Influence of High Mn-Cu-Mo on Microstructure and Fatigue characteristics of Austempered Ductile Iron

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Abstract. The impacts of high Mn content on microstructure and fatigue characteristics of ADI at 300, 350 and 400 °C for 120 min have been examined. Optical microscopy images reveals bainite morphology only at 300°C. Higher Mn contents hinders bainite transformation in the locales of Mn and Mo segregation, where in stage II reaction initiates near the graphite nodules before stage I reaction ends away from the nodules which creates more unreacted austenite volume after cooling forming martensite around the periphery creating austenite-martensite zone at 350 °C and tremendously articulated at 400°C. Feathery ferrite laths, stable retained austenite and uniform density hardness in the matrix, promotes higher toughness and fatigue properties (250 MPa @ 10^6 cycles) at 300 °C. Presence of stage II carbides in the eutectic cell and austenite-martensite zone in the intercellular regions, due to their embrittlement in the matrix, makes easy crack path for initiation and propagation deteriorating properties at 350°C and above. SEM images of fatigue fractured surface revealed that at 300°C, showed a regular crack interconnecting graphite nodule, fatigue striation and quasi-cleavage fracture mode, and at 350 & 400°C reveals the carbide, austenite-martensite and porosity/defect final fracture region.

1. Introduction

The revelation of ductile iron was another group cast iron family. By consolidating the properties of gray iron and steel [1]. Austempered ductile iron, where properties can be customized for applications requiring high tensile strength, wear properties and fatigue strength in the range 200-500 MPa. [2,3]. For the most part, it is required to add Mn, Mo, Ni and Cu for delving eligible hardenability in ADI. Mn, Cu, Mo and Ni are customarily alloyed ADI created so as to reduce the cost with cast steel and forged steel in automotive industry for gears [20] and wear resistance material [10], to broaden the use of ADI, having several advantages such as lower density, lower noise and work hardening of austenite [4,5]. Higher toughness prompts increase in fatigue strength [3]. In the vast majority of these reviews, combination plans depend on the conventional low-manganese idea. Alloying with higher Mn and enhancing the manganese level can altogether enhance the strength and hardness of ductile iron alongside increasing retained austenite content. From the past writing it is not clearly said about impact of higher manganese content on microstucture and fatigue characteristics of ductile iron. Along these, ductile iron with high Mn-Cu-Mo was concentrated their impact on microstructure and fatigue characteristics of austempered ductile iron.

2. Experimental procedure

The alloy choosen castings were prepared in Survail castings Mangaluru, produced in the form of slabs with dimensions 150*200*40 mm³. The as-received material was checked for its chemical composition by using Optical Emission Spectrometer, which was carried out at Gwasf quality castings, Mangaluru, India. Composition details are shown in the table 1. A dummy sample (14 mm diameter and 25 mm length) was also used along with the tensile and fatigue sample during heat treatment, which was helpful to carry out hardness and microstructural characterization before testing.
Austempering heat treatment samples were followed by austenitized at 900 °C for 45 minutes. The furnace temperature is controlled to ± 5 degrees of the set temperature value. Next step is austempering heat treatment at 300, 350 & 400 °C, which was carried out in a salt bath, which was controlled to ± 5 degrees of the set temperature value. The salt mixture consists of 55% KNO₃ and 45% NaNO₃ by weight. Etching was done with 2% nital solution. The microstructural characterization was done using AITM optical microscope. JEOL JSM-6380LA SEM was also used for fracture surface analysis of fatigue samples. X-ray diffractometry (XRD) analysis was carried out using JEOL-JDX-8P X-ray diffractometer. Scan range was in between 2θ- 40° to 50° at rate of 1°/min. XRD profile consisted of two peaks within this angular range namely the (111) for austenite and (110) for ferrite. Direct comparison method suggested by B.D. Cullity [25] was used to identify the respective volume fractions of austenite and ferrite. Shimadzu HMV-G 20ST micro-vickers hardness tester was used to determine hardness. Tensile specimens were followed as per ASTM E8M standards [26] testing was done on Shimadzu AG-X plus™ 100kN universal testing machine with strain rate 1mm/min. Fatigue Specimen were machined as per ASTM E 466- 82 [27], test was carried out in rotating bending four point loading designed machine at R= -1 as per ASTM E 466 [28] at 1440 RPM, 9 specimens were tested under each heat treated conditions to plot S-N curve.

