ISSN (Online) 2393-8021
ISSN (Print) 2394-1588

International Advanced Research Journal in Science, Engineering and Technology
Vol. 2, Issue 6, June 2015

Investigation of Microstructural aspects and Wear behaviour of Cryotreated Austempered Ductile Iron (ADI)

Vivek K.M[1], K. Rajendra Udupa[2], Ravishankar K.S.[3]

M.Tech Scholar, Dept. of Metallurgical and Materials Engineering,

National Institute of Technology Karnataka, Surathkal, India [1]

Professor, Department of Metallurgical and Materials Engineering,

National Institute of Technology Karnataka, Surathkal, India[2]

Assistant Professor, Department of Metallurgical and Materials Engineering,

National Institute of Technology Karnataka, Surathkal, India[3]

Abstract: Austempered ductile iron (ADI) is a heat treated ductile Iron or S.G. iron with a unique micro-structure, which consists of retained austenite and bainitic ferrite with graphite nodules dispersed in it. Due to this excellent microstructure it possesses high yield strength with good ductility, good fatigue strength, fracture toughness and wear resistance. ADI has emerged as a major engineering material in recent years because of its many attractive properties. The properties can be achieved upon adequate heat treatment which yields optimum microstructure for a given chemical composition. But this type of heat treatment is bit tricky, since it requires controlled heating and isothermal holding of the material. The wear properties of ADI not only depends upon the hardness but also depends on the microstructure of the material.

Present investigation was carried out to assess the influence of cryogenic processing on the microstructure and wear properties of ADI. Experiment was carried out on two categories of ADI, one is austempered at 400ºC and other is at 340ºC with austempering time of 2.5, 3 and 3.5 hours respectively. These specimens were then cryogenically processed. The morphological changes, hardness values and wear rate of these samples were evaluated and compared with the noncryogenically treated samples. Test results indicates that the cryogenic processing can improve the wear properties, the lowest wear rate is observed for cryotreated ADI austempered at 400ºC for 2.5hour.

Keywords: Austempered Ductile Iron (ADI), Cryogenic Treatment, Austenite, Martensitic Transformation.

I. INTRODUCTION

Austempered ductile iron (ADI) is considered to be an important engineering material because of its attractive properties such as good ductility at high strength, good wear resistance and fatigue strength and fracture toughness [1]. According to Olivera et. al., the manufacturing cost of ADI is substantially lower than wrought or forged steel.

Furthermore, the density of ADI is 7.2 g/cc compared to 7.8 g/cc of steel, thus adding an advantage for ADI with respect to specific strength i.e strength per unit weight. [2, 3]. The mechanical properties of ADI can be tailored to suit particular applications by adjusting heat treatment parameters or material composition [4]. This is achieved through the control of the proportion and morphology of phases present in the microstructure of the matrix.

The matrix is referred to as ausferrite which is the mixture of acicular ferrite and retained austenite [5]. Ausferrite exhibits twice the strength for a given level of ductility compared to the pearlitic, ferritic or martensitic structures formed by conventional heat treatments. [6].

In the matrix of ausferrite the phase occurs in the form (i)super cooled and stable austenite which contains carbon more than 1.6 wt% (ii) super cooled metastable austenite containing carbon in between 1 to 1.6 wt% and (iii) unstable untransformed austenite containing carbon less than 1.0 wt%.

All these varieties of austenite could get enriched with Ni and Mo during the heat treatment. Each one of these austenites will feature a different characteristic and its proportion will be able to influence the final properties of the entire casting. It opine that the most desired type of austenite in ADI would be that which is thermally and mechanically stable at ambient temperature. However, undoubtedly interesting from the point of view of obtaining the best properties from ADI is the metastable austenite. Converting the metastable austenite into phases like martensite particularly can alter the property of ADI [7].

The present paper aims at characterising and assessing the mechanical properties like hardness and wear resistance of ADI material before and after cryotreatment. The microstructures of ADI material are sought to be changed by changing the austempering parameters and restoring to cryogenic treatment.

ISSN (Online) 2393-8021
ISSN (Print) 2394-1588
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II. EXPERIMENTAL PROCEDURE

A. Material and sample preparation

The chemical composition of ductile iron material is presented in the Table I.

TABLE I: CHEMICAL COMPOSITION OF DUCTILE IRON USED IN THE PRESENT INVESTIGATION

Elements	Composition Wt.%
C	3.6
Si	2.8
Mn	0.31
Mo	0.30
S	0.01
P	0.01
Mg	0.04

The sample of the size 40mm×15mm×10mm were cut from the 30mm thick cast ductile iron blocks.

