Effect of austempering temperature and manganese content on the impact energy of austempered ductile iron

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Abstract: Austempered ductile iron (ADI) is a revolutionary material that has the ability to replace some of the commonly used steel forgings. ADI is being used in wide range of products because of the availability of this material with relatively lesser cost and its higher strength to weight ratio. Manganese is an important alloying element added to ductile iron for improving the hardenability. However, the amount of manganese has to be carefully selected to avoid any adverse effect on the mechanical properties. In this study, impact energy and tensile properties are determined for the various austempering temperatures and manganese content. Statistical analysis is carried out to determine the relative contribution of each factor on the results. Higher impact energy is observed at the higher austempering temperature of 420°C. No adverse effect on impact energy is observed for the addition manganese up to 1 wt%. Response surface methodology is used to optimize the parameters for obtaining the superior combination of ultimate tensile strength and impact energy. Regression equations are fit to predict the impact energy and ductility of manganese alloyed ADI.

Subjects: Material Science; Metals & Alloys; Materials Processing; Metals & Alloys

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PUBLIC INTEREST STATEMENT
Austempered Ductile Irons (ADI) are widely used in automobile industry and agriculture equipment. It is essential to control the properties of the ADI during production so that it has superior combination of mechanical properties. It is important to improve the hardenability of the ADI without compromising the mechanical properties. “Austempering” is a heat treatment process which improves the strength and hardness of the ductile iron without compromising in toughness. Manganese addition helps increasing the hardenability of the ADI. Hence, it is essential to select the optimum parameters for austempering heat treatment with optimum addition of manganese, so that ADI acquires required properties. This article reports the relationship between variations of mechanical properties of ADI along with manganese addition and heat treatment parameters. Thus, it helps to obtain the ADI with required properties by carrying out the heat treatment process by selecting suitable heat treatment parameters.
Keywords: ADI; impact; manganese; austempering

1. Introduction

Austempered ductile iron (ADI) is an extraordinary material which received significant attention after the research findings about its excellent combination of mechanical properties. Owing to its relatively lower manufacturing cost and excellent mechanical properties, it has become a suitable substitution for the commonly used steel forging and few alloys of aluminium (Olawale & Oluwasegun, 2016; Pereiraa et al., 2020; Sehzudin et al., 2020). The values for heat treatment parameters have to be carefully selected for austempering treatment as it affects the microstructure and mechanical properties. Also, addition of alloying element to the ductile iron affects the final properties of the material (Gazda et al., 2018; Pereiraa et al., 2020; Yang & Putatunda, 2004). Lower austempering temperature produces finer grains, whereas higher austempering temperature results in coarser grain. Hence, higher hardness is obtained for the ADI which is austempered at lower austempering temperature. However, austempering carried out at lower temperature results in relatively lower impact energy owing to lower carbon content in the austenite (Gazda et al., 2018). Along with hardness and strength, impact energy is one of the important properties required in most of the applications. ADI has wide range of applications including agriculture equipment, automobile parts and railway (Barbosa et al., 2015; Cemal Cakir & Yahya Isik, 2008; Klocke et al., 2010; Narasimha Murthy et al., 2009; Şeker & Hasirci, 2006). The requirement for the higher impact energy along with good strength is specifically required for ground engaging tools. Addition of alloying elements mainly alter the mechanical properties of ADI. Manganese addition to the ductile iron is required in order to improve hardenability and austemperability (Narasimha Murthy et al., 2009). However, addition of higher amount of manganese may affect adversely on the mechanical properties. It is therefore important to study the effect of manganese content on the impact properties of ADI.

Few reports are available regarding the impact and ductility properties of ADI. Grech and Young (Grech & Young, 1988) reported about the impact properties of copper-nickel alloyed ADI. It is reported that optimum impact strength is obtained at 350°C of austempering temperature which was austempered for two hours. Kim et al. (Yoon-Jun et al., 2008) studied the mechanical properties of ADI alloyed with copper and molybdenum. It is reported that increasing the austempering temperature has increased the impact energy considerably till 410°C. Zimba et al. (Zimba et al., 2003) reported about the usage of ADI in earth moving components. It is reported that higher strength is obtained at the austempering temperature of 340–375°C with marginal reduction in impact energy. Also, increasing the austempering temperature has resulted in higher impact properties owing to higher amount of retained austenite. Mallia and Grech (Mallia & Grech, 1997) reported about the effect of silicon on the impact toughness of ADI. It is reported that higher silicon content when austempered at higher austempering temperature has resulted in optimum impact toughness.

Even though few reports are available regarding the impact properties of ADI, not many literature is available regarding the effect of manganese on impact properties of ADI. Manganese is an important alloy added to increase the hardenability of the material. In this study, manganese is varied in different proportion to assess its effect on impact energy and ductility of ADI. Statistical analysis is carried out to fit the regression model so that impact energy may be predicted for various austempering temperature. In the earlier study of the same project, it is reported about the tensile strength of ADI at various condition (Hegde et al., 2019). In the current study, with the additional results of impact energy and ductility, manganese content and austempering temperature are optimized in order to obtain superior combination of tensile strength and impact energy.
Table 1. Details of ductile iron constituents

| Element in wt% | Carbon | Silicon | Manganese | Phosphorus | Sulphur | Chromium | Fe |
|----------------|--------|---------|-----------|------------|---------|----------|----|
| Alloy 1        | 3.37   | 2.60    | 0.268     | 0.015      | 0.013   | 0.017    | 93.6|
| Alloy 2        | 3.71   | 2.58    | 0.64      | 0.015      | 0.013   | 0.023    | 93  |
| Alloy 3        | 3.86   | 2.55    | 1.01      | 0.021      | 0.013   | 0.024    | 92.4|

2. Materials and methods

In this section, explanation is given regarding the casting of the ductile iron as per the required study. Also, details are provided regarding the heat treatment carried out on ADI and different mechanical tests that have been carried out to assess the mechanical properties of the ADI.

2.1. Material

ASTM standard A897/A897M (ASTM A897/A897M −15, 2015) is used for casting the ductile iron. As it is needed to determine the effect of variation in manganese on the impact energy, casting is produced in three categories with three different amount of manganese. All other elements in the castings are kept close. Spectroscopy is used to determine the exact composition of the cast. Table 1 gives the chemical composition of the cast.

The experiments are conducted according to the full factorial approach. The total trails according to full factorial method = $3^2 = 09$.

2.2. Design of experiments

The aim of the study is to determine the effect of austempering temperature and manganese on the impact properties ADI. Hence, other heat treatment parameters such as austenitization temperature, austenitization time, austempering time are kept constant. Austempering temperature and manganese content are varied in three different levels each as given in Table 2.

2.3. Heat treatment and characterization

The test samples are subjected to heat treatment in two different furnaces as per the information given below. Initially, the specimens are heated to austenitization temperature of 950°C in a muffle furnace. The specimens are held at that temperature for 2 h in the furnace. Specimens are then rapidly taken out and put in another salt bath comprising of sodium nitrate and sodium nitrite which is maintained at the pre-determined austempering temperature (320, 370 and 420°C). The test samples are kept at the austempering temperature in the salt bath for the fixed duration of 2 h. The austenitization temperature, austenitization time and austempering time are not varied, whose values are based on literature review (Hegde et al., 2019). Specimens are finally taken out from the salt bath and cooled in the air till it attains room temperature.

The impact energy of the heat treated material was determined by conducting the “Charpy” test. The test was conducted following the instruction given in the ASTM A327/A327M (ASTM A327/A327M, 2011). The impact test was conducted on the “V” notched bar using the “Charpy” impact test machine (FIE, Model IT-30). The machine has the maximum capacity of 300 J. The tensile test specimen was machined

Table 2. Experiment details with factor variation

| Factors                        | 1  | 2  | 3  |
|--------------------------------|----|----|----|
| Austempering temperature (°C)  | 320| 370| 420|
| Manganese content (wt %)       | 0.258| 0.64| 1.01|
Table 3. Impact energy and tensile properties results

| Mