Journal of Materials and Environmental Science
ISSN : 2028-2508
CODEN : JMESCN

J. Mater. Environ. Sci., 2020, Volume 11, Issue 7, Page 1150-1162

http://www.jmaterenvironsci.com

Evaluation of Fracture toughness of Permanent moulded Austempered Ductile Iron using single edge notched bending Specimens

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Received 22 May 2020,
Revised 21 June 2020,
Accepted 25 June 2020

Keywords
- ✓ Fracture Toughness,
- ✓ PMADI,
- ✓ SENB,
- ✓ Austenite,
- ✓ Bainite

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Abstract
Austempered Ductile Iron (ADI) castings have been increasingly utilized as a major engineering material in recent years by the automotive and power plant industries. Researchers have been aiming on enhancing the fracture toughness (K_{IC}) of ADI to compete with forged steels and steel alloys. Ductile Iron cast in permanent moulds have been found to possess superior strength and wear characteristics. This work focuses mainly on the fracture toughness studies of permanent moulded Austempered Ductile Iron (PMADI). Single edge notch bending (SENB) samples austempered at 300^{0}C, 350^{0}C and 400^{0}C for 150 minutes were subjected to three point bending test to evaluate the plane strain fracture toughness and the same has been compared with sand cast counter parts. The results indicated that PMADI samples austempered at 350^{0}C was found to possess about 8% and 12% increase in fracture toughness as compared to PMADI samples austempered at 300^{0}C and 400^{0}C. The samples poured into permanent moulds showed an increase of about 16% in the fracture toughness as compared to its sand cast counter parts. The results were analysed based on the distribution of Bainite and retained austenite.

1. Introduction
ADI has emerged as a major engineering material in recent years for automotive, earth moving, mining and power plant applications because of its superior mechanical properties. ADI belongs to ductile family of Iron where the percentage of carbon is 3.2% to 3.6%.The first step in producing ADI is Development of Nodular Iron with good nodularity poured into either sand or permanent moulds. The next step is Nodular Iron is subjected to special heat treatment process at different austempering temperatures and time. ADI is microstructure sensitive and the microstructure of ADI reveals that Austenite is converted to Bainite with some retained Austenite [1, 2]. ADI is competing fiercely with forged steel and aluminium alloys in terms of mechanical properties, manufacturing cost, physical properties and weight savings [2].
The automotive industry has recognized the potential benefits of austempering solutions many years ago. Typical examples include diesel engine timing gear, suspension brackets, gear housings, wheel hubs and sprockets [3]. The utilization of permanent moulds offers better advantages in the form of lower environmental pollution, excellent dimensional stability and higher production rate [4]. Seetharamu et al. [4] have reported that permanent moulded ADI castings have superior abrasion and

jet erosion resistance than sand moulded counter parts. Rundman [5] has suggested utilization of permanent moulds to produce ADI so as to minimize the negative effects of alloy segregation.

Jiwang Zhang et al. [6] highlighted the influence of microstructure on mechanical properties of ADI and concluded that, with the decreased austempering temperature, yield strength and tensile strength of the ADI increased where as impact energy and percentage elongation decreased. Bingxu Wang et al. [7] highlighted the importance of austempering process for ADI and concluded that austempering is an effective method to enhance the mechanical properties. S.K Swain [8] highlighted the phase investigation of ADI using XRD analysis at different austempering temperatures and concluded that, the maximum intensity of the austenite line is increased with increased temperature but ferrite line is increased with increased austempering time and decreased with austempering temperature. Uma et al. [9] reported the influence of nickel on mechanical and slurry erosive wear behaviour of permanent moulded toughened austempered ductile iron.

Fracture Mechanics is used to evaluate the structural integrity of the component in the presence of a crack or flaw. Fracture mechanics has been used heavily in the aerospace, automotive, nuclear and ship industries with a recent extension to the ground vehicle industry. Fracture mechanics uses the stress Intensity Factor (K_I), Energy release rate (G), Crack opening displacement (COD) and the J-Integral [10]. The catastrophic failure of mechanical component occurs when the stress intensity factor exceeds the fracture toughness of the material. Fracture testing of materials is an integral part of fracture mechanics for modern engineering design. This essentially involves the determination of fracture toughness and fatigue crack propagation characteristics. Fracture testing is an extension of conventional procedures on samples containing cracks. Plasticity effects can be constrained by designing fracture specimens to exhibit plane strain conditions [11].

The actual load-carrying capacity in the presence of a crack is popularly known as the "Fracture Toughness (K_{IC}). Stress Intensity factor (K_I) is a unique design parameter which represents the magnitude of stress field severity near a crack tip. When the stress intensity factor (K_I) exceeds the fracture toughness (K_{IC}) of the material, It results in sudden and catastrophic failure [11]. The critical value of K_I denoted by K_{IC} commonly referred to as fracture toughness, depends on the material. Linear elastic fracture mechanics (LEFM) of a material is calculated from the stress intensity factor (SIF) at which a thin crack in the material begins to grow [11].

Dragi Stamenkovic [12] highlighted the importance of stress intensity factor in a cracked structural component and illustrated the importance of J integral to analyze the same. Ayman H Elsayed et al. [13] reported the influence of both conventional and two step austempering process to achieve improved fracture toughness and mechanical properties of both alloyed and unalloyed ADI. Saranya Panneerselvam et al. [14] studied the influence of intercritical austempering process on the microstructure and mechanical properties of ADI considering different austenitization and austempering temperatures. A.A Abioye [15] studied the microstructure and mechanical behaviour of ADI and concluded that, austempering operation has a significant effect on the mechanical and microstructural properties of ADI. Ananda Hegde et al. [16] studied the influence of austenitizing temperature, alloying element, Austempering temperature and time and concluded that, the heat treatment parameters has a significant effect on the microstructure and mechanical properties of ADI. Susil K Putatunda et al. [17] highlighted the influence of austenitizing temperature on the fracture toughness of ADI and concluded that, austenitizing temperature above 982^0C has a detrimental effect on the fracture toughness.

The literature review reveals that, the considerable research has been carried on evaluation of mechanical properties and wear characteristics of ADI. However, the information regarding the fracture toughness characteristics of PMADI samples is scanty. Experiments have been planned systematically to study the mechanical properties and fracture toughness characteristics of PMADI samples. The effect of austempering temperature, time on ADI poured into permanent moulds has been planned and the results of fracture toughness were compared with their sand moulded counter parts. Analyzing the better fracture toughness characteristics helps industries to enhance the life expectancy of Mining, Agricultural, Power plants and marine applications.

2. Experimental Procedure

2.1. Material

Nodular Iron castings [Grade–EN– GJS–400/15] with good nodularity were melted in a coreless induction furnace of 50kg capacity. The melt was superheated to 1520^{0}C with Fe - Si – Mg alloy was used as nodularizer and ferrosilicon was used as inoculant. The melt was poured at 1450 – 1480^{0}C into a pre-heated (220^{0}C) moulds made of grey cast iron and also into green sand moulds with the help of patterns after de-slagging.

The castings obtained had dimensions of 60mm X 60mm X 550mm utilizing permanent moulds and and 30mm X 60mm X 460mm utilizing sand moulds. Both sand moulded ADI (SMADI) and PMADI castings were stripped from the mould at room temperature. The samples were prepared by utilizing conventional machining processes as per the ASTM standards to analyze the fracture toughness characteristics and other mechanical properties. The specimen for the metallurgical examination is as per ASTM standards (ASTM E3 and ASTM E7). The presence of nodules in nodular iron is confirmed by metallurgical microscope. Figure.1 clearly shows the photo micrograph of Nodular Iron (As cast) utilizing optical microscope in etched condition using 2% NITAL solution and it is clear that, the graphite structure is in the form of well-formed nodules in a ferrite matrix and also the Nodular Iron structure is like a bull's eye structure in a ferrite matrix. The microstructure shows a ferrite matrix with the pearlite and graphite nodules dispersed in it.

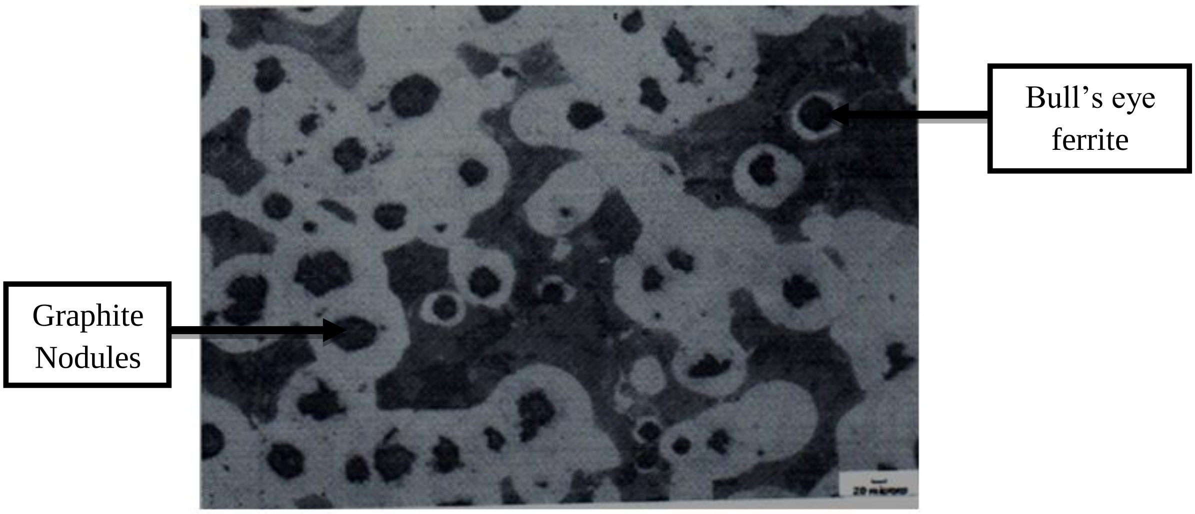

Figure 1: Microstructure of as cast Nodular Iron

The nodule count of the samples are determined as 120 – 140 number/mm^2 for sand moulded and 180 – 200 number/mm^2 for permanent moulded As-cast Nodular Iron. The nodular iron is confirmed by the presence of carbon between 3.2 to 3.6%. Table.1 clearly indicates the chemical composition test for unalloyed Nodular Iron utilizing optical emission spectrometer.

Table 1: Chemical Composition of ductile iron casting

Element in wt%	C	P	S	Si	Mg	Fe
Nodular Iron (As cast)	3.493	0.013	0.006	2.704	0.044	93.74

2.2. Heat Treatment Process

The next step is nodular iron is subjected to unique heat treatment process called Conventional Austempering process.

The conventional Austempering process planned for PMADI samples can be divided as follows:

- All the samples were austenitized to 900°C for 120 Min duration
- Samples were quenched in a salt bath furnace consisting a mixture of 45% sodium nitrate and 55% potassium nitrate by weight and subjected to Isothermal heat treatment at the Austempering temperature of 300°C, 350°C and 400°C for 150 min duration.
- Cooling to room temperature (Air cooled to room temperature)
- SMADI samples were austenitized to 900°C for 120 Min duration and austempered at 350°C for 150 min duration.

2.3. Microstructural Analysis of SMADI and PMADI samples

The specimen for the metallurgical examination is as per ASTM standards (ASTM E3 and ASTM E7). The microstructure of sand moulded ADI and PMADI samples in etched condition using 2% NITAL solution and the same has been analysed using electron microscope.

Figure.2 (a) shows the representative photomicrograph of sand moulded ADI structure (SMADI) and matrix microstructure reveals the distribution of Bainite is minimum with lower level of retained austenite (RA: 17.5%). Figure.2 (b) shows the photomicrograph of PMADI - 300 structure austempered at 300°C for 150 Min duration reveals the presence of lower Bainite and retained austenite (RA:27.5%). Figure.2 (c) shows the photomicrograph of PMADI - 350 structure austempered at 350°C for 150 Min duration reveals the presence of feathery Bainite with a maximum level of retained austenite (RA: 32.5%) and Figure.2 (d) shows the photomicrograph of PMADI - 400 structure austempered at 400°C for 150 Min duration and matrix microstructure reveals the presence of martensite with a retained austenite (RA: 22.5%).

Table 2: Microstructural Details of SMADI and PMADI samples

Sample Designation	**Graphite – Nodule count (number / mm^2)**	**Nodule size (ave) μm**	**Retained austenite (RA) (Vol %)**
*SMADI	200 - 240	4	15 - 20
PMADI - 300	280 - 300	5.5	25 - 30
PMADI - 350	350 - 380	8	30 - 35
PMADI - 400	260 - 280	5	20 - 25

*represents sand moulded ADI (Austempered at 350^{0}C for 150 Min duration)

Table.2 shows the microstructural observations such as nodule count, its size and retained austenite content (RA) of SMADI and PMADI castings.

3. XRD Analysis

X – ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions [8]. A diffractometer is a measuring instrument for analysing the structure of a material from the scattering pattern produced when a beam of radiation or particles such as X – rays or neutrons interacts with it

[8]. X-ray diffraction analysis was performed to confirm the presence of acicular ferrite and the high carbon retained austenite. [19]. In the present investigation, Powder X-ray diffractometer (Bruker make) analysis using a monochromatic cu K_α radiation is performed to estimate the volume fraction of retained austenite in the ADI after heat treatment process. A typical X-ray diffraction pattern of a PMADI – 300, PMADI – 350 and PMADI -400 samples are clearly presented in Figure.3 (a), 3 (b) and 3 (c). The peak positions are represented as 1, 2 and 3 and the integrated intensities of (200) plane of ferrite (α phase), (220) (γ phase) plane of austenite, and (211) plane of ferrite (α phase) were observed from all the profiles. Considering XRD analysis of PMADI – 300, PMADI – 350 and PMADI – 400, Peak 1 represents Ferrite phase (200) at 2θ of 44.6^0, peak 2 represents Austenite phase (220) at 2θ of 65.3^0, peak 3 represents ferrite phase (211) at 2θ of 82.1^0, and the same is clearly depicted in Figure.3 (a) , 3 (b) and 3 (c). The presence of retained austenite was confirmed from the XRD analysis.