B. Austempering and cryo-treatment cycle

Prior to austempering, the ADI samples were austenitised at 890°C for one hour in a muffle type electrical furnace the temperature of which can be controlled automatically in the range of ±5°C. ADI samples in austenitised condition were dropped into salt bath containing the mixture of sodium nitrate and potassium nitrate in the ratio of 55:45 by weight to give the austempering treatment. The austempering treatment was carried out at two different temperatures i.e 400°C and 300°C for different time periods: 2.5, 3.0 and 3.5 hours. The temperature of austempering furnace controlled automatically in the range of ±2°C. The austempering categories are shown in Table II.

TABLE II: DIFFERENT AUSTEMPERING CONDITIONS

Austempering category	Austempering temperature	Austempering time in hours
I	400°C	2.5
		3
		3.5
I I	340°C	2.5
		3
		3.5

The cryogenic heat treatment cycle consists of several steps such as (i) austentisation process (ii) austempering process (iii) cryogenic temperature holding (iv) ramp up stage to ambient temperature. Here the austentising temperature chosen was 890°C for both category as mentioned in Table II and also the austempering temperature and time was chosen from the same table. After the completion of the austempering process, the specimens were subjected to cryogenic treatment in liquid nitrogen having temperature of -196°C. Samples were soaked in the nitrogen container for a duration of 5hours for deep freezing followed by bringing back the sample to ambient temperature. The graphical representation of heat treatment cycle (Austempering and cryotreatment) is shown in the Fig. 1.

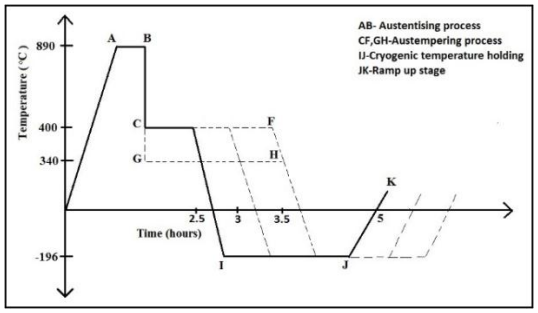

Fig. 1. The austempering and cryotreatment cycle

C. Microstructural investigation

Standard metallographic practice was used to prepare the sample. They were etched with 3% nital and observed under optical and scanning electron microscope of JEOL JSM-6380A.

D. X-ray diffraction (XRD) analysis

Quantitative information on the amount of retained austenite and ferrite were obtained through X-ray diffraction using Jeol JDX 8P diffractometer. Diffraction studies were carried out using CuKα radiation and scanning was carried out over the 2θ range of 40-50° at a scan speed of 0.5°/min. The volume fraction of the retained austenite was determined by the direct comparison method as suggested by Cullity using integrated intensities of (110) peak of ferrite and (111) peak of austenite. Assuming that ferrite and austenite were the only matrix phase present, the ratios of integrated intensities of diffraction peaks from these phases can be written as

$$\frac{I\gamma}{I\alpha} = \frac{R\gamma \,.\, X\gamma}{R\alpha.\ X\alpha}$$

Where Iγ and Iα are the integrated intensities of a given (hkl) plane from the γ phase and α phase respectively. Xγ and Xα are the volume fractions of retained austenite and ferrite respectively. The constants $R\gamma$ and $R\alpha$ are given by the following expression for each peak.

$$R = 1/v^2\ (|F|^2 pL)\ e^{-2m}$$

Where v is the volume of the unit cell; F is the structure factor; p is the multiplicity factor; L is the Lorentz polarization factor; and e^{-2m} is the temperature factor[8, 9].

E. Hardness test

Hardness values of the samples over the ausferrite matrix were measured before and after cryogenic treatment using Vickers Hardness Test with the load of 500gm for 15 seconds.

F. Wear test

The samples were machined in a lathe as per the ASTM standard G-99 is shown in Fig. 2.

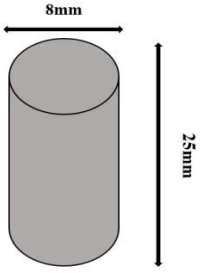

Fig. 2. Wear test sample

DOI 10.17148/IARJSET.2015.2620

ISSN (Online) 2393-8021
ISSN (Print) 2394-1588
International Advanced Research Journal in Science, Engineering and Technology
Vol. 2, Issue 6, June 2015

Unlubricated sliding wear test was carried out on a conventional pin on disc machine. It is shown in Fig. 3.

Fig. 1. Wear testing machine

Wear test was carried out on differently austempered ADI samples before and after cryotreatment. Testing procedures are listed below

- Cylindrical specimen was cleaned with acetone and weighed the sample using electronic weighing balance.
- Weighed sample was secured in the specimen holder, care taken for maintaining flat surface contact with rotating disc.
- Load of 2kg put on the weighing pan.
- Switch on the machine and run it for 30 minutes continuously.
- Sample was cleaned using acetone and again noted down its weight. This procedure is repeated in every 30 minute interval up to reaching 180 minutes.
- Using the weight loss data in every 30 minute a graph between cumulative weight loss and time was plotted and found out the wear rate by finding slope of the graph.

