were obtained through X-ray diffraction. Figure 5 shows the variation in the retained austenite content as the austenitising temperature is increased. At the austempering temperature of 300 °C, it was found that the retained austenite content increased from a little over 13 vol.% at 850 °C to around 25 vol.% at 950 °C. Similar trend was observed at all the austempering temperatures. At a given austenitising temperature, the retained austenite content increased as the austempering temperature was increased. These results agree well with the observations of the previous investigators and can be explained on the same lines [2, 3, 13, 14].

The carbon content of the retained austenite was also found to increase with the increasing austenitising

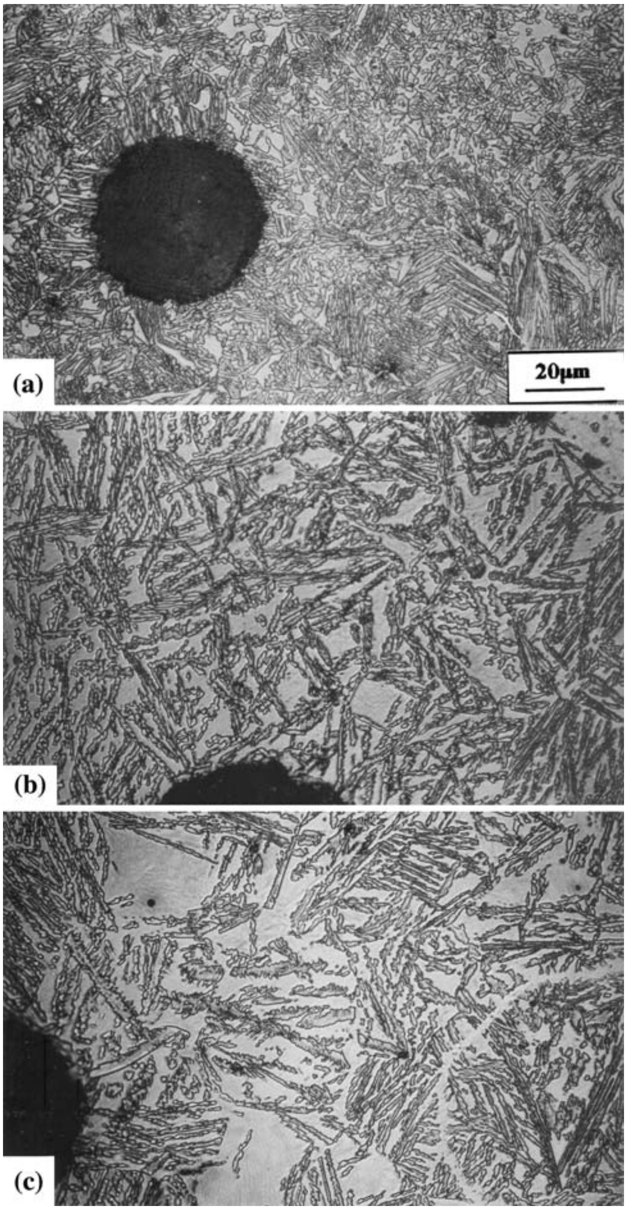

Fig. 4 Microstructures of samples subjected to austempering at 400 °C after austenitising at (**a**) 850 °C, (**b**) 900 °C, (**c**) 950 °C

temperature at a given austempering temperature. When austempered at 300 °C, the carbon content increased from 1.62 wt.% at the austenitising temperature of 850 °C, to 2.06 wt.% at the austenitising temperature of 950 °C. It was found that at a constant austenitising temperature, the carbon content of the retained austenite decreased as the austempering temperature was raised. The results are presented in Fig. 6.

The total carbon in the ausferrite is give by the product $X_\gamma C_\gamma$. Figure 7 shows the variation of this parameter with austenitising temperature. The total carbon was found to increase with increasing austenitising temperature as well as with increasing austempering temperature. The following empirical relationship due to Voigt and Loper [15] gives the carbon content of the austenite at the austenitising temperature T_γ:

