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Influence of Intercritical Austenitizing Temperature and Different Quenching Medium on Mechanical Properties and Wear Behaviour of Dual Matrix Structured Ductile Iron

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Abstract : An investigation was performed to study the effect of intercritical austenitizing temperature and quenching medium on the martensite volume fraction of ductile iron (DI) with dual matrix structure (DMS). For this purpose, alloyed ductile iron (3.61 C wt. %, 2.1 Si wt. %, 0.2 Mn wt. %) is partially austenitized in two phase region (a - γ) at temperatures of 785°C and 815°C and subsequently samples were quenched into two different medium water and oil. Dual matrix structure (ferrite -martensite) ductile iron with different martensite volume fraction was obtained. Samples were tested for ductility, tensile strength and wear phenomena. The result shows that the volume fraction of ferrite and martensite can be controlled to influence the strength and ductility. Optical micrographs and X-ray diffraction pattern shows that with the increase in intercritical austenitizing temperature (ICAT) and degree of cooling rate, martensitic volume fraction increases. The resistance to weight loss and tensile strength increased and ductility decreased with increasing martensite volume fraction. Among the samples, the highest weight loss was obtained for the sample austenitized at 785°C oil quenched; however the weight loss was approximately linearly to the applied load. The worn surface was studied under SEM photographs, it was found that wear mechanism is mainly caused by the delamination of the subsurface.

Keywords: Ductile iron, Dual matrix structure, Martensite volume fraction, Weight loss.

1. Introduction

The mechanical properties of ductile irons are controlled primarily by the amount and distribution of micro-constituents. The newly developed ductile cast iron with dual matrix structure consists of ferrite and martensite or ausferrite which is called dual matrix structure (DMS)[1,2,3].Infact, DI possesses a good combination of mechanical properties and there is an increasing demand in automotive industries for DI with increasing ductility without affecting the

strength of the material. Recent works have suggested that optimal ductility can be achieved through the development of dual matrix structure (ferrite-martensite or ausferrite) obtained by heat treatment [4]. The DI with DMS having ferrite and martensite structure was obtained by intercritically austenitizing the DI in two phase region($\alpha - \gamma$) at various temperatures followed by a fast cooling step to transform austenite into martensite. The two phase region is delimited by the upper and lower critical temperatures, where ferrite, graphite and austenite coexist. Such temperatures define the starting point at which ferrite transform into austenite and/or austenite into ferrite in heating and cooling processes respectively. Here oil and water are used as quenching medium to obtain different martensite volume fraction. This dual matrix heat treatment has an advantage of precise control of martensite and ferrite volume fraction[5].

With the increasing applications of the DI as a substitute for other cast iron and fabricated steels, the understanding of the wear behaviour of DI is crucial. Wear properties is not an intrinsic property of a material but depends upon the properties of the micro-constituents of the materials, test condition and environment [6]. Recently, several attempts have been made to study the wear behaviour of different class of DI.Behera et.al [7] studied the effect of percentage carbon equivalent (%CE) on mechanical properties of ductile iron for different section thickness through artificial neural network approach. In another study by Behera et.al [8] have investigated the effect of different heat treatment processes on mechanical properties of SG iron and reported that with increase in pearlite content the strength and hardness increases and ductility decreases. Islam et.al[9]studied the wear behaviour of as-cast and heat treated DI under dry sliding condition. The results show that the wear mechanism is mainly adhesive wear for heat treated samples and a combination of delamination and adhesive wear for ascast samples, this difference in wear behaviour lies in matrix structure of the DI. Zimba et.al [10] investigated the wear resistance of austempered ductile iron (ADI). It was concluded that wear resistance of ADI is much superior to that of the parent DI and almost same to that of steel whose hardness is approximately twice that of ADI.Sahinet.al[11]examined the wear behaviour of ADI with dual matrix structure (ausferrite) quenched in salt solution. It was found that the wear resistance increased with increasing ausferrite volume fraction and austempering time. In another study Sahin et.al[12]reports the effects of martensite volume fraction (MVF) and tempering time on the abrasive wear of ferritic DI with DMS. The conclusions drawn were that the wear of the tested samples increased approximately linearly with increasing applied load and decreasing

MVF.Movahed et.al [13]investigated the tensile properties and work hardening behaviour of dual phase (ferrite-martensite) steels. It was reported that dual phase steels with equal amount of ferrite and martensite shows excellent mechanical properties.