III. RESULTS AND DISCUSSION

A. Analysis of ADI Austempered at 400°C

The optical photomicrograph of the sample in as cast condition before starting of heat treatment operation was shown in Fig. 4. It exhibits predominantly pearlitic structure with graphite nodules embedded in it. The microstructures of ADI austempered at 400ºC with time periods 2.5hr, 3hr, 3.5hr is presented in Fig. 5a, 6a and 7a respectively. While the microstructures of the samples processed by cryogenic heat treatment are presented in Fig. 5b, 6b and 7b respectively.

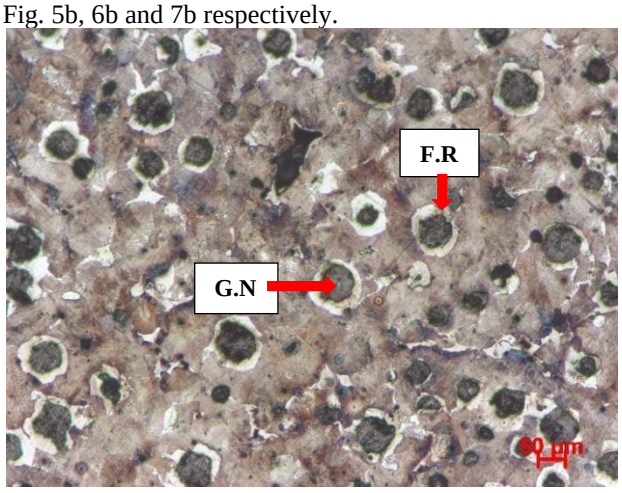

Fig. 4. Photomicrographs of fine pearlitic ductile iron in as cast condition. G.N- Graphite Nodule, F.R- Ferrite Ring

The microstructures of austempered samples showed ausferritic structure, which is a mixture of lath is shaped ferrite and stable retained austenite. It has the appearance of feathery shaped upper bainitic microstructure. The ferrite appears as dark needles, whereas austenite appears as white in the micrograph. Due to high austempering temperature carbon diffusion is more rapid so most of the carbon is able to diffuse out the growing bainitic ferrite platelets, enriching the residual austenite, particularly between the growing ferrite plates. As the isothermal treatment is prolonged, the carbon content of the residual austenite increase, which would leads to inhibition of bainitic transformation. Furthermore the martensite start temperature (M_s) is depressed, resulting in austenite being retained after cooling to ambient temperature. When the time of soaking in salt bath increases from 2.5 to 3.5hr the bainitic formation increases so that the retained austenite content decreases.Detailed observation of microstructure after cryotreatment reveals that austenite transforms into martensite in special micro regions. The observed phase is martensite which can be inferred from the morphology of lens-like platelets arranged in characteristic morphology "zigzags". Further, the absence of transformation of austenite into martensite in regions between the ferrite platelets is noticed. This is due to the fact that these regions get saturated with carbon sufficient to lower the MS temperature of austenite to below -196ºC [10].

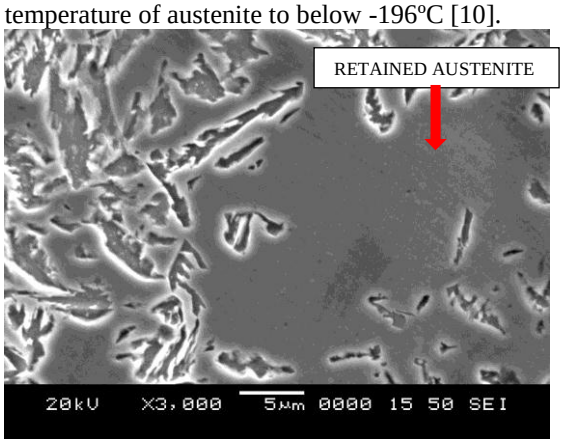

Fig. 5a. SEM photomicrograph of sample austempered at 400ºC with austempering time of 2.5 hr

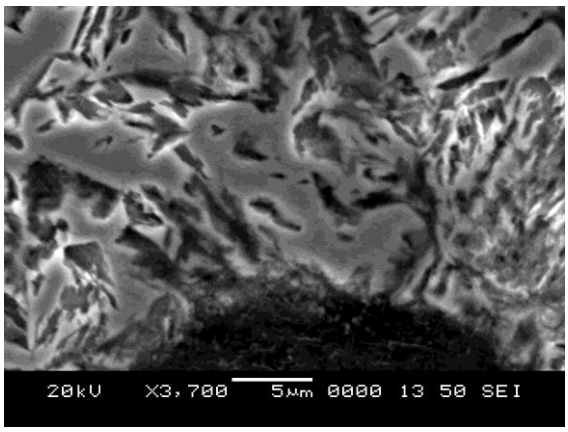

Fig. 6a. SEM photomicrograph of sample austempered at 400ºC with austempering time of 3 hr