Table 1. Chemical composition in wt%.

| Element | C   | Si  | Mn  | Ni  | Cu  | Mo  | P   | S   | Mg  |
|---------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| %       | 3.5 | 2.8 | 0.9 | 1.5 | 0.5 | 0.3 | 0.02| 0.02| 0.04|

3. Results and analysis

3.1 Optical Microscopy

Alloying with higher Mn and Cu, behave much as similar way during austempering. As represented in fig 1 (b-d) the ausferrite laths formed at (300 °C) austempering were very coarse, ferrite laths was less effective after austempered at (350 °C) and (400 °C) [10]. Generally higher Mn slower carbon diffusion, at higher temperature resulting in lesser amount of ferrite and more of untransformed austenite [6,7] which can be seen at 350 and 400 °C. During air-cooling, much of the intercellular unreacted austenite will change to martensite forming austenite-martensite zone. Addition of Mo causes microshrinkage porosity [11] in the intercellular areas at higher temperature 350 and 400 °C appeared in fig 1(c & d). Mn and Mo delays the stage I reaction away from the graphite nodules compare to the graphite nodules region, so much of untransformed austenite volume observed in the intercellular region observed at higher temperatures 350 and 400 °C, so as seen in micrographs large unreacted austenite which has not finished stage I due to the slower kinetics in the intercellular region due to high Mn and Mo [6,7]. MnC and Mo₃C carbides are formed in the intercellular region due to the addition of Mn and Mo above 350 °C shown in fig 1 (c-d).
Figure 1. Microstructure of sample (a) As-cast (b) Austempered at 300 °C (c) 350 °C (d) 400 °C for constant duration of 120 minutes.

3.2 Scanning electron microscopy

Figure 2. SEM images of samples (a) Austempered at 300 b) 350 c) 400 °C for constant duration of 120 minutes.

In fig 2 (a-c) SEM images show the feathery morphology of the ferrite laths is the most common at all temperatures, with edgewise growth during phase transformation. Twinned martensite was observed in figures 2 (b and c). Mn increased the retained austenite content due to their strong austenite-stabilizing effect, as well as blocky shape austenite [10].

3.3 X-ray diffraction (XRD) studies
Table 2 shows how the retained austenite was getting lean with carbon during austempering heat treatment at higher temperatures. At 300 °C due to slower carbon diffusion and effect of Mn segregation was less leading to the formation of stable austenite with higher carbon content. Reverse
situation was prevailed resulting in unstable/unreacted austenite at higher temperatures (i.e. 350 & 400 °C). Mn slow down the bainitic transformation near intercellular regions and the carbon content in austenite. At 400 °C due to the presence of more martensite on cooling at the intercellular region was confirmed with doublet peaks [12,13]. The driving force for the transformation therefore scales with $(C_\gamma - C^0_\gamma)$. C$_\gamma$ concentration in austenite is estimated empirically mentioned in [14].

3.4 Mechanical Properties

Table 2. Mechanical properties, Retained austenite/Unreacted austenite & carbon content.

| Sl No | Tin [°C] | Tensile stress [MPa] | Yield stress [MPa] | Strain [%] | Toughness [MPa] | UAV [%] | Carbon Content [MPa] | Fatigue Strength @ 10$^6$ cycles [MPa] |
|-------|----------|----------------------|---------------------|------------|-----------------|--------|----------------------|-------------------------------------|
| 1     | 300      | 1017                 | 819                 | 9.3        | 95              | 25     | 1.72                 | 260                                 |
| 2     | 350      | 780                  | 702                 | 3.4        | 24              | 54     | 1.25                 | 120                                 |
| 3     | 400      | 527                  | 480                 | 2.6        | 15              | 41     | 1.03                 | 95                                  |
| 4     | As-cast  | 760                  | 595                 | 4.6        | 35              | --     | --                   | 150                                 |

Note: Retained austenite at 300 °C, Unreacted austenite volume at 350 & 400 °C.

Table 2 shows the engineering stress-strain data’s. The experimental results indicated for as-cast in table 2, similar results were reported by [15]. High hardness was due to presence of pearlite content increased sharply when the manganese content reaches 0.9% which was due to some complex reaction occurred between Mn,Fe and C. hardness points HV$_{430}$ fig 1 (a) . The optimum toughness is obtained at temperature 300 °C.

It was observed that at lower temperature there is no much variation in hardness value with different hardness points HV$_{530}$ and HV$_{503}$ measured as shown in fig 1 (b). The mechanical properties (strength and toughness) of the ausferrite structure mainly depend on the microstructure. At a lower transformation temperature at 300 °C, the bainitic ferrite laths with some reduced grain size coarser with stable austenite and higher carbon content even some martensite present at 300 °C in the microstructure in 1 (a) showed a better strength and elongation in table 2 i.e. The martensite containing ADI can be treated as fiber reinforced ductile material. Even if the fiber is brittle, it will not make the composite brittle, as a beneficiary and will improve the toughness of the ADI [22]. Increased austempering temperature to 350 °C, decrease in tensile strength, increase in hardness which reduced elongation and toughness. Similarly at austempering temperature 400 °C, worst combination of strength and ductility was observed , which is due to strong Mn and Mo segregation and formation of much austenitic-martensitic zone during ausmtempering and carbide precipitation in eutectic cell and Mn$_2$C, Mo$_2$C HV$_{560,660}$ in intercellular regions reduced the toughness and strength values due to brittleness nature of carbides HV$_{800}$ and austenite- martensitic zone in 1 (c-d) [21].Cu content will increase elongation [16,17].