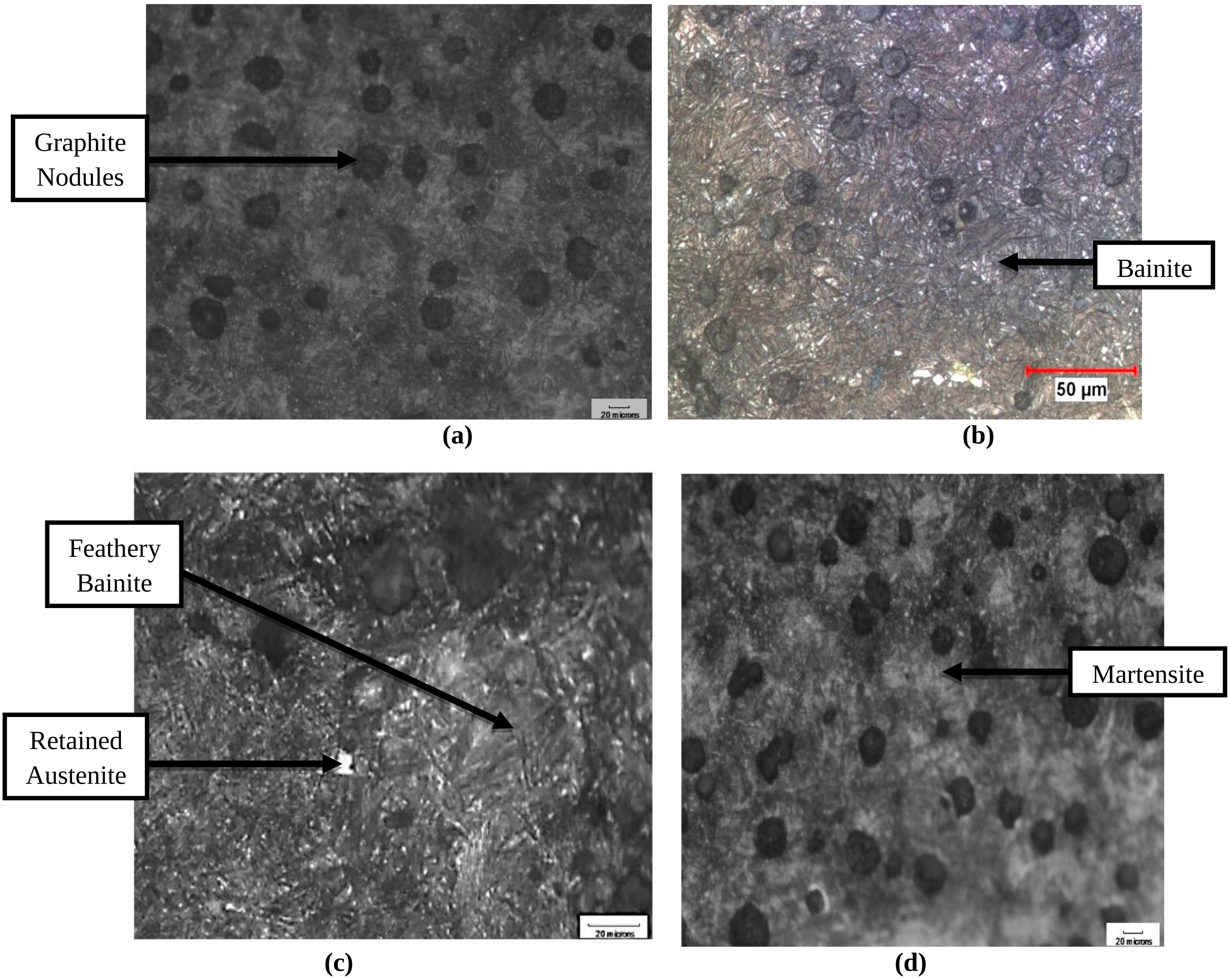

Figure 2: (a) Austempered structure of SMADI sample (b) Austempered structure of PMADI – 300 (c) Austempered structure of PMADI – 350 (d) Austempered structure of PMADI – 400

The retained austenite (vol %) was found to be maximum for PMADI – 350 sample and the same is clearly depicted in the Figure. 3 (b). The detailed results of retained austenite (vol %) were clearly tabulated in the Table.2.