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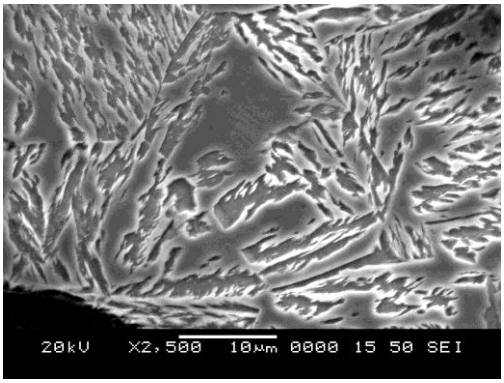

Fig. 7a. SEM photomicrograph of sample austempered at 400ºC with austempering time of 3.5 hr

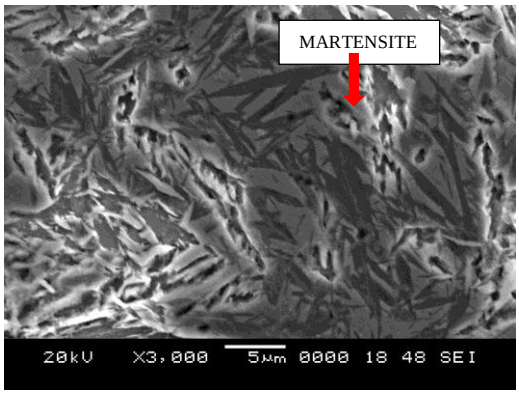

Fig. 5b. SEM photomicrograph of cryotreated ADI austempered at 400ºC with austempering time of 2.5 hr

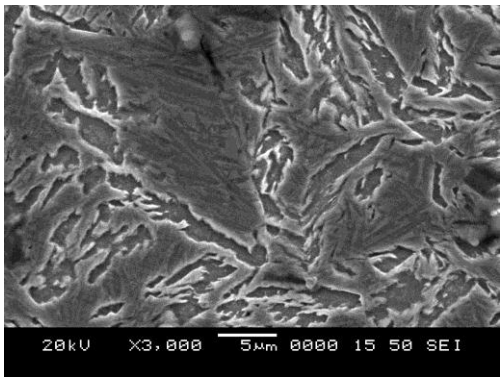

Fig. 6b. SEM photomicrograph of cryotreated ADI austempered at 400ºC with austempering time of 3 hr

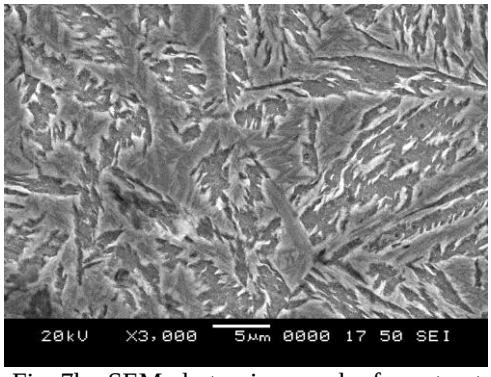

Fig. 7b. SEM photomicrograph of cryotreated ADI austempered at 400ºC with austempering time of 3.5 hr

The combined XRD patterns of ADI samples before and after cryogenic treatment austempered at 400ºC with time periods 2.5hr, 3hr and 3.5hr is demonstrated in Fig. 8, 9 and 10.

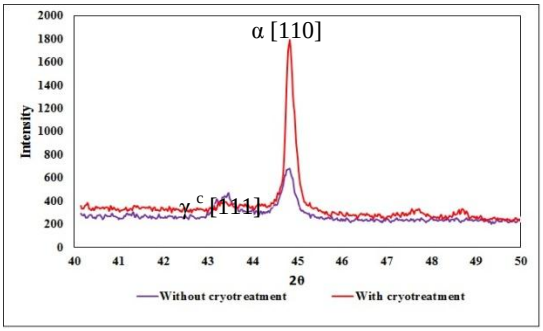

Fig. 8. Combined XRD profile of ADI before and after cryogenic treatment austempered at 400ºC for 2.5hr. γ^{c} _ Retained austenite, α- Ferrite

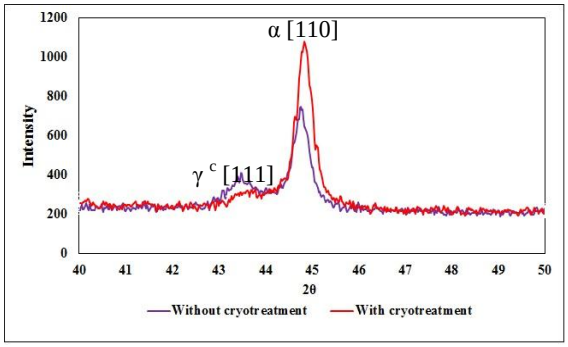

Fig. 9. Combined XRD profile of ADI before and after cryogenic treatment austempered at 400ºC for 3hr. γ^{c} _ Retained austenite, α- Ferrite