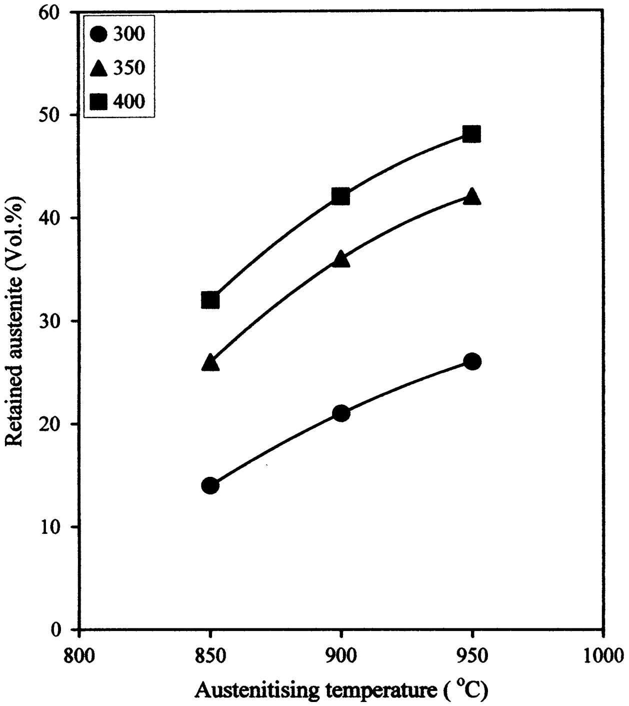

Fig. 5 The influence of the austenitising temperature on the retained austenite content at the three austempering temperatures indicated on the figure

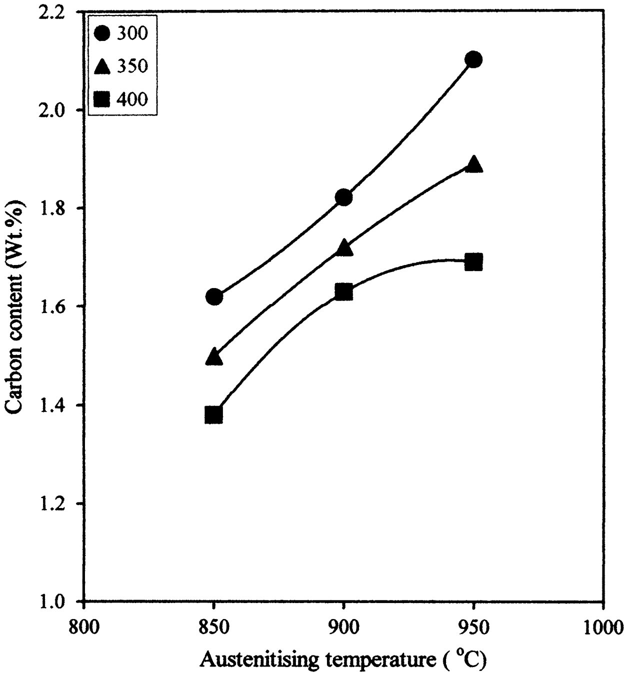

Fig. 6 The influence of the austenitising temperature on the carbon content of the retained austenite at the three austempering temperatures indicated on the figure

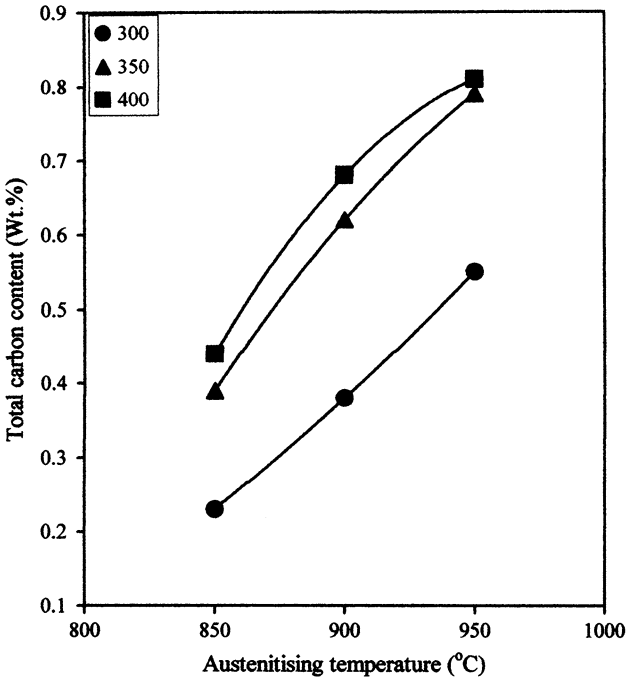

Fig. 7 The influence of austenitising temperature on the total carbon at the three austempering temperatures indicated on the figure