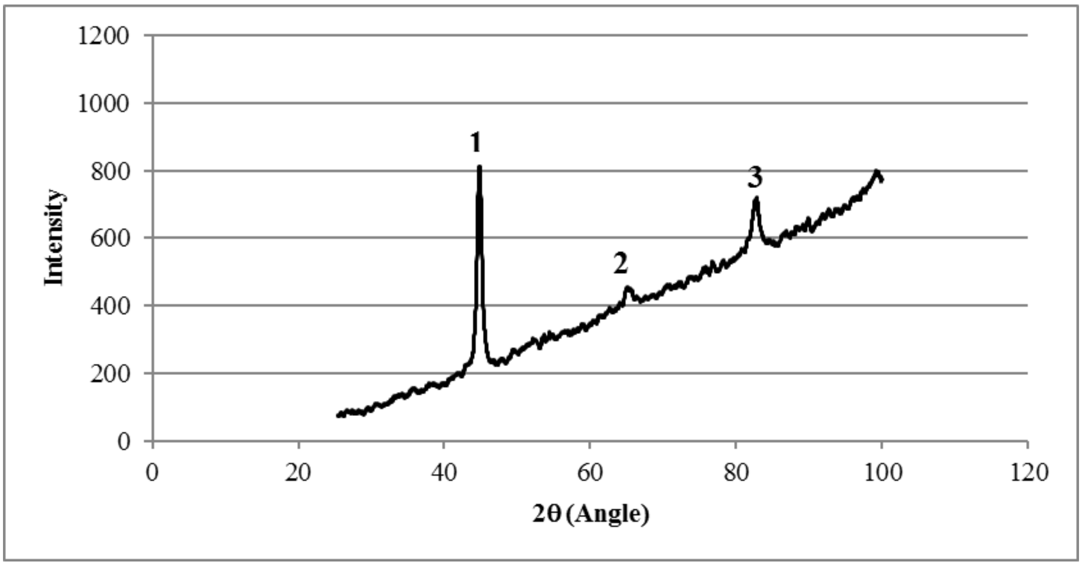

Figure.3 (a): XRD Analysis of PMADI – 300 sample

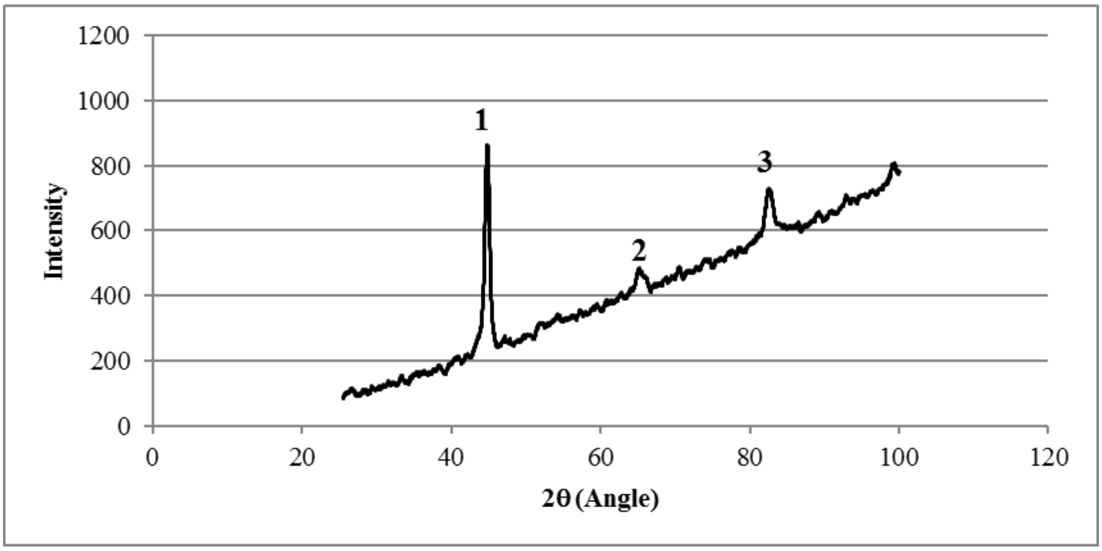

Figure.3 (b): XRD Analysis of PMADI – 350 sample

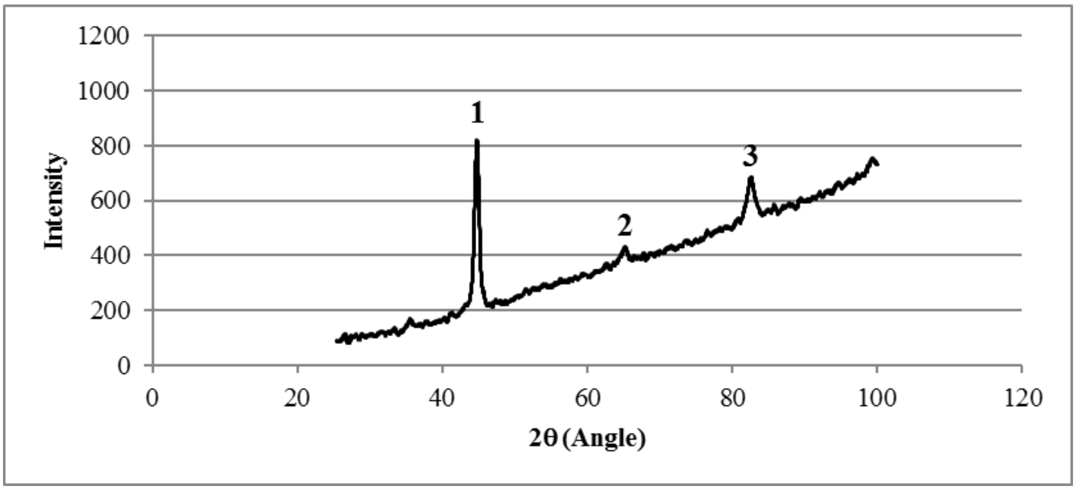

Figure.3 (c): XRD Analysis of PMADI – 400 sample

4. Fracture Toughness Test

Fracture testing of materials is an important part of fracture mechanics for modern engineering design. Fracture testing is an extension of conventional procedures on samples containing cracks [13].

The main dimensions are the crack length (a) and the width (w). The specimen thickness (B) is sufficiently large in comparison with the plastic zone size to ensure plane strain testing conditions. Specimen of size 50mm X 25mm X 220mm, span (S = 4w) = 200mm and notch size of 19 mm as per ASTM standard ASTM E 399 [18] as depicted in Figure.4. For fracture toughness test, an average readings of two samples were recorded and reported.

Axial servo-hydraulic fatigue testing machine (INSTRON 8801) of capacity 100 kN is used to create Fatigue pre-cracking and the crack prepared is 1mm for crack to width (a/w) ratio =0.40 in accordance with the ASTM standard E 399. Extreme care should be taken to apply small levels of fatigue loading. The fracture toughness test is carried out on SENB specimens subjected to three point bending test on computerized universal testing machine (UTM).

Load vs Displacement curve were utilized for a/w ratio of 0.40 to record the reading of critical load (P_q). The Load vs Displacement readings were recorded for SMADI, PMADI-300, PMADI-350 and PMADI-400 samples and the curve obtained was Type III [18] curve. The critical load (P_q) is equal to fracture load (P_f) in case of Type III curve. The load deflection records were evaluated to determine the fracture toughness.

