Sliding wear and corrosion behaviour of alloyed austempered ductile iron subjected to novel two step austempering treatment

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Abstract: Austempered Ductile Iron(ADI) is an exciting alloy of iron which offers the design engineers the best combination high strength-to-weight ratio, low cost design flexibility, good toughness, wear resistance along with fatigue strength. The two step austempering procedure helps in simultaneously improving the tensile strength as-well as the ductility to more than that of the conventional austempering process. Extensive literature survey reveals that its mechanical and wear behaviour are dependent on heat treatment and alloy additions. Current work focuses on characterizing the two-step ADI samples (TSADI) developed by novel heat treatment process for resistance to corrosion and wear. The samples of Ductile Iron were austempered by the two-Step Austempering process at temperatures 300°C to 450°C in the steps of 50°C. Temperatures are gradually increased at the rate of 14°C/Hour. In acidic medium (H₂SO₄), the austempered samples showed better corrosive resistance compared to conventional ductile iron. It has been observed from the wear studies that TSADI sample at 350°C is showing better wear resistance compared to ductile iron. The results are discussed in terms of fractographs, process variables and microstructural features of TSADI samples.

Key words: Permanent moulded austempered ductile iron, Two step austempering, Corrosion

1. Introduction
Austempered ductile irons are being widely used in the recent past for automotive applications[1,2,3]. It is normal that the demand for these iron will increase in the near future, in the automotive sector, as well as in numerous different engineering fields. The interest for these irons is because of the way that they offer an excellent mix of high strength, toughness and ductility. This uncommon blend of properties comes about due to their extraordinary microstructure, which comprises of ferrite and austenite, instead of ferrite and carbide.

Austempered ductile iron is prepared by subjecting a ductile iron samples to an austenitization soak followed by tempering in salt bath. This aids in dissolving the carbides formed in the as-cast condition. The microstructure created in ADI is exceptionally unique. Graphite nodules are encompassed by a grid of bainite and ferrite with a high retention of austenite content. This unique microstructure will lead to better strength and resistance to wear.

Significant work has been reported for the understanding micro-structural characteristics of ADIs for the improvement of strength and wear resistance. Seetharamu et al. [4] have reported beneficial effects of utilizing permanent moulds to produce ADI. The reported results have indicated that there is an improvement in the mechanical and tribological properties of ADI produced from permanent moulds as compared to ADI produced from sand moulds. Murthy et al. [5] have reported improved mechanical
properties and wear resistance of permanent moulded ADI with manganese addition. The mechanical properties and wear resistance of ADI are dependent on the matrix microstructure which consists of bainite and retained austenite. Research is being carried by adopting various heat treatment processes to achieve improved strength and wear resistance of ADI. Putatunda et al. [6] have successfully developed a novel two-step austempering process, and microstructure and mechanical properties have been investigated. It is observed that the strength and fracture toughness of the ADIs have been improved significantly. It is also reported by Ayman et al. [7]. From the literature it is found that the novel heat treatment process results in improvement in mechanical strength compared to conventional heat treated ADIs. Mechanical and wear behaviour of ADIs mainly depend on heat treatment parameters and alloying elements. In the current work, resistance to wear and corrosion of ADI samples poured in gray cast iron permanent moulds and subjected to novel heat treatment process [6]. The samples have been subjected to initial austempering temperatures 300°C to 450°C in steps of 50°C and subjected to gradual temperature increment of 14°C/Hour.

2. EXPERIMENTAL SETUP AND PROCEDURE

By using 15kg induction furnace, the ductile iron castings were made. The raw materials used were of high grade mild steel, Ferro-Silicon, coke and Nickel-Magnesium. The charge is super-heated to 1300°C to 1350°C and was subjected to spherodizing treatment using Ni-Mg alloy. Post inoculation was carried out by adding Ferro-Silicon alloy and melt is stirred well prior to pouring. The liquid melt was then poured into the preheated permanent gray iron mould.

| Chemical element | Composition |
|------------------|-------------|
| Carbon           | 3.50%       |
| Silicon          | 2.35%       |
| Manganese        | 0.45%       |
| Sulphur          | 0.015%      |
| Phosphorus       | 0.031%      |

2.1 Austempering heat treatment

The austenitized specimens were quenched immediately into a austempering furnace containing a mixture of potassium nitrate and sodium nitrate. The samples of Ductile Iron (DI) were austempered in two-Steps.

The test specimens taken from the castings were given the following heat treatment processes. The specimen is heated up to 900°C for 90 minutes. In this quenching process, specimen temperature above
the transformation range is brought down to the upper limit of martensite formation and hold at this
temperature till the austenite is transformed completely to the intermediate structure.
Then the samples are quenched into the furnace maintained at 300°C. Then the temperature is increased
from 300°C to 314°C for one hour at the rate of 14°C/hr. Similarly, same process is repeated for second
set at 350°C, third set at 400°C, fourth set at 450°C. Later the specimen is taken out and cooled at room
temperature in air medium. Austempered specimens were air cooled and possible decarburized layers
were removed. Thermocouples are placed in the salt bath very close to the samples for monitoring the
temperatures. The temperature of the salt bath remains constant during quenching as the salt bath is very
large compared to the size of the test samples.