$$C^{\circ}_{\gamma} = (T_{\gamma}/420) - 0.17(\mathrm{Si}) - 0.95 \qquad (2)$$

Si is the weight percent of silicon in the ductile iron. Using the above formula, the carbon content of the austenite at the austenitising temperatures of 850, 900 and 950 °C are found to be 0.60, 0.72 and 0.84, respectively. It was found that only when austenitised at the high temperatures of 900 and 950 °C and austempered at the highest temperature of 400 °C, the total carbon in the ausferrite was close to the original carbon content of the austenite. At lower austempering temperature of 300 °C, the $X_{\gamma}C_{\gamma}$ values at the three-austenitising temperatures of 850, 900 and 950 °C were 0.22, 0.38 and 0.58 respectively, which showed that much of the carbon in the original austenite was trapped as carbides in the ferrite.

The above results show considerable influence of the austenitising temperature on the microstructure. On the one extreme, when the austenitising temperature was 950 °C and the austempering temperature was 400 °C, the microstructure was very coarse. It consisted of long broad sheaths of ferrite well separated from each other by bulky retained austenite. The retained austenite content was very large, nearly 47 vol.%. The carbon content as measured by C_{γ} was very low. However, the total carbon as given by $X_{\gamma}C_{\gamma}$ was very large indicating that practically all the carbon in the original austenite had found its way into the retained austenite. At the other extreme, when the austenitising temperature was 850 °C and the austempering temperature was 300 °C the microstructure was very fine. It consisted of short narrow sheaths of ferrite very closely spaced. Austenite was present as thin slivers between these ferrite sheaths. The retained austenite content was very low, about 13.5 vol.%. The carbon content of the retained austenite as measured by $C\gamma$ was quite high. However, the total carbon as given by $X_{\gamma}C_{\gamma}$ was very low indicating that very little of the carbon in the original austenite found its way into the retained austenite. Most of the carbon would have been trapped in the ferrite as carbides due to the low diffusion of carbon at the low austempering temperature [14]. Other combinations of austenitising temperature and austempering temperature exhibited microstructural features in-between the two extremes detailed above.

Mechanical properties

The influence of austenitising temperature on the tensile properties is shown in Table 2. The tensile studies revealed that at all the three-austenitising temperatures strength values decreased as the austempering temperature was increased. This can be attributed to the microstructural changes that are associated with increasing austempering temperature. These are, increasing retained austenite content and the broadening of the ferrite blades. It has been shown that the change in mechanical properties can be related to the microstructural features by the following relationship [16, 17].

$$\sigma_{\mathrm{YS}} = \sigma_{\mathrm{o}} + \mathrm{A}d^{-1/2} + \mathrm{B}X_{\gamma} \qquad (3)$$

where d is the ferrite lath width, X_{γ} is the volume fraction of the retained austenite, and σ_{o}, A and B are constants. Both Hayrynen et al. [16] and Ali et al. [17] showed that ferrite particle size is the dominant factor and the volume fraction of the retained austenite plays relatively small role. Therefore, the increasing particle size '*d*', of the ferrite as

Table 2 Influence of austenitising temperature on the tensile properties at the three austempering temperatures

Austempering temperature (°C)	Austenitising temperature (°C)	Tensile properties		
		Yiled strength (MPa)	Tensile strength (MPa)	Elongation (%)
300	850	1123	1196	0.65
	900	1385	1535	2.9
	950	1099	1166	0.7
350	850	873	979	0.93
	900	1100	1303	7.2
	950	537	964	3.96
400	850	645	911	3.47
	900	510	712	11.8
	950	458	749	5.3

the austenitising temperature is increased will lead to lowering of the strength.

At a constant austempering temperature of 300 °C, maximum tensile strength and yield strength were observed on austenitising at 900 °C, while lower values were observed at 850 and 950 °C. The austenitising temperature of 900 °C appears to be the optimum. It is generally believed [18] that an optimum retained austenite content of 25 vol.% is desirable for best tensile properties. In the present investigation, the sample austenitised at 900 °C and austempered at 300 °C had a retained austenite content of 24 vol.% as shown in Fig. 5. When austeniteised at 850 and 950 °C, and then austempered at 300 °C, these were respectively 14 and 31 vol.%. Thus the best mechanical properties were observed on austenitising at 900 °C. Similarly, the samples austempered at 400 °C showed the best properties at 850 °C, as it had a retained austenite content of 26 vol.%.