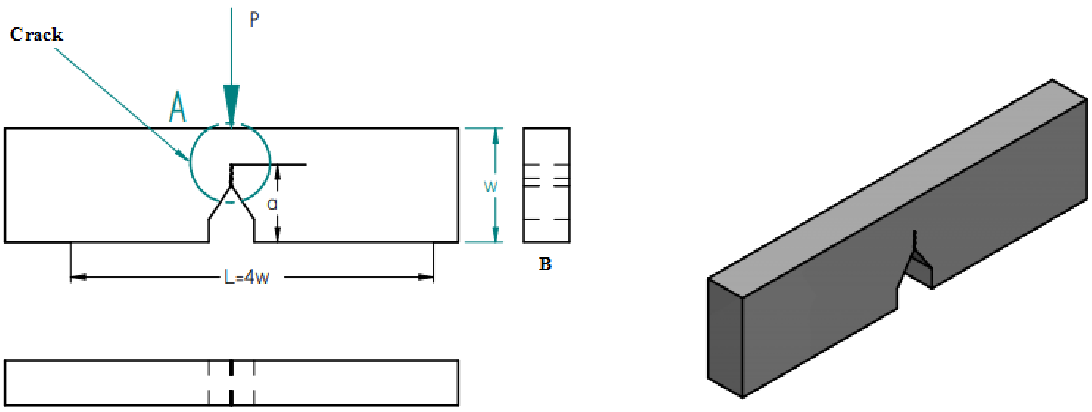

Figure 4: SENB Specimen

The Fracture Toughness (K_{IC}) is evaluated utilizing the formula (A) and the experimental results are clearly tabulated in Table.3.

$$K_{1C} = \frac{P_q S}{B w^{\frac{3}{2}}} f_2\left(\frac{a}{w}\right) \quad (A)$$

$$f_2\left(\frac{a}{w}\right) = \frac{3\left(\frac{a}{w}\right)^{\frac{1}{2}}\left(1.99 - \frac{a}{w}\left(1-\frac{a}{w}\right)\left(2.15 - 3.93\left(\frac{a}{w}\right) + 2.7\left(\frac{a}{w}\right)^2\right)\right)}{2\left(1+\frac{2a}{w}\right)\left(1-\frac{a}{w}\right)^{\frac{3}{2}}}$$

$f_2\left(\frac{a}{w}\right)$ is the geometry correction factor [18]

Considering the dimensions as follows, $a = 20mm, w = 50mm, B = 25mm, S = L = 4w = 200mm$

The value of K_{IC} is calculated from crack length and critical load (P_q). ASTM E 399 standards are followed to conduct the fracture toughness test [18]. In computerized UTM, the SENB samples are subjected to bending test. The fracture load (P_f) is recorded for a/w ratio of 0.4. The fracture load (P_f) acquired is used for consideration to determine the fracture toughness utilizing the empirical Eq. A.
Figure.5 shows the load – displacement plot for SMADI, PMADI-300, PMADI-350 and PMADI-400 samples. It was found that as load increases, the displacement also increases. The maximum displacement recorded was 1.5mm for PMADI – 350 sample.

Table 3: Fracture Toughness results

Casting Designation	a/w ratio	Fracture Load Average Value (P_q) (kN)	Fracture Toughness (K_{1c}) (MPa√m)
SMADI	0.40	47.34	67.12
PMADI - 300	0.40	50.86	72.12
PMADI - 350	0.40	55.12	78.16
PMADI - 400	0.40	49.20	69.76