The wear studies carried in the past are mainly leaned towards the DI, cast iron and ADI's. However, a few attemptshave so far been made in determining the wear behaviour of DI with ferrite- martensite matrix by controlling the ferrite and martensite volume fraction. This study has been therefore, undertaken to investigate the influence of intercriticalaustenitizing temperature (ICAT) and different quenching medium on mechanical properties and wear behaviour of DI with DMS.

2. Experimental procedure

2.1 Sample preparation

The material used in the present investigation is a low alloy ductile cast iron; the chemical composition is detailed in Table 1.The material was originally cast in a bulk size at L&T Kansbahal, India. From these block, the tensile specimenswere sectioned as per ASTM standards E-8. Each of the sectioned specimens had a small amount of additional material left to account for any distortion resulting from heat treatment.

Table 1. Chemical composit	tion of employe	d ductile cast iron.
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Elements	С	Si	Mn	S	Р	Cr	Ni	Mo	Cu	Mg	Ce	Fe
Wt. %	3.56	2.07	0.17	0.009	0.018	0.03	0.44	0.001	0.009	0.038	0.003	Rest

2.2 Heat treatment

After fabrication the test specimens were intercriticallyaustenitized in two phase region ($\alpha - \gamma$) at various temperatures followed by quenching process. For the experiment, specimens were intercritically austenitized at temperatures of 785°C and 815°C and two quenching medium paraffin liquid light oil and water were selected for a detailed study of the development of DMS with different MVF and microstructural refinement. The parent matrix had a combination of ferrite and graphite structure. This microstructure was the starting point for subsequent DMS heat treatment. The heat treated samples were mounted and polished in accordance with standard procedures; all were etched with 2% Nital. The microstructure of all the samples was examined by optical microscopy.

The specimens were coded according to ICAT and different quenching medium. For example, in specimen code A785O, A785 stands for intercritical austenitizing temperature of 785° C and O stands for oil quenching. In specimen code A815W, A815 stands for intercritical austenitizing temperature of 815° C and W stands for water quenching.

2.3 Tensile and Hardness testing

After machining to final dimensions, tensile testing was carried out as per ASTM E-8. The test was performed on a UTM (Instron1195) with 100 KN loading capacity at a cross-head speed of 1 mm min⁻¹. All of the samples were performed in ambient air at room temperature. Load and displacement plots were obtained on an X-Y recorder, from these load-displacement diagrams, the ultimate tensile strength and % elongation values were noted.

The hardness was measured in Vicker hardness tester by applying 20 Kg load and dwell time being 10 seconds. All the samples were evaluated in the same condition. At least ten intends were made at each location and average values were taken.

Samples code	ICAT (⁰ C)	Quenching medium	Martensite Vol(%)	Ferrite Vol (%)	Tensile Strength (MPa)	Elongation (%)	Hardness (xHV50)
A785O	785	Oil	30	70	481	12.45	421
A785W	785	Water	73	27	590	11.01	515
A815O	815	Oil	62	38	538	11.8	484
A815W	815	Water	78	22	643	9.36	572

 Table 2. Metallographic measurements and mechanical properties of tested samples

XRD and Volume fraction analysis

To determine the volume fraction of ferrite and martensite for each heat treatment process an X-ray diffraction (XRD) method was used with monochromatic Cu-K α radiation (wavelength λ = 1.54A⁰) at 40 kV and 100mA. The recorded files were analysed to obtain the precise diffraction peak positions and integrated intensities of {111}, {200} and {211} planes of ferrite and martensite. The volume fraction of martensite and ferrite was determined by direct comparison method using the integrated intensities of the above peaks [14]. The volume fraction was determined by the following equations:

$$\frac{I_m}{I_f} = \frac{R_m C_m}{R_f C_f} \quad \text{and} \quad C_m + C_f = 1$$

where I_m and I_f are respective intensities of martensite and ferrite, R_m/R_f and is a constant dependable on θ hkl and kind of substance. C_m and C_f are respective volume fraction of martensite and ferrite. The XRD pattern of treated samples is shown in Fig. 1.



Fig.1: XRDplot for all tested samples

2.4 Wear test

Dry sliding wear test of the DMS ductile iron were carried out with a Ducom TR-208-M1 Ball-on-Plate type wear tester using a diamond intender. Unlubricated wear tests with a sliding distance of 7.54 m at a linear speed of 0.063 m/s were performed at room temperature with normal loads of 20N, 40N and 60N.A constant 4mm track diameter was used throughout the wear test. The weight loss for corresponding specimens was measured with the help of electronic balance of 0.1mg accuracy, prior to the weight measurement specimens was cleaned ultrasonically with acetone before and after the wear took place. Worn surfaces were examined by scanning electron microscope.