In order to understand the influence of the microstructure on the mechanical behaviour, the strain hardening behaviour was studied under all the heat treatment conditions. It was assumed that the following relationship due to Hollomon [19] was obeyed:

$$\sigma = \mathrm{K} \cdot \varepsilon^{n} \tag{4}$$

where σ is true stress, ε is true plastic strain, n is the strain hardening coefficient and K is the strength factor. If the above relationship is valid, the plot of true stress against true strain should result in straight line. Figure 8a shows such a plot for samples austempered at 300 °C. The figure shows three sets of straight lines for the three-austenitising temperatures. However, the samples austempered at 350 °C show the change of slope at the two higher austenitising temperatures of 900 and 950 °C (Fig. 8b), while those austempered at 400 °C show steps at all the three-austenitising temperatures as shown in Fig. 8c. A change in slope of the plot of ln σ against ln ε is taken to indicate a change in the strain hardening mechanism. When austempered at 400 °C, after austenitising at 850 °C, the value of n was found to increase from 0.05 at low strain values to 0.15 at high strain values. Similarly, when austenitised at 900 °C, it increased from about 0.02 at low strain values to 0.08 at intermediate strain values and increases further to 0.15 at high strains. Corresponding values for the samples austenitised at 950 °C were 0.11, 0.17 and 0.24 respectively.

The analysis of the strain hardening behavior showed that there was no change in the strain-hardening behavior of the samples austempered at 300 °C, irrespective of the austenitising temperature. However, the samples austempered at 400 °C showed a marked difference. The strain hardening tendency was found to increase as they were increasingly strained. This could be the result of changes that took place in the microstructure as the material was deformed. Figure 9 shows the microstructures of the fracture surfaces of the samples austempered at 300 °C after austenitising at the three temperatures of 850, 900 and 950 °C. These are very similar to those in Fig. 2, the microstructures of the undeformed samples under identical heat treatment conditions, and there is no discernible change. Figure 10 shows the microstructures of the fracture surfaces of the samples austempered at 400 °C. These show marked differences from those of the undeformed samples as shown in Fig. 4. Considerable formation of martensite could be observed at all the three-austenitising temperatures. These microstructures are shown at a lower magnification to show the extensive formation of martensite. Martensite was found to form in the intercellular regions, within the cells and also adjacent to graphite nodules. Figure 11a shows corresponding image as observed under scanning electron microscope. Martensite

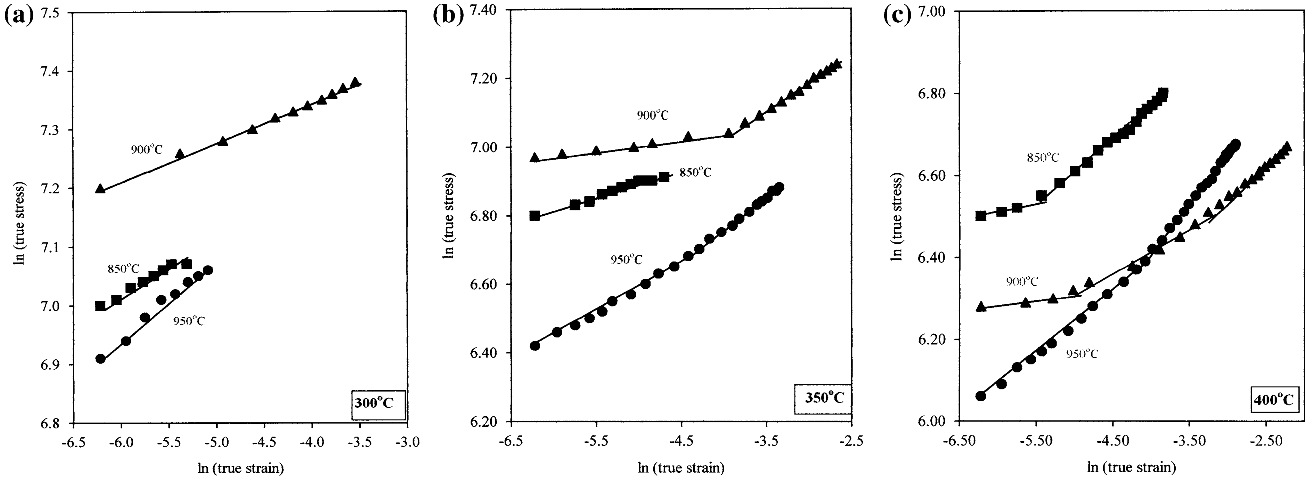

Fig. 8 Plot of log true stress against log true plastic strain in samples subjected to austempering at (**a**) 300 °C, (**b**) 350 °C and (**c**) 400 °C. Austenitising temperatures are indicated on the figures