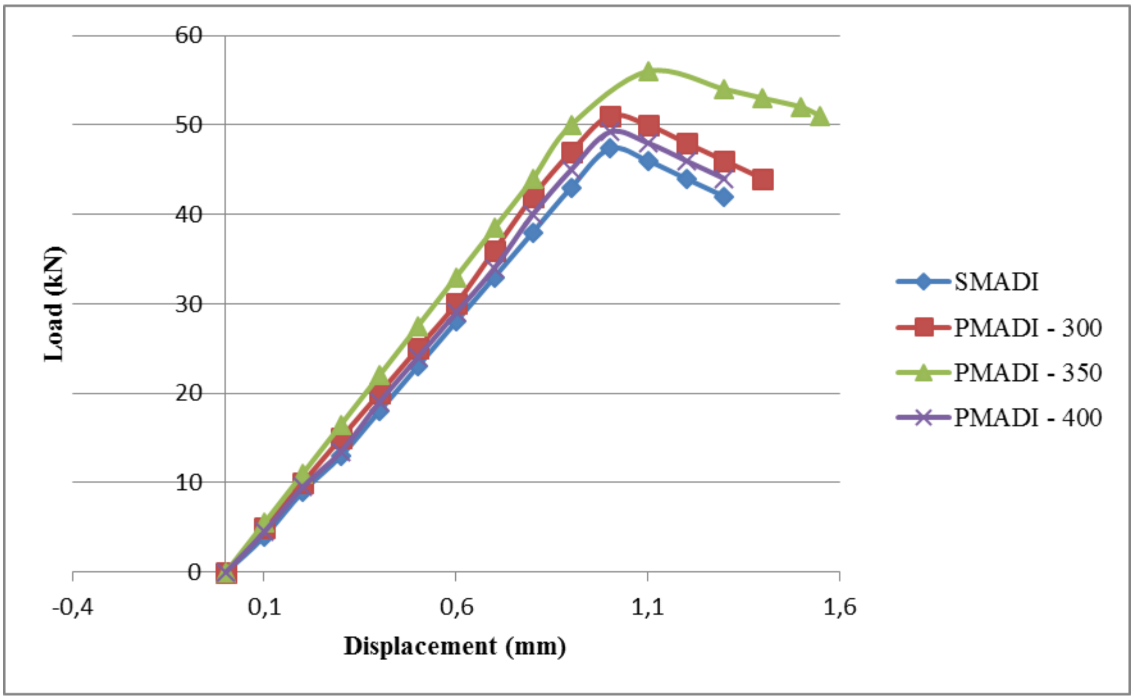

Figure 5: Load Vs Displacement graph

Table.3 depicts the fracture load and fracture toughness values of SMADI, PMADI – 300, PMADI – 350 and PMADI - 400 samples considering a/w ratio of 0.40.

4.1. Hardness Testing

Hardness test was performed utilizing Brinell hardness tester (Venus Instruments make – VAB - 250) with applied load of 250 Kgf as per ASTM standards ASTM E10-18 for the samples austempered at 300^0C, 350^0C and 400^0C for 150 minutes duration. Three readings were taken for each specimen and the results were averaged and the same is depicted in Table.4.
The Ball Indenter diameter (D) of 5mm and dwell time of 12seconds was selected for all the samples. The indentation diameter (d) was analysed utilizing Brinell microscope with the help of the indentation created on the specimen.

The Brinell hardness number as per ASTM E10-18 was evaluated by utilizing the equation (B)

$$BHN = \frac{2F}{\pi X D\left(D - \sqrt{D^2 - d^2}\right)} (B)$$

From the test results depicted in Table.4, the maximum hardness was found to be for PMADI sample austempered at 400^0C for 150 Min duration.

Table 4: Hardness Test Results

Casting Designation	Avg Indentation dia (d) mm	Brinell Hardness Number (BHN)
PMADI- 300	0.96	341.90
PMADI- 350	1	290.69
PMADI- 400	0.91	380.77

4.2. Tensile Testing

The tensile properties of PMADI samples like ultimate tensile strength (UTS) and percentage elongation were evaluated using a computerized universal testing machine of 400 kN capacity (Venus instruments make). The test was carried out as per ASTM standard E8M specifications. Uni-axial load is applied to the specimen and Load vs. Displacement was recorded. An average of two measurements were recorded and reported. The important tensile test properties of PMADI sample austempered at 300^0C, 350^0C and 400^0C for 150 minutes duration are tabulated in Table.5.

Table 5: Tensile test results of PMADI sample

Parameters	PMADI - 300	PMADI - 350	PMADI - 400
Ultimate Tensile strength (UTS)	1100 MPa	1050 MPa	1168 MPa
Percentage Elongation	4.5%	5.2%	3.8%

From the test results depicted in Table.5, the maximum ultimate tensile strength was found to be for PMADI sample austempered at 400^0C for 150 Min duration.

4.3. Impact Toughness Test

The impact test was conducted on the PMADI sample austempered at 300^0C, 350^0C and 400^0C for 150 minutes duration using a pendulum type drop weight impact tester (Venus instruments). The preparation of the specimen is as per ASTM E23 standard. The samples of size 10mm X 10mm X 55mm were prepared for this purpose. The V-notch of size 2mm is prepared and the energy required to fracture the sample was determined considering the impact velocity of 3.99 m/sec. A test piece notched in the middle and simply supported at each end. The notch acts as a point of stress concentration for high velocity impact blow. The details of the results are tabulated in Table.6. The average of two readings were taken for all the samples and analysed.

Table 6: Impact test results

Casting Designation	PMADI - 300	PMADI -350	PMADI - 400
Impact Toughness (Joules)	**71.4**	**83.2**	**68.2**

From the test results depicted in Table.6, the maximum Impact toughness was found to be for PMADI sample austempered at 350^0C for 150 Min duration.

5. Fractography Studies

In the present investigation, the fracture surfaces were examined on a Hitachi S 3500 Model scanning electron microscope (SEM) using an accelerating voltage of 10 kV to determine the mode of failure. Figure.6(a) exhibits the fracture surface morphology of SENB specimnes austenitized at 912^{0}C for 2hrs, austempering temperature 350^{0}C for 150 Min duration indicates a river pattern in the fracture and also, It is clear that PMADI - 350 sample indicated ductile fracture due to the presence of large number of knots. These features indicates the improved fracture toughness characteristics achieved for the PMADI sample austempered at 350^{0}C for 150 Min duration.

