Characterization and Sliding Wear Analysis of Austempered Ductile Iron

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Abstract: The present research work was carried out to examine the influence of various combinations of austempering heat treatments on the wear behaviour of Austempered Ductile Iron (ADI). Eight samples were taken for the analysis and all the samples were austenised at a temperature of 950°C for 30 minutes. A set of four samples were austempered at temperatures of 300°C and 400°C for 5, 15, 30 and 120 minutes and resulting microstructures were characterized through optical microscopy and X-ray diffraction. By varying the austempering temperature, significant changes were obtained in their morphology and rate of bainitic ferrite and residual austenite. Wear test was carried out using a pin on disc machine for a load of 2kg in which the test was undertaken on each of the sample for total time duration of 6 hours in step of 1 hour. The cumulative weight loss was used to determine the wear rate by the slope of weight loss against time. At low austenising temperature (300°C), it led to the formation of ferritic thin and compact needles, while, at higher austempering temperature like 400°C, wide feather-like blades of ferrite were observed in microstructure and for various austempering temperature, the morphology of residual austenite phase has changed, in a way that it changed from thin and delicate state at low temperatures into thick block-shaped state at high austempering temperatures. The coarse austenite microstructure exhibited higher wear rate than fine ausferrite microstructure. The results indicated that samples austempered at 300°C shows better wear resistance than samples austempered at 400° C for respective austempered time.

Key words: Austempered Ductile Iron (ADI), austenite, critical temperature, wear

1. Introduction

In recent years, there is a significant importance in energy saving which has led to the advancement of light weight, durable and cost effective materials. For this purpose, there is a requirement to continually formulate new materials and checkout those already in account. One such material is ductile iron. A ductile iron which subjected to a peculiar isothermal heat treatment process i.e. heating to the austenitizing temperature, followed by quenching into a salt or oil bath at a temperature in the range of 200°C to 445°C and holding for the time required for transformation to occur at this temperature, is known as austempered ductile iron (ADI) and the process is known as austempering [1].

Austempered Ductile Irons 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 stabilized austenite rather than ferrite and carbide as in austempered steels. Because of the presence of stabilized austenite, ADI exhibits excellent combination of strength and ductility, together with good fatigue and wear properties. Compared to the conventional grades of Ductile Iron, ADI delivers twice the strength for a given level of ductility in the form of elongation.

Accessing the desired mechanical properties of ADI irons is possible by controlling final microstructure. Microscopic structure is affected by temperature, time of austenitization and austempering. The most important and critical part of the microscopic structure of ADI is the residual high carbon austenite. Mechanical properties and wear behavior of ADI are largely affected by the volume fraction, morphology and carbon content of residual austenite.

Wear means the damage on or the removal of material from the surface when solid parts are in sliding, rolling, or impact motion in relation to each other. Wear can affect either one or both of two solid surfaces. Wear damage precedes the actual loss of material, and it may also occur independently despite the fact that generally the definition of wear is based on the loss of material. The consequences of wear process are usually described by wear rate. The reciprocal of wear rate is wear resistance. It measures how well the body resists the removal of material by wear processes [2].

The material characterization of ADI consists of studying the microstructure to understand the behavior of material for determining the mechanical properties which itself depends upon casting and alloying elements. The ductile iron austempered at lower temperatures say 300°C, will result fine laths of bainitic ferrite and small amount of retained austenite. But at 400°C microstructure shows coarse bainitic ferrite along with blocky austenite [3].

In recent past, several attempts have been made to investigate the wear behaviour of ADI with respect to their heat treatment parameters and mechanical properties. Meanwhile, a clear investigation on the wear behaviour and microstructure of ADI has yet to be carried out. The primary objective of the present investigation is to study the wear behaviour of ADI–austenised at a fixed temperature with different austempering temperatures and austempering times.

The aim of the work presented here is to investigate the variation in wear resistance of ADI and the material characterization of ADI also be carried out.

2. Experimental Procedure

2.1. Material

The alloy of ductile iron for the present study was cast in the form of slab $100 \times 25 \times 25$ cm. Compositional analysis was done by energy dispersive spectroscope (EDS).

Table 1. Chemical Composition of Ductile Used in Present Investigation				
Elements	Weight %			
Carbon	3.48			
Silicon	2.70			
Nickel	1.50			
Manganese	0.31			
Copper	0.05			
Molybdenum	0.31			
Phosphorous	0.01			
Sulphur	0.01			
Magnesium	0.05			

Table 1. Chemical Composition of Ductile Used in Present Investigation

The as-cast material was taken for microscopic analysis after polishing and etched with natal 3%. From the microstructural point of view the basic material consist of ferrite-pearlitic nodular cast iron with 60% of pearlite in the matrix. Graphite occurs only in a perfectly-nodular (80%) and imperfectly-nodular (20%) shape (not in flakes).