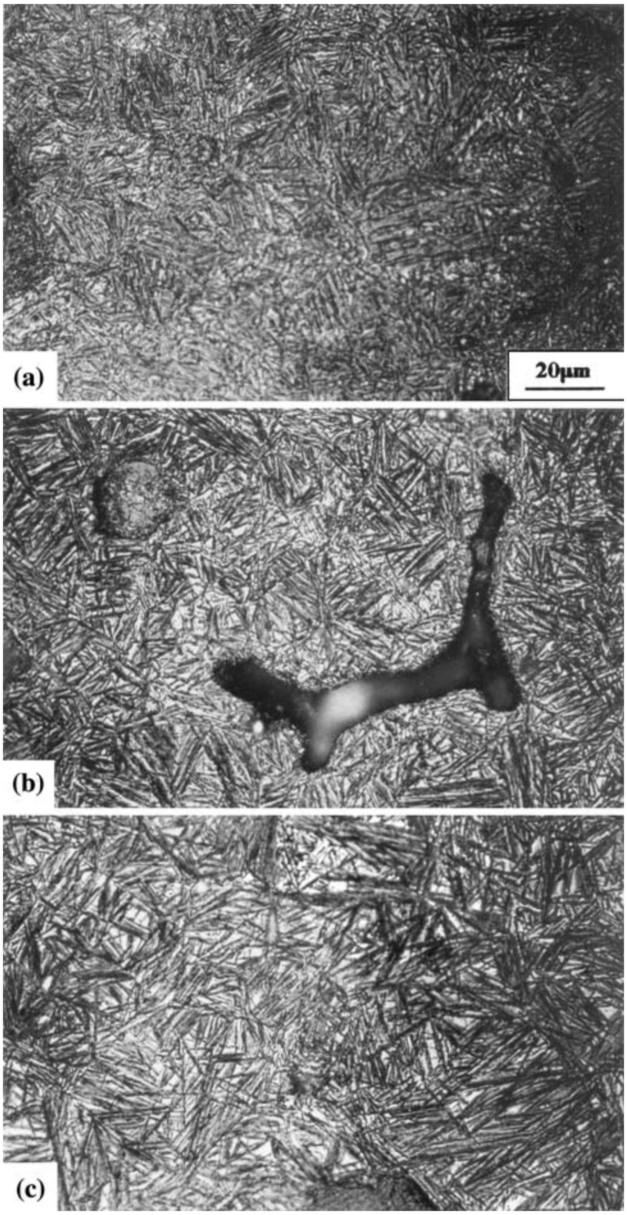

Fig. 9 Microstructures of fracture surfaces of samples austempered at 300 °C after austenitising at (**a**) 850 °C, (**b**) 900 °C, (**c**) 950 °C