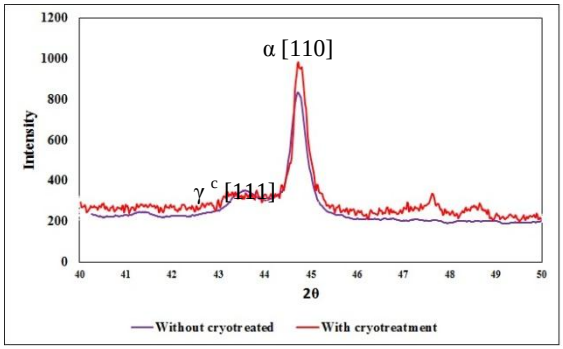

Fig. 10. Combined XRD profile of ADI before and after cryogenic treatment austempered at 400ºC for 3.5hr. γ^{c} _ Retained austenite, α- Ferrite

The transformation of austenite into martensite is confirmed by the results of the X-ray diffraction patterns. The successive curves representing austenite (111) and ferrite (110) show some typical changes after cryogenic treatment on ADI samples in which the XRD peak of austenite decreases and the XRD peak of ferrite increases.

As ferrite does not undergo any transformation due to cryotreatment, it could be conceived that the (110) and (011) diffraction peaks of martensite as well as a strong (110) peak of ferrite overlap, which makes their separation

ISSN (Online) 2393-8021
ISSN (Print) 2394-1588
International Advanced Research Journal in Science, Engineering and Technology
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difficult. This is the reason why considerable increase in (110) peak after cryotreatment [11].

B. Analysis of ADI Austempered at 340°C

The microstructures of ADI austempered at 340°C with time periods 2.5hr, 3hr and 3.5hr are shown in Fig. 11a, 12a and 13a respectively. While the microstructure of the samples processed by cryogenic heat treatment is reported in Fig. 11b, 12b and 13b respectively.

Even under this condition the structure obtained is upper bainitc one. The mechanism and microstructural aspects are same as the above case but the rate of carbon diffusion is relatively low due to lower temperature compared to first case. Thereby, it leads to low retained austenite content and high ferrite content.

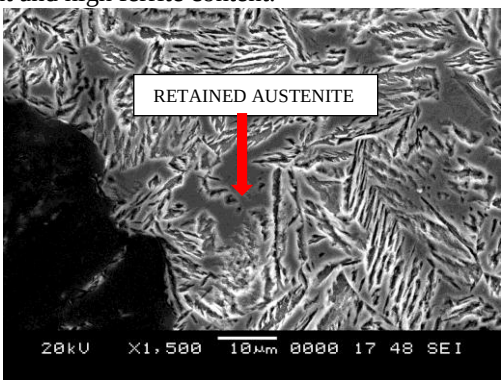

Fig. 11a. SEM photomicrograph of sample austempered at 340°C with austempering time of 2.5 hr

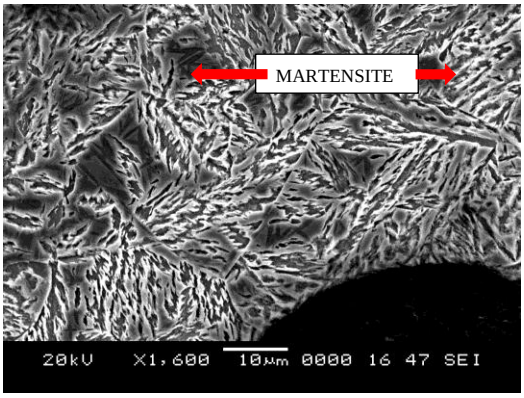

Fig. 11b. SEM photomicrograph of cryotreated ADI austempered at 340°C with austempering time of 2.5 hr

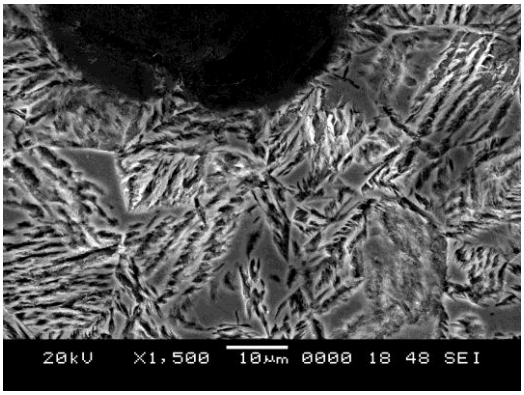

Fig. 12a. SEM photomicrograph of sample austempered at 340°C with austempering time of 3 hr