2.2 Corrosion Test Procedure
The corrosion test set up consists of namely: Corrosion analysis software, Electrodes, Specimen holder,
and power supply.
Electrodes:
Platinum Electrode (Counter Electrode)
Ag AgCl electrode (Reference Electrode)
Austempered iron specimens are polished and cleaned well. The specimen is then completely insulated using
insulating tape leaving square cm area. Exposed area then made to corrode in corrosive medium like
(0.5N, 0.75N and 1N) NaCl and H2SO4.
Current is made to flow through specimen using Ag AgCl and Platinum electrodes. Specimen is immersed in
corrosive medium and allowed to corrode by flowing current for 60s. Using the analysis software, first find
out the open circuit potential of the specimen for given conditions. Manually set the range of potential as
well as current sensitivity. Later obtain the Tafel plot for the given normality and temperature. Find out
density, number of electrons, area, and formula weight from pre-calculations. By entering these values, find
results namely corrosion current, linear polar resistance and corrosion rates in terms of milli-inch per year.
Similarly follow the same procedure for different normality’s of NaCl and H2SO4. Thus, try to analyze and
compare the results for different Austenizing temperatures.

2.3 Wear Test Procedure
Insert the specimen into the holder and set it perpendicular to the disk. Add suitable weights to the lever.
Start the motor and adjust the speed thus making the specimen in contact with the disc. Start the clock and
note down the maximum frictional force for each 5 min at different weights. Stop the motor and remove the
specimen. Measure the specimen dimension and weight. Repeat the test with other set of specimens.

3. Results & Discussions

3.1 Corrosion and Tafel Plots:
The experimental results are noted and tabulated as shown in the table 2. In acidic medium (H2SO4),
the conventional ductile iron is showing an increase in corrosion rate as compared to the TSADI samples as seen
Figure 3 and figure 4. This results in formation of salt layer on the surface of austempered specimens. In
acidic medium (H2SO4) due to the formation of nearly neutral pH value, the corrosion effect on TSADI
samples is very less than that of Ductile Iron.

| Normality | As cast | 300°C | 350°C | 400°C | 450°C |
|-----------|---------|-------|-------|-------|-------|
| 1N H2SO4  | 2.52E-02| 9.21E-03| 9.18E-03| 6.59E-03| 9.03E-03|
| 0.75N H2SO4| 1.75E-02| 6.34E-03| 3.37E-03| 5.89E-03| 4.60E-03|
| 0.5N H2SO4| 1.57E-02| 5.44E-03| 3.26E-03| 2.70E-03| 2.41E-03|
| 1N NaCl   | 2.894E-04| 5.032E-04| 5.237E-04| 5.269E-04| 8.132E-04|
| 0.75N NaCl| 2.620E-04| 4.216E-04| 3.340E-04| 3.322E-04| 7.521E-04|
| 0.5N NaCl | 2.441E-04| 3.142E-04| 3.095E-04| 2.891E-04| 5.214E-04|

In base medium (NaCl), due to the formation of salt layer on the surface of Austempered samples, the pH
value will be more basic in nature, thus it enhances more corrosion effect in basic medium. Hence the
corrosion effect is much larger in case of TSADI samples than the Ductile Iron.
Figure 3: Tafel graph for varying concentrations of H$_2$SO$_4$ and NaCl at 300°C
Figure 4: Tafel graph for varying concentrations of H$_2$SO$_4$ and NaCl at 350°C
3.1.1 Bar Charts for Optimum Heat Treatment Parameter:

![Bar Chart for 1N H₂SO₄ and 1N NaCl](image)

**Figure 5:** Bar chart for 1N H₂SO₄ and 1N NaCl

3.2 Wear

The test results of wear for various samples at varying loads are tabulated as shown in the table 3. It is observed that the as cast samples have shown the highest wear at all the loads. The TSADI sample at 350°C exhibited the highest wear resistance at all the loads.

| Loss of Weight | As cast | 300°C | 350°C | 400°C | 450°C |
|---------------|--------|-------|-------|-------|-------|
| 1 Kg          | 0.015  | 0.007 | 0.004 | 0.009 | 0.012 |
| 2 Kg          | 0.019  | 0.009 | 0.006 | 0.015 | 0.013 |
| 3 Kg          | 0.021  | 0.012 | 0.010 | 0.018 | 0.015 |

**Figure 6:** Bar chart for wear at 1kg load  
**Figure 7:** Bar chart for wear at 2kg load  
**Figure 8:** Bar chart for wear at 3kg load  
**Figure 9:** Bar chart for VHN
3.3 Surface Hardness
The test results of surface hardness measured by Vickers hardness test for various samples is tabulated as shown in the table 4. It is observed that the as cast samples have shown the highest wear at all the loads. The TSADI sample at 350°C exhibited the highest wear resistance at all the loads.

| Samples       | Vickers Hardness Number |
|---------------|-------------------------|
| Ductile Iron  | 216.925                 |
| ADI 300°C     | 240.325                 |
| ADI 350°C     | 308.833                 |
| ADI 400°C     | 258.65                  |
| ADI 450°C     | 249.25                  |

3.4 Microstructure Fractographic Studies:
Microstructures of TSADI samples are shown in Figure 10 and Figure 11. The ADI heat treated at 350°C showed the increased hardness, wear resistance compared to others. This is due to the presence of graphite nodules which are surrounded by a matrix of bainite and ferrite with more amount of retained austenite.

SEM photograph of fractured ADI samples as cast and TSADI at 350°C are as shown in figure 12 and figure 13 with magnification x200. The samples have indicated note of a brittle fracture with a little elongation in Fig 12. This is an indication of a mixture of cleavage and cup & cone structure. In samples of TSADI at 350°C, we can observe dimples this indicates the increased toughness of the material.
4. Conclusions:
In acidic medium (H₂SO₄), the conventional ductile iron is showing an increase in corrosion rate as compared to the TSADI samples. In base medium (NaCl), the corrosion effect is more in case of TSADI samples than that of Ductile Iron samples. It has been observed that TSADI Austempered at 350°C has highest surface hardness. It has been observed from the wear test that TSADI Austempered at 350°C is showing better wear resistance compared to conventional ADI and ductile iron samples.

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