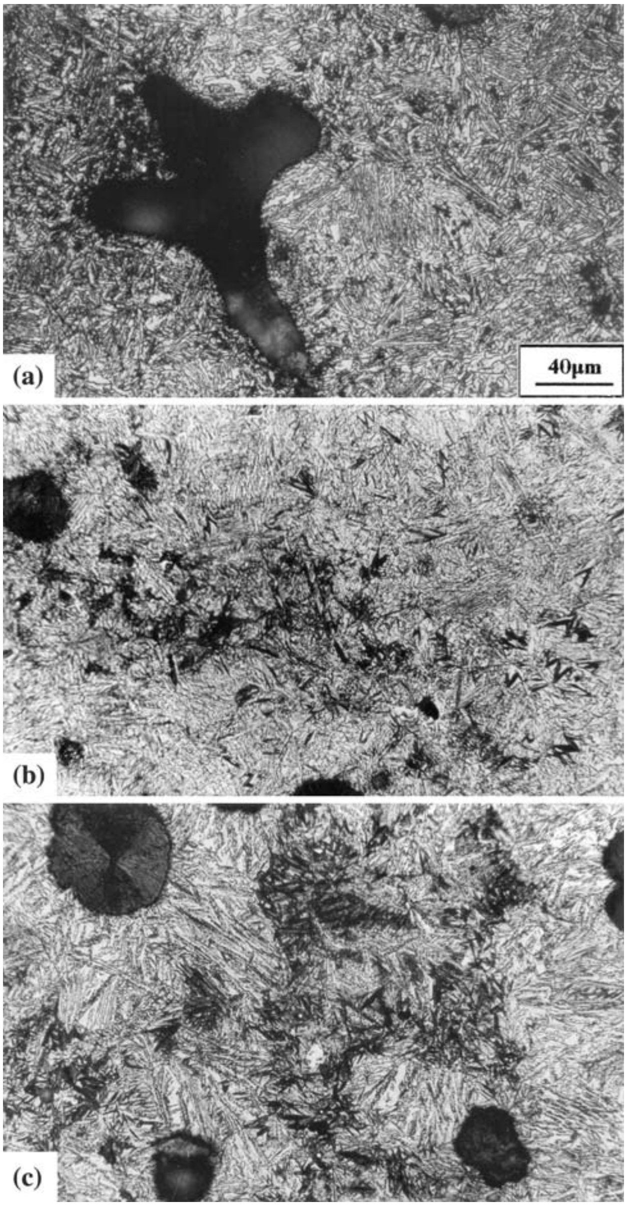

Fig. 10 Microstructures of fracture surfaces of samples austempered at 400 °C after austenitising at (**a**) 850 °C, (**b**) 900 °C, (**c**) 950 °C

is clearly observed confined to within the retained austenite. Figure 11b shows the scanning electron micrograph of the fracture surface of the sample austempered at 300 °C. No martensite formation is visible within the retained austenite, even though this is at a magnification more than twice that of Fig. 11a. It is quite clear that in the samples austempered at 400 °C, strain induced martensite is formed from the retained austenite during the deformation. Soft and ductile austenite is being replaced by hard and brittle martensite which provides greater resistance to the movement of dislocations. This is the cause of increasing strain hardening in these samples. The fact that the strain induced martensite formation is observed in the samples austempered at 400 °C, and not in those austempered at 300 °C may be attributed to the lower stability of the retained austenite in the former than in the latter. Carbon content of the retained austenite in the samples austempered at 400 °C was lower than that in the samples austempered at 300 °C as seen in Fig. 6. The samples austempered at 350 °C showed formation of some amount of martensite, as shown in Fig. 12. The amount of martensite was less than that in the samples austempered at 400 °C. Thus they showed behaviour intermediate to those austempered at 300 °C and 400 °C. This is to be expected since they have retained austenite content and its carbon content intermediate of those of the other two.

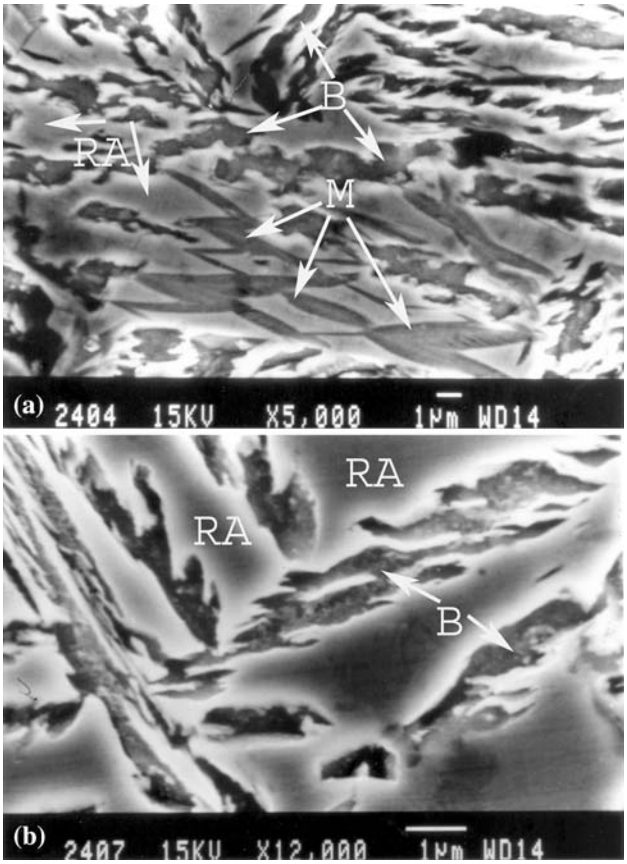

Fig. 11 Scanning electron micrographs of fracture surfaces of samples austempered at (**a**) 400 °C and (**b**) 300 °C. RA is retained austenite, B is ferrite and M is martensite