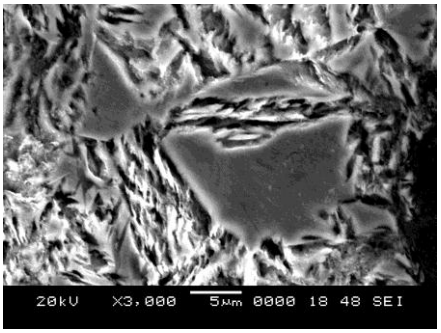

Fig. 13a. SEM photomicrograph of sample austempered at 340°C with austempering time of 3.5 hr

After the cryotreatment as in the previous case here also got the lens-like platelets arranged in characteristic shape "zigzags" in the matrix known as martensite. Only difference between the first case and the second case is the amount of metastable austenite transformed into martensite that is clearly obtained by analysing the combined XRD patterns of ADI samples before and after cryogenic treatment austempered at 400°C with time periods 2.5hr, 3hr and 3.5hr is demonstrated in Fig. 14, 15 and 16.Using the equation suggested by Cullity the proportion of austenite in the ausferrite matrix before and after cryotreatment found out by substituting the values of area under the XRD peaks. It shows drastic lowering of austenite percentage after cryotreatment shown in Fig 17.

The change in percentage of austenite after the cryotreatment shows that large amount of it transforms to martensite. Percentage of martensite is calculated by taking the difference between the amount of austenite before and after cryotreatment. It is illustrated in Fig 18. But, extent to which martensite getting transformed decreases as time of austempering increases.

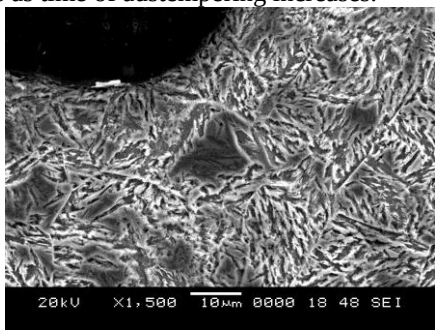

Fig. 12b. SEM photomicrograph of cryotreated ADI austempered at 340°C with austempering time of 3 hr

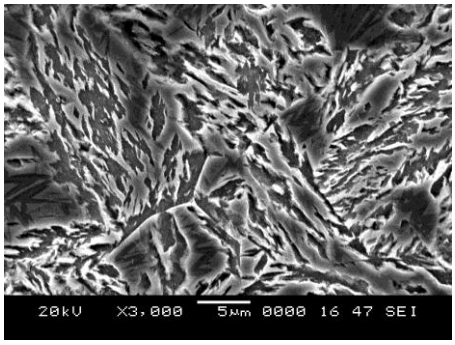

Fig. 13b. SEM photomicrograph of cryotreated ADI austempered at 340°C with austempering time of 3.5 hr

ISSN (Online) 2393-8021
ISSN (Print) 2394-1588
International Advanced Research Journal in Science, Engineering and Technology
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It is because at higher time duration of austempering more and more carbon would diffuse into austenite which would result in lowering temperature M_s below -196°C.

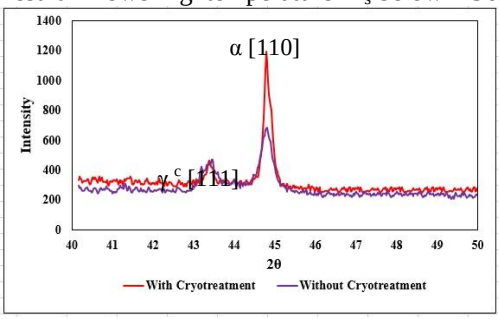

Fig. 14. Combined XRD profile of ADI before and after cryogenic treatment austempered at 340°C for 2.5hr. γ^c – Retained austenite, α- Ferrite

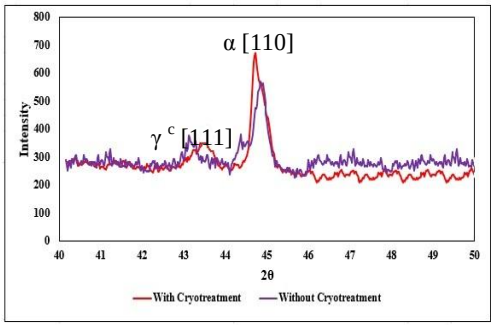

Fig. 15. Combined XRD profile of ADI before and after cryogenic treatment austempered at 340°C for 3hr. γ^c – Retained austenite, α- Ferrite

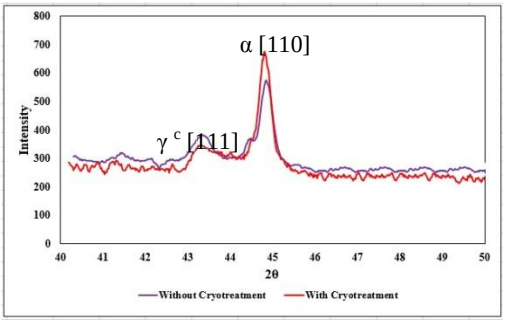

Fig. 16. Combined XRD profile of ADI before and after cryogenic treatment austempered at 340°C for 3.5hr. γ^c – Retained austenite, α- Ferrite

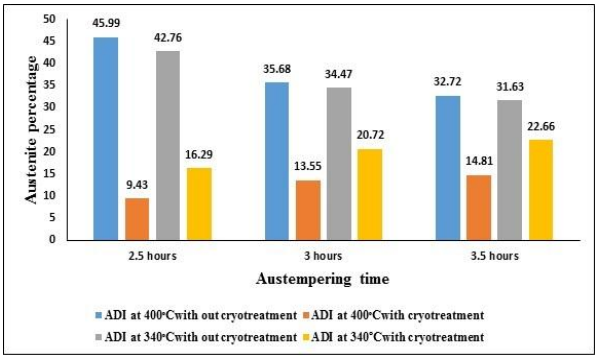

Fig. 17. Comparison of austenite percentage before and after cryotreatment

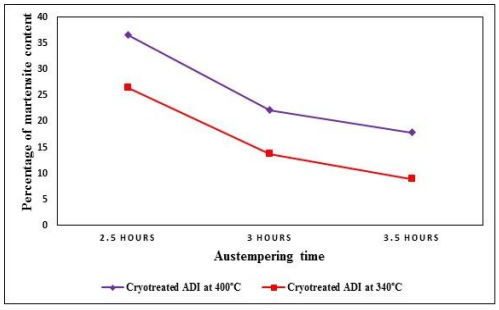

Fig. 18. Martensite content in percentage w.r.t austempering time

C. Vickers hardness test results

Hardness measurement is done for both untreated and cryo-treated test specimen. Vickers hardness measurement technique is used to determine the hardness of the ADI matrix. The hardness is determined by taking average of six readings. Hardness graph is shown in Fig. 19.Hardness values show that there is appreciable increase in the hardness of the matrix after cryotreatment. This is the implication of transformation of retained austenite to martensite.

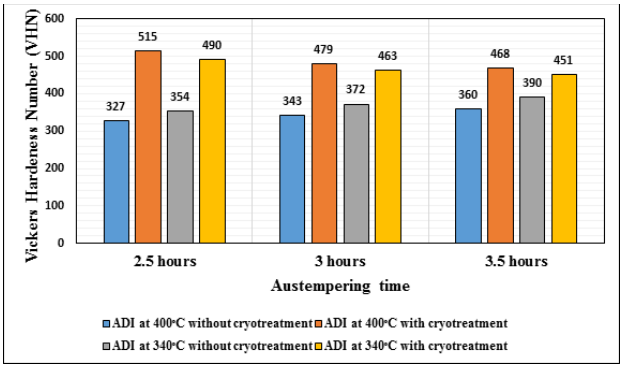

Fig. 19. Vickers hardness number before and after cryotreatment

D. Wear studies

The slope of cumulative weight loss versus time of sliding reported the wear rate of ADI samples. Table III represents the wear rate of ADI sample before and after cryotreatment under two different austempering temperature and three austempering time intervals.

TABLE III: WEAR RATE OF ADI SAMPLE BEFORE AND AFTER CRYOTREATMENT

Austempering temperature (°C)	Time (hours)	Wear rate (grams/minutes)×10^{-4}	
		Without cryotreatment	With cryotreatment
400°C	2.5	8.7167	4.9333
	3	6.3167	5.5083
	3.5	6.2444	5.5666
340°C	2.5	6.2916	5.1555
	3	6.0500	5.1917
	3.5	5.4917	5.4012

When the pin on the rotating disc starts wearing, the graphite nodules starts wearing, and the graphite nodules on the pin surface get distorted and forms a small hump on

ISSN (Online) 2393-8021
ISSN (Print) 2394-1588
International Advanced Research Journal in Science, Engineering and Technology
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its surface, due to greater deformation of the matrix over the graphite nodule under a given load 2Kg also due to compressibility of the graphite. The graphite nodules are drawn into stringers as seen in Fig 20, parallel to the wear surface, subsequently by fracturing at the leading edge of the pin, falls out to form wear debris there by causing pitting on the surface.

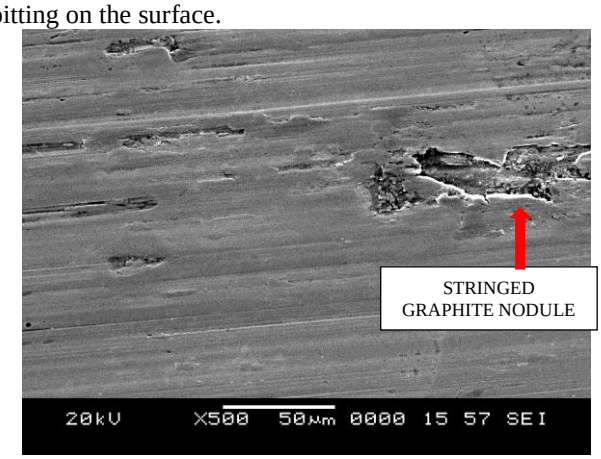

Fig. 20. Photomicrographs of worn out surface of ADI shows the stringed form of spheroidal graphite parallel to sliding surface.

In such conditions wear resistance is expected to be high because graphite act as a lubricant preventing any damage of the matrix. When the sliding time increases the asperities flow plastically and hang over the top of the pore created due to removal of graphite nodule by scuffing process, forming a tongue [12]. The wear surface showing plastic flow of asperities is shown in Fig. 21.

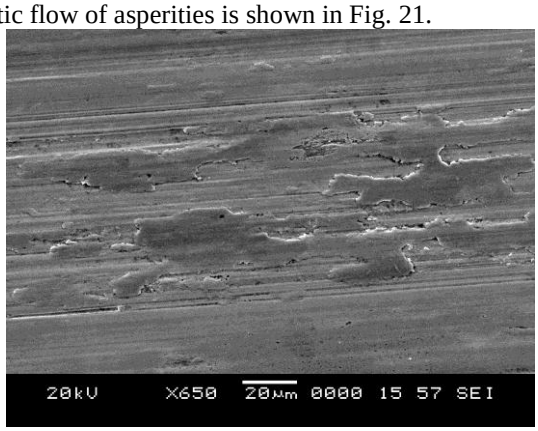

Fig. 21. Photomicrographs of worn out surface of ADI showing plastic flow of asperities

In the absence of graphite particles, matrix comprising of bainite and retained austenite are exposing to the sliding surface. Under such circumstances the wear resistance of the material depends on the morphology of bainite and behaviour of retained austenite. From the Table III it is observed that wear loss is minimum for samples austempered for longer periods because when the austempering time increases the amount of bainite increases which will withstand the wear conditions. It should be noted that delamination process being the most severe form of wear deterioration process, is associated with propagation of subsurface cracks as shown in Fig. 22. Delamination has taken place by the propagation and linking of voids nucleated by decohesion of matrix around distorted graphite.

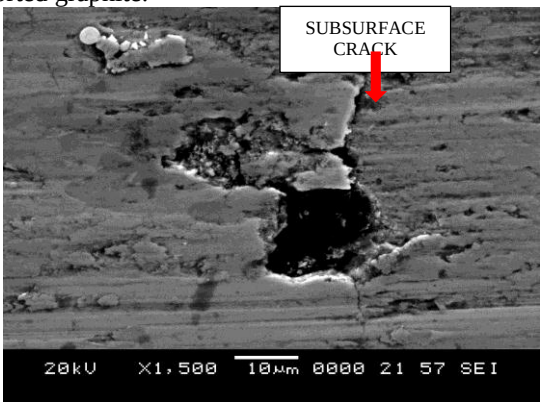

Fig. 22. Photomicrographs of worn out surface of ADI showing subsurface crack

By analysing the wear rate of cryotreated ADI samples shown in Table III, austempered at 400°C and 340°C with austempering time of 2.5, 3, and 3.5 hours it is found that lowest wear rate is observed for cryotreated ADI austempered at 400°C for 2.5hours because this sample is having the highest hardness compared to all other samples and also its microstructure contains considerable amount of martensite along with bainitic needles. The combination of highest hardness and microstructural constituents made it highest wear resistant material compared other ADI samples.

IV. CONCLUSION

The following conclusion were drawn from the present study on microstructural aspects and wear behaviour of heat treated cryotreated ADI

- The effect of cryogenic treatment on ADI is quite noticeable, its microstructure as well as hardness properties changed. ADI austempered at 400°C with austempering period of 2.5 hours showed highest hardness.
- Cryogenic treatment enables to transform the metastable austenite into martensite, but it is observed that the percentage of martensite transformed is more in ADI with high austempering temperature and low austempering time.
- Increasing austempering time increases the stabilisation of retained austenite for a given austempering temperature and increasing austempering temperature increases the amount of retained austenite for a given austempering time.
- For ADI samples before cryotreatment wear rate decreases with increases in austempering time for a given austempering temperature and it is found to increase with increase in austempering temperature.
- After cryotreatment wear rate of all the ADI samples decreased and the lowest wear rate is observed for cryotreated ADI austempered at 400°C for 2.5hour.

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