3.5 Fatigue study

Table (2) shows the fatigue results for as-cast and austempered samples. Fatigue strength for as-cast condition at 10$^6$ cycles was around 150 MPa, reduction in endurance strength due to the secondary grain boundary segregates. Fatigue strength increased at the austempering temperature 300 °C due to
the coarserness and a feathery bainite morphology with high carbon content stable austenite [24, 23]. The structure property correlation was evaluated depending on the retained austenite content, $X_c$, and its carbon content, $C_c$. The fatigue strength is related to toughness [18] and retained austenite content but not the tensile values, can be seen in table (2). Presence of austenite-martensite phase at 350 & 400 °C caused embrittlement and reduces the fatigue life [24]. Stage 2 carbide precipitates near the graphite nodules will reduce the fatigue strength table 2 above 350 °C. Fatigue strength is higher at 300 °C compare to 350 & 400 °C because of more toughness obtained where this satisfies the above empirical $S_e$ & $Q_i$ [1].

3.6 Fractography

The fracture surface of austempered ductile iron at 300, 350 and 400 °C resulting from four point rotating bending fatigue test. It is clearly indicating the change in the fracture surface morphology in all fig 3 (a-f).
Figure 3. SEM images of fatigue fractured samples austempered at 300 °C (a) Quazi-cleavage mode & Crack initiation at GN (b) Crack path in ausferrite matrix, at 350 °C (c) Transgranular cleavage (d) Carbide final fracture region, at 400 °C (e) Austenite-martensite final fracture region (f) Final fracture regions in the presence of defects/inclusions.

All the matrix showed brittle fracture in fig’s 3 (a-f). Even though there is some can also be seen clearly and discussed below. The fractographic image austempered at 300 °C shown in fig. 3 (a and b ) shows the regular striation and quasi cleavage mode. Crack is initiated at the graphite nodules and propagates through the austenite matrix [24] fig 3 (a and b). Fatigue strength decreased at 350 and 400°C due to the segregation effect of Mo and Mn as shown in fig 3 (c-f) , [24] low fatigue and tensile values of this matrix due to the carbide formation in the microstructure by the second reaction 3 (d) represents sample at 350 °C resulted in the carbide fracture. Due to the increase in the embrittlement of austenitic-martensitic zone, causes a transgranular type of fracture observed 3 (c) and fig 3 (e) represents carbide fracture [19] at 350 °C and fig 3 (e) cracks in austenite-martensite zone [8] observed in samples austempered at 300 °C. Due to the poor surface quality of specimens resulted in final fracture shown in fig 3 (f).

4. Conclusions

Alloy containing high Mn-Cu along with Mo austempered at 300, 350 & 400 °C for 120 min resulted in bainite morphology only at lower temperature 300°C with slight coarser ferrite .At higher austemperaturer temperature 350 & 400 °C, that the stage 2 reaction starts near the graphite nodules before the stage 1 reaction is completed away from the graphite nodules for austempering time 120 min. At higher temperatures 350 & 400 °C , and the effect of slower kinetics for higher manganese, retarding the carbon diffusion and bainite transformation rate along with Mo resulted in formation of more untransformed austenite volume upon cooling transfers to martensite in the region away from the graphite nodules. Austenite-martensite zone was observed at austempering tempering 350 & much more pronounced surrounding the martensite at 400°C. Due to the brittleness in nature of austenite-martensite zone, stage 2 carbides ,MnC ,Mo3C and much variation in matrix hardness ,the mechanical properties (i.e Tensile strength, toughness decreases. Fatigue strength increased at 300 °C due to the coarser bainite, presence of fine martensite and higher carbon content stable retained austenite resulting fatigue endurance of 230 MPa @ cycles. Fatigue strength decreased at temperature 350 & 400°C, due to the carbide precipitation and austenite-martensite zone provided an easy crack path which decreased fatigue strength. SEM images of fractured surface revealed that specimen austempered at 300 °C showed a regular striation and quazi-cleavage fracture , and samples
austempered at 350 & 400 °C showed the carbide, austenite-martensite and inclusion sub surface final fracture region.

5. References

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