The fracture surfaces were studied by X-ray diffraction to estimate the amount of retained austenite after the samples had undergone deformation. The results are presented in Fig. 13. It was found that the samples austempered at 400 °C showed considerably less austenite content after deformation. The martensite observed in these samples can be directly correlated to the drop in the retained austenite content. This drop is found to increase with increasing austenitising temperature. While this was about 50% in the sample austenitised at 950 °C, it was only about 38% at 900 °C and about 19% at 850 °C. Similar trend was observed at the other austempering temperatures also. This is shown in Fig. 14.

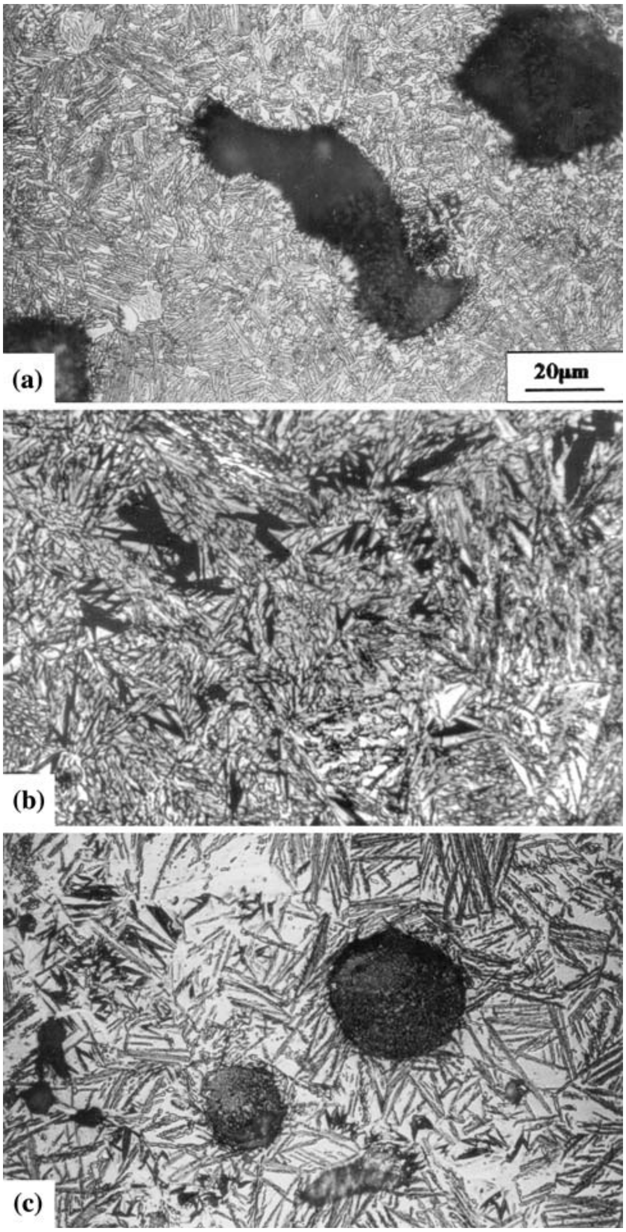

Fig. 12 Microstructures of fracture surfaces of samples austempered at 350 °C after austenitising at (**a**) 850 °C (**b**) 900 °C (**c**) 950 °C

It is pertinent to point out here that Daber and Rao studied the influence of plastic deformation on the stability of retained austenite in ADI. For this, ADI samples which were austenitised at 950 °C and austempered at 400 °C for 2 h were subjected to different plastic deformation in uniaxial tension. It was found that at 3% strain X_{γ} decreased to 40% from the initial value of 48% (17% decrement) while at 5% strain, it dropped to 26% (46% decrement). It is, therefore, appropriate to look at the amount of plastic strain at fracture in the present set of samples. This varied depending on the heat treatment condition as shown in Table 2. The samples austempered at 300 °C showed ductility in the range of 0.65–2.9%, while those austempered at 400 °C showed ductility in the range of 4–12%. The samples austempered at 350 °C showed an intermediate value of 1–7%. The three curves in Fig. 13 shift to higher values as the plastic deformation at fracture increases. The samples austempered at 300 °C, which had the minimum ductility, exhibited the minimum amount of strain-induced martensite. However, at a given austempering temperature, the curves of Fig. 13 did not follow the trend in Table 2. Thus, the austenitising temperature