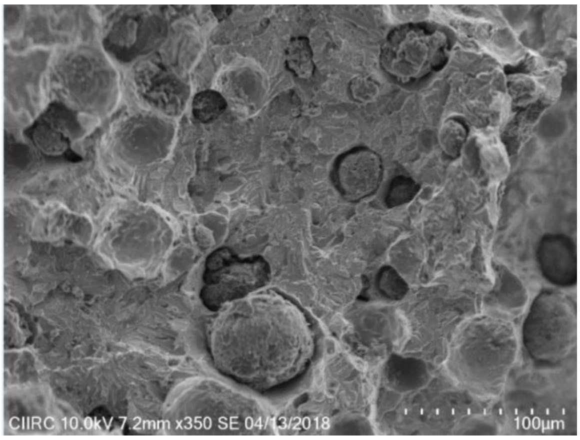

Figure 6(a): Fractography of PMADI - 350 sample

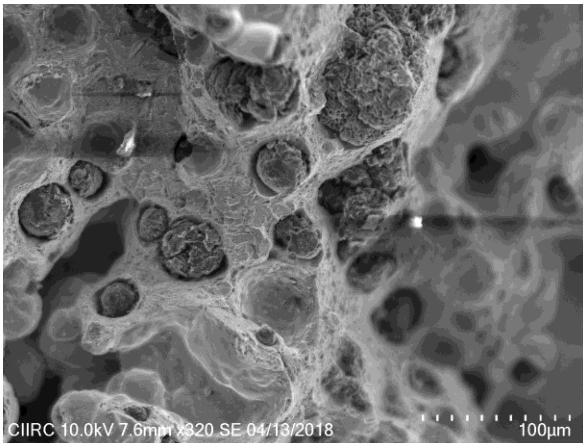

Figure 6(b): Fractography of PMADI - 400 sample

Figure.6 (b) exhibits the fracture surface morphology of SENB specimnes austempered at 400^{0}C for 150 Min duration indicated brittle fracture as there was very little elongation before fracture.

6. Discussions

The nodule morphological features such as counts and its size, volume of retained austenite and also the distribution of Bainite are responsible for the enhancement of fracture toughness characteristics.

Conventionally ADI is cast in sand moulds. However, the utilization of permanent moulds offers better advantages in the form of higher production rate, excellent dimensional stability and lower environmental pollution. Utilizing permanent moulds, the graphite nodules are more finer and denser in comparison with sand moulds. [4]

Uma etal [9] also reported the role of permanent moulds in enhancing nodule size and nodule distribution during solidification process. The nodule size, and also the graphite morphology favors the enhancement of fracture toughness for PMADI sample austempered at 350^{0}C for 150 min duration.

The impact toughness of PMADI sample austempered at 350^{0}C for 150 min duration was found to be more in comparison with PMADI sample austempered at 300^{0}C and 400^{0}C for 150 min duration and the same can be attributed due to the increase in percentage elongation and reduced hardness.

Considering PMADI – 400, the reason for increase in the hardness is due to the fact that, the work hardening ability of austenite phase getting transformed to martensite phase and the same is clear from the matrix microstructure Figure.2 (d).

The hardness of the sample PMADI – 400 affects the fracture toughness characteristics and Hence, PMADI sample austempered at 400^{0}C for 150 min duration results in the formation of martensite which reduces the fracture toughness and the same is depicted in the Table.3.

The lower Bainite showed higher hardness levels than feathery Bainite and also retained austenite is a tough phase, the hardness of PMADI sample decreases when its percentage increases in the matrix and the same enhances the fracture toughness of PMADI sample austempered at 350^{0}C for 150 min duration.

The PMADI sample austempered at 350^{0}C for 150 min duration consisting a mixture of feathery Bainite [Figure.2 (c)] and stable carbon enriched austenite [Figure.3 (b)] and these features are supportive for the enhancement of fracture toughness.

The fracture toughness characteristics Increases as Hardness and ultimate tensile strength decreases. The same behaviour can be observed in most of the engineering materials including ADI and the same is reported in the literature. [13]

From the outcome of fracture toughness testing, it is clear that PMADI sample austempered at 350^{0}C for 150 min duration, the fracture toughness was found to be 16% superior in comparison with SMADI sample austempered under similar condition.

Previous studies [17] on ADI (after austenitizing at 871^{0}C for 2hrs and Austempering at 302^{0}C for 2hr) shows fracture toughness was varying between 67 to 68 MPa√m utilizing compact tension (CT) specimen. In the present investigation the fracture toughness of PMADI - 350 sample austempered at 350^{0}C for 150 Min duration was 78.16 MPa√m utilizing SENB specimens considering conventional austempering process.

The work may be summarized that, the fracture toughness was evaluated for both SMADI and PMADI sample considering different austempering temperatures with constant austempering time. The combination of superior strength, Impact toughness and boosted by an increase in fracture toughness helps to develop an alternate candidate material to be used in automotive, mining and power plant applications.

Conclusions

- PMADI sample austempered at 350^{0}C for 150 Min duration reveals the presence of feathery Bainite distribution with higher retained austenite content. These factors make the material to be a suitable candidate for the application requiring superior fracture toughness.
- The fracture toughness of PMADI sample austempered at 350^{0}C for 150 Min duration is found to be 16% more in comparison with sand moulded ADI austempered under similar conditions.
- The fracture toughness of PMADI samples austempered at 350^{0}C for 150 Min is almost 8% more in comparison with PMADI samples austempered at 300^{0}C for 150 Min and 12% more in comparison with PMADI samples austempered at 400^{0}C for 150 Min.
- The ultimate tensile strength and Impact toughness of PMADI sample austempered at 350^{0}C for 150 Min duration is found to be 1050 MPa and 83.2 Joules.

Acknowledgement: The authors wished to acknowledge Jyothy Institute of Technology & Sophisticated Instrument Facility (SIF), CIIRC Bengaluru and Atria Institute of Technology, Bengaluru for granting permission to present this paper.

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