2.2. Heat Treatment

Heat treatment was carried out using two furnaces. An electric resistance muffle furnace with an accuracy of ± 5 °C, and a salt bath furnace with an accuracy of ± 2 °C, having 45 wt% sodium nitrate and 55 weight percentage of potassium nitrate solution, was used here for austenising and austempering process respectively. Conventional austempering process was carried out in the present investigation.



Fig. 1. Microstructure of the ductile iron in as cast condition, (mag. 400X).

The process involved austenising the sample at 950°C for 30 minutes and then subjected to austempering at 300°C and 400°C for different durations of times such as 5 minutes, 15minutes, 30 minutes, and 120 minutes. 300°C and 400°C is taken so that lower temperature is expected to produce lower bainitic microstructure with less amount of austensite while the higher temperature shows upper bainitic ferrite with considerable amount of austensite with the matrix of the materials. Fig. 2 shows the schematic diagram of austempering process adopted in the present investigation.



Time (minutes)

Fig. 2. Schematic diagram of conventional heat treatment.

2.3. Metallography

To elicit the information regarding the morphology, all the austempered samples under each heat treated condition were polished, polishing was done using fine emery paper and finally by cloth disc with alumina.

The samples were etched with 3% nital and viewed under optical microscope. The austempered samples were also analyzed under XRD and SEM at higher temperature.

2.4. Variation in Retained Austenite

Optical microscopy gives a qualitative picture of the phases present. Quantitative study can be done by X-Ray diffraction technique. X-ray diffractometer (model JEOL JPx8) with Cu K α radiation (λ =1.54 Å) was used as the source. Scanning was done in angular range 2 θ from 40° to 50° at a scanning speed of 2°/min. The XRD graphs were plotted using Origin Pro software.

The volume fraction of the retained austenite can be estimated quantitatively by X-Ray diffraction technique. Assuming that the ferrite and the austenite are the only matrix phases present, the ratios of integrated intensities of diffraction peaks from these phases can be written by the equation,

$$\frac{I_{\gamma(hkl)}}{I_{\alpha(hkl)}} = \frac{R_{\gamma(hkl)}}{R_{\alpha(hkl)}} \times \frac{X_{\gamma}}{X_{\alpha}}$$
(1)

where,

 $I_{\gamma}(hkl)$ is the integrated intensity from the given (hkl) plane from austenite,

 $I_{\alpha}(hkl)$ is the integrated intensity from the given (hkl) plane from ferrite,

 X_{γ}, X_{α} are the volume fraction of retained austenite and ferrite respectively, and

 $R\gamma(hkl)$, $R_{\alpha}(hkl)$ are constants and are given by the following expression

$$R = \frac{1}{V \times V} \times (F \times F \times P \times LP) \times e^{-2M}$$
⁽²⁾

where,

V is the atomic volume of unit cell, F is the structure factor, P is the multiplicity factor, LP is the Lorentz polarization factor e^{-2M} is the temperature factor. And

 $X\gamma + X\alpha = 1 \tag{3}$

Using the above equation one can determine the amount of austenite and ferrite present in the matrix.

2.5. Variation in Carbon Content

The lattice parameter of the austenite increases with increase in carbon content. They are related by the following equation,

$$a_{\gamma} = 0.3548 + 0.0044C_{\gamma} \tag{4}$$

where, a_{γ} is the lattice parameter of austenite in nanometer and C_{γ} is the carbon content of austenite in weight percent. The lattice parameter a_{γ} can be calculated by the equation,

$$a_{\gamma} = d_{(hkl)} \times (h^2 + k^2 + l^2)^{1/2}$$
(5)

And,

$$d_{hkl} = \frac{\lambda}{2\sin\theta_{\gamma}} \tag{6}$$

where λ is the wavelength of CuK_{α} radiation.

2.6. Hardness Testing

Hardness was measured for all the heat treatment conditions before and after wear test. Hardness values were taken on a Rockwell hardness testing machine in Rc scale for a load of 150kg. The indenter used was diamond indenter and the reported values are an average of at least five readings.

2.7. Wear Testing

A pin on disc wear testing machine was used to study the dry sliding wear behavior of different ADI samples, the schematic diagram of the machine is shown in Fig. 4. Samples of 8 mm diameter and a length of 25 mm were machined for the wear study. The experiments on all heat treated samples were conducted for a load of 2kg. The specimen was tightened and care has been taken so that cross section of the specimen was in contact with the disc during the experiment. The wear test was carried on each of the sample for 6 hours. The weight loss method was used to determine the wear rate in gm/min. The difference in weight before and after the test was used to determine the weight loss. The reduction in weight was measured after an interval of 1 hour using electronics balance. The weight loss experienced by the ADI under each heat treatment condition was then analyzed. After wear testing, micrographs of each deformed samples were taken through Scanning Electron Microscope. Then difference between non-deformed and deformed samples of ADI under each heat treatment condition is analyzed.



Fig. 3. Wear pin sample

Fig. 4. Pin on disc set up

Table 2. Main reactives of r in on Disc Machine.		
Disc Size	Dimension	
Outer diameter	215mm	
Inner diameter	45mm	
Thickness	20mm	
Hardness	52HRC	

Table 2.	Main	Features	of Pin on	Disc	Machine.
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Table 3. Testing Parameters Used in the Wear Measurement of the

Present Study				
Parameters	Values			
Load	2kg			
Sliding speed	4.71 m/s			
Sample dimension	8mm diameter and 25mm length			
Sliding distance in 6 hours	101.736 km			

3. Results and Discussions

3.1. Qualitative Analysis

3.1.1. Metallography

In the present investigation, the variation in austempering conditions resulted in a variation in the microstructure. The result of the optical metallography is shown in Fig. 5 and Fig. 6. These are the microstructure obtained for the austempering times of 5, 15 minutes, 30 minutes and 120 minutes for the austempering temperature of 300°C after austenitisng at 950°C for 30 minutes.



Fig. 5 (a-d). Samples austempered at 300°C for austempering times of 05, 15 30 and 120 minutes respectively. (Mag.400X)

For the austempered of 5 minutes, very small amount of bainite could be seen in the intermediate vicinity of the graphite particles as shown in Fig. 5 (a). Bainite ferrite is seen as the dark etching sheaves, unstabilised austenite is seen as the large white etching blocky areas. These regions were believed to transform into martensite during cooling to room temperature. When the time was increased to 15 minutes, one can notice a corresponding increase in the volume fraction of bainitic ferrite with a reduction in the unstabilised austenite regions. However the samples austempered for 30 minutes showed considerable amount of bainitic ferrite and its growth also as shown in Fig. 5 (c). The 120 minutes austempered sample showed full transformation as shown in Fig. 5 (d).

The samples austempered for different timings in the same way but at 400° C showed similar trend as shown in Fig. 6(a-d). Since at higher temperature the nucleation of bainitic ferrite is low but iths growth rate is favored, the volume fraction of ferrite is found to be less. Considerable amount of blocky auistenite can be observed in samples austempered for 120 minutes. These ferrites are feathery type and well

separated from each other from the islands of austenite.



Fig. 6 (a-d). Samples austempered at 400°C for austempering times of 05, 15 30 and 120 minutes respectively. (Mag.400X)

3.2. Quantitative Analysis

3.2.1. X-RD Analysis



Fig. 7. XRD profile for samples austempered at 300°C for 5, 15, 30 and 120 minutes.

The X-ray diffraction technique helped in estimating the exact quantity of retained austenite and bainitic ferrite. The results are shown in Fig. 7 and Fig. 8 for the samples austenised at 950° C and subjected to

austempering conditions which are indicated on the figure. The XRD patters shows a shift in the austenite peak as the austempering time was increased which indicates increase in carbon content of retained austenite or in other words stability of austenite.



Fig. 8. XRD profile for samples austermpered at 400°C for 5, 15, 30 and 120 minutes.



Fig. 9. Variation of retained austenite volume fraction with austempering time for the samples austenitised at 950°C.



Fig. 10. Variation in carbon content of retained austenite volume fraction with austempering time for the samples austenitised at 950°C.

The volume fraction of retained austenite estimated is shown in Fig. 9, the retained austenite content goes on increasing with increase in austempering time for a given austemring temperature. This is mainly due to the sufficient time available for transformation with increase in austempering time.

In Fig. 10 explains how the retained austenite is getting enriched with carbon with increase austempering time. This increase in trend both in austenite and carbon content can also be observed for the other set of samples which are austempered at 400° C.

3.3. Wear Study

The wear behavior of the material in the present investigation is studied at austempering temperatures 300° C and 400° C and the results are shown in Figs. 11 and 12 shown below:



Fig. 11 (a). Wear rate: 4.398 x 10 ⁻⁴gm/min.



Fig. 11. (c). Wear rate: 3.67 x 10 ⁻⁴gm/min.



Fig. 11. (b). Wear rate: 3.885 x 10 ⁻⁴gm/min.



Fig. 11. (d). Wear rate: 2.81 x 10 -4gm/min.

Fig. 11. Weight loss against time for austempering temperature at 300°C for different austempering time.



Fig. 12 (a). Wear rate: 4.681 x 10 -4gm/min



Fig. 12 (b). Wear rate: 4.428 x 10 -4gm/min



Fig. 12 (c). Wear rate: 3.871 x 10 -4gm/minFig. 12 (d) Wear rate: 3.493 x 10 -4gm/minFig. 12. Weight loss against time for austempering temperature at 400 °C for different austempering
time



Fig. 13. Wear rate of samples austempered at 300°C and 400°C for 5, 15, 30 and 120 minutes.

The slope values calculated for each heat treated condition gives the wear rate and result is shown in Fig. 13. From this figure, it can be observed that the wear rate decreased with increase in austempering times for both the austempering temperatures. However, the samples austempered at 300°C shows decrease in wear rate at any of the austempering time compared to that of 400° C.

It should be noted that the samples austempered for the times 5, 15 and 30 minutes for both 300°C and 400°C contain unsterilized austenite (or martensite) and hence they are very brittle. This has led to easier removal of the material and resulted in higher wear rate. Fig. 14 shows the microstructure for sample contains unsterilized austenite austempered at 400°C for 5 minutes.





Fig. 14(a-c). SEM images for samples austempered at (a) 400° C for 5 minutes, (b) 300°C for 120 minutes, (c) 400°C for 120 minutes

The samples austempered for 120 minutes resulted in lower wear rates and in this lowest value is observed in samples austempered at 300°C rather than 400°C. This may be attributed to the fact that at 400°C, some deformation induced martensite may form which once again induces brittleness to the material. Whereas at 300°C for 120 minutes the formation of fine acicular ferrite along with stabilized austenite contributes to the lower wear rate for the material. Fig. 14(b) and 14(c) shows the microstructure for sample austempered at 300°C and 400°C for 120 minutes. From these figures, it can be observed that the sample austempered at 300°C for 120 minutes is free from strain induced martensite while that at 400C for 120 minutes shows small martensitic needles which is responsible for inducing brittleness to the material.

After discussion of plots, the examination of ADI samples through SEM helps to understand the wear mechanism of ADI. The worn out surface when seen under the SEM showed abrasive and ductile nature of material removal. The samples austempered for lower timings such as 5, 15 and 30 minutes showed predominately abrasive nature as shown in Fig. 15(a-c) and 16(a-c). Whereas, in Fig. 15(d) and 16(d) for 120 minutes resulted in plough and tongue formation which shows that adhesive nature of wear.



(c). 30 minutes (d). 120 minutes Fig. 15(a-d). SEM images of worn out surface of ADI samples austempered at 300°C for different time.



(a). 05 minutes

(b). 15 minutes



Fig. 16. SEM images of worn out surface of ADI samples austempered at 400 °C for different time

3.4. Hardness:

The above Fig. 17 shows the hardness values taken for the samples austempered for 300°C and 400°C before wear test.



Fig. 17. Variation of hardness with austempering temperature and time before wear test

By analyzing the above figure higher hardness values were observed for the samples austempered at 300°C than for 400°C. However the hardness values are found to be decreased with increase in austempering time as the volume fraction of unstabilised austenite which are likely to transform in to martensite gets reduced with increase in time. So, the decrease in hardness at 400°C compared to 300°C for a given austempering time is attributed to the presence of feathery bainite and quick stabilization austenite due to faster diffusion of carbon in to the surrounding austenite.

Another factor for the observed wear behavior under different austempering conditions is the more or less a constant hardness values. The hardness values taken before and after wear test are shown in Fig. 18. If there is considerable strain hardening in the material during wear, then it should result in increase in hardness. But it is clear from the figure that there is hardly any increase in hardness or in other words the material has undergone negligible amount of plastic deformation. Therefore the observed decrease in wear rate at 300°C and 400°C for 120 minutes is mainly due to the microstructure.





wear test

4. Conclusion

- 1. Increasing austempering time increases the stabilization of retained austenite for a given austempering temperature and increasing austempering temperature increases the amount of retained austenite for a given austempering time.
- 2. Hardness decreases with increasing asutempering time for a given austempering temperature and this decrease is true for increasing temperature for a given austempering time.
- 3. The amount of bainitic ferrite increases with increasing austempering time.
- 4. The carbon concentration of the residual austenite increases during bainitic transformation.
- 5. Wear rate decreases with increase in austempeing time for a given austempering temperature and is found to increase with increase in austempering temperature.

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