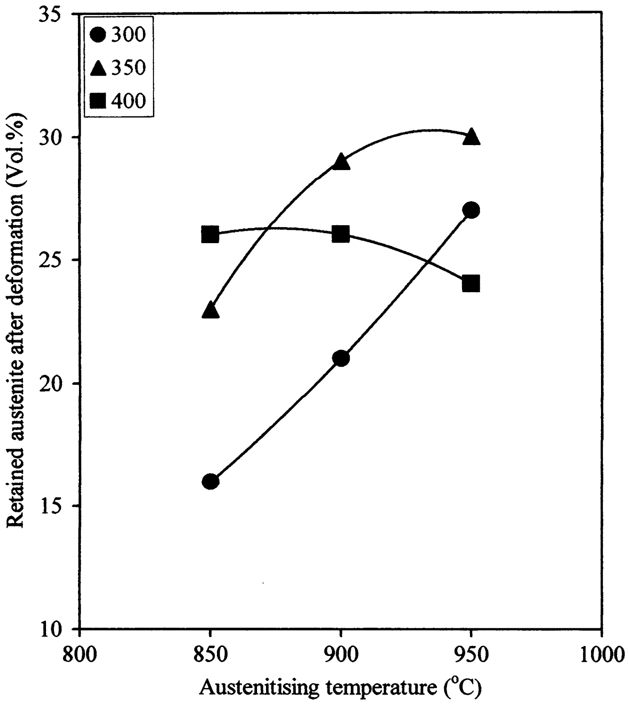

Fig. 13 Retained austenite content in deformed samples under different heat treatment conditions

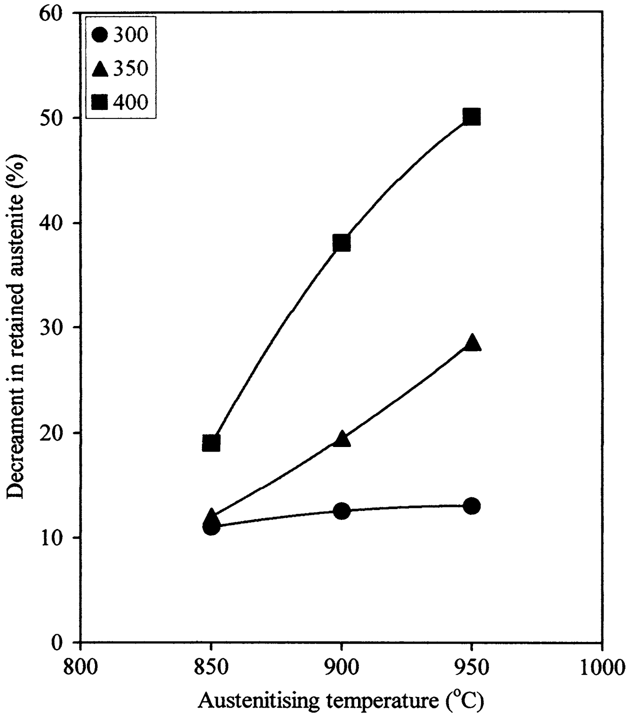

Fig. 14 Drop in the retained austenite content after deformation in samples austenitised at different temperatures

appears to be a more dominant factor than the extent of plastic deformation.

The results of the present investigation clearly show that the austenitising temperature has a considerable influence on the formation of the strain-induced martensite. Higher the austenitising temperature, greater is the tendency. This can be directly attributed to the microstructural changes associated with the change of austenitising temperature. The high austenitising temperature is associated with large volume fraction of bulky retained austenite. Bulky austenite is relatively unstable, and can easily transform to martensite under strain.

Conclusions

The present investigation shows that the retained austenite can transform to martensite through a TRIP like phenomenon. The propensity to transform to martensite under strain depends to a large extent on the austenitising temperature. As the austenitising temperature is increased the tendency to transform to martensite increases at all the austempering temperatures. High austenitising temperature together with high austempering temperature forms retained austenite with low stability, and therefore greater tendency to form martensite under strain.

Acknowledgement This work was financially supported by the Council of Scientific and Industrial Research, New Delhi, India.

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