3. Result and Discussions

3.1 Microstructure and mechanical properties

Metallographic samples were taken from each of the heat-treated conditions. These samples were mounted and polished in accordance with standard

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procedures; all were etched with 2% Nital.At ICAT of 785^oC and 815^oC, the specimens are in the austenite-ferrite region $(\alpha - \gamma)$. On heating ferritic microstructure (as-cast) to ICAT, austenite nucleate at prior ferrite/ferrite grain boundaries which are present at the eutectic cells and then grew into ferrite. Also the presence of alloying elements (Ni and Mo) close the interval temperatures between A₁-A_f, (where A₁ is the minimal temperature to start austenite transformation during heating and A_f is the highest temperature at which ferrite is still stable) these leading to a higher austenite volume fraction due to higher nucleation and growth of the austenite phase [13]. Subsequently, on quenching of the samples from different ICAT produced DMS with different martensite volume fraction. The optical micrographs of treated DI samples are shown in Fig. 2.



Fig. 2: Microstructure of treated DIsamples (**a**) partially austenitized t 785° C and then water quenched, etchant,(**b**) partially austenitized t 785° C and then oil quenched,(**c**) partially austenitized t 815° C and then water quenched, (**d**) partially austenitized t 815° C and then oil quenched.F- ferrite and M- martensite.

Influence of intercritical austensitizing temperature (ICAT)

Considering the microstructure of the samples at different ICAT range with same quenching medium is discussed. The microstructure of the specimens shows a network of continuous and quasi-continuous martensite structure along the

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eutectic cell boundaries formed depending on the MVF. For the specimen A785W and A815W or A785O and A815O the MVF increases and FVF decreases with the increase of ICAT (Table 2). Because during ICAT heat treatment, in the austenite - ferrite region, the austenite volume fraction depends on the ICAT. When ICAT increases, the austenite volume fraction and its carbon content increases and ferrite volume fraction decreases as defined by the lever rule[2]. This means that the MVF and FVF can be controlled by using this heat treatment since parent austenite formed during partially austenitizing transforms into martensite on quenching.

The average value of UTS, hardness and elongation % are given in the Table 2. From the table, it is readily apparent that both the UTS and hardness value increased and elongation decrease with the increasing ICAT. The specimens at ICAT of 815^oC with its nearly wholly martensitic structure throughout the specimen havemuch higher tensile strength and low elongation values compared to the specimens at ICAT of 785^oC. Thevariation of the mechanical properties with ICAT is a good indication of the martensite carbon content due to the diffusion less nature of the martensite transformation from austenite. This result is in agreement with much of the existing literature, which indicates an approximately linear relationship with MVF and ICAT[15,16].

Influence of different quenching medium

The quenching of the samples from different ICAT in water and oil produced DMS with different martensite volume fraction. The water quenched samples are having a continuous network of martensite structure whereas the oil quenched samples shows a quasi-continuous network of martensitic structure. For the specimen A785W and A785O or A815W and A815O, the MVF of water quenched samples were higher compared to the oil quenched samples at the same ICAT. Since martensite transformation occurs at high cooling rates, this result may be attributed to the fast cooling process in water quenching medium compared to the oil quenching medium having relative density of 0.86. The reason for continuous network of martensite on water quenching (Fig. 2a and 2d) is due to the incremental increase in the MVF compared to oil quenched samples (Fig. 2b and 2d).

In the present study, oil quenched samples having high FVF shows greater elongation and low tensile strength compared to water quenched samples. Martensite particles are almost isolated in the ferrite matrix in the oil quenched samples. The degree of continuity of the martensite structure network along intercellular boundary could be an important factor in determining the degree of ferrite deformation along the graphite nodules. Therefore, ductility increases with

the decreasing continuity of martensitic structure along eutectic cell boundaries. The result demonstrates that the introduction of the ferrite is very effective for improving the ductility of DI with DMS. The ductility is very effective to the FVF. Total elongation also appears to be effected by variation in the degree of dispersion and size of second hard phase of martensite in ferrite matrix. This result is in good agreement with the existing literature [12, 17].

3.3 Fractography

Typical fracture surfaces of broken tensile specimens of DI with DMS are presented in the Fig.3.It reveals mixtures of cleavage and dimples for all the specimens. This type of fracture is called quasi-cleavage fracture. The observation of SEM fractographs of the oil quenched samples (Fig 3a and 3b)shows mainly dimple depression reflecting dominant ductile nature since ferrite fails in a more ductile fashion and its contribution to the fracture resistance increases with the increasing FVF. The fracture surface of the specimen A785O having the highest FVF is shown in Fig. 3a, the fracture mode reveals more uniform equiaxed dimple depressions along the graphite nodules compared to other specimens confirming the highest elongation %.Furthermore, the water quenched samples (Fig.3c and 3d) having low amount of FVF compared to oil quenched samples shows mainly the cleavage type of rupture morphology, expressing dominant brittle fracture[18]. In addition, fracture surface of the specimen A815W (Fig.3d) shows a micro-crack formed at the graphite-matrix interface reflecting high strength of the sample. This result is in good agreement with the mechanical properties of the DI with DMS.



Fig.3 : Typical fracture surface of broken tensile specimen of (a) A785O, (b) A815O, (c) A785W and (d) A815W at 500X.

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3.4 Wear Behaviour

he effect of varying loads on DI with DMS having different MVF and FVF were investigated. The weight loss of samples was determined and is presented graphically in Fig.4 as a function of applied load for samples with different ICAT and quenching medium.



Fig. 4 Graphical representation of weight loss as a function of applied load.

Influence of intercritical austenitizing temperatures (ICAT)

It could be seen in Fig. 4 that the weight loss of samples austenitized at 815^oC is lower than that of the samples austenitized at 785^oC with same quenching medium irrespective of all loads because of changes in the MVF and hardness. It means that the ICAT influences the wear behaviour of DI samples. This might be attributable to the increasing MVF and increasing hardness of the samples with the increase in ICAT[12].Furthermore, the weight loss of all the samples tested increased as the applied load was increased since an increase in the load might increase the contact stress, thus resulting in further surface damage.

Influence of different quenching medium

The lowest weight loss was obtained for the water quenched sample(A815W) compared to oil quenched samples. This result comes from the microstructure of this sample which consisted of a continuous network of martensite throughout the specimen having the highest MVF (78%). The highest

weight loss was obtained with increasing load for the oil quenched sample (A785O), due to the lower MVF (25%) effect of this sample. Itmay be due the increase of ductility with the decreasing continuity of martensitic structure along eutectic cell boundaries.

3.5 Worn surface



Fig. 5: Worm surface in SEM image (a) A785W at a load of 20N (b) A785W at a load of 40N (c) A785W at a load of 60N (d) A785O at a load of 20N (e) A785O at a load of 40 N (f) A785O at a load of 60 N (g) A815W at a load of 20 N (h) A815W at a load of 40 N (i) A815W at a load of 60 N (j) A815O at a load of 20 N (k) A815O at a load of 40 N (l) A815O at a load of 60 N.

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Fig. 5 shows the worn surface morphologies under various loads of the tested samples.It can be found that the wear is mainly caused by delamination of subsurface. Under lower applied load of 20N, the worn surface of all the tested samples presents almost smooth surface with small delamination crater. When the load increases to 40N, the worn surface shows rough surfaces with significant delamination damage. On increasing the load to 60N, the worn surface shows intensively severe delamination damage and more material pull-out was observed. Meanwhile, at 60N some oxidized particles are evident due to generation of heat at higher applied load. Obviously, the highest wear scar was observed for the sample A785O (Fig.5d-e)at each loads compared to other tested samples. The surface of the graphite nodule was almost covered by the deforming and smearing of the material which is the indication of more ductile nature. The worn surface for the sample A815W (Fig.5 g-i) shows a better maintained surface with relatively slight delamination due to higher hardness and increased MVF which resist the initiation and propagation of cracks. Moreover, this sample shows higher oxide formation (Fig. 6) at 60N load indicating more brittle nature of the material compared to other tested samples which is consistent with the fractography result.



Fig.6 EDAX analysis of the worn surface of A815W at 60N load of Fig. 5(i)

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4. Conclusions

In this study, the influence of intercritical austenitizing temperature and quenching medium on mechanical properties and wear behaviour of dual matrix structured ductile iron has been investigated. The following conclusions can be drawn:

- 1. In the specimens with dual matrix structure, for any combination of martensite and ferrite volume fractions, the amount of tensile strength and ductility can satisfactorily be optimized.
- 2. At different ICAT range, MVF increases and FVF decreases with the increase of ICAT in same quenching medium. Whereas, the MVF of water quenched samples are higher compared to the oil quenched samples at the same ICAT, since martensite transformation occurs at high cooling rates
- 3. The strength increases and ductility decreases with increasing MVF.
- 4. The different fracture mechanism corresponds to the different level of MVF and observed mechanical properties. With increasing the MVF the fracture pattern changes from ductile to moderate ductile.
- 5. The weight loss increased approximately linearly with the applied loads for all the tested samples. However, with the increasing load severe delamination damage has been observed and some oxidised particles are evident due to generation of heat at higher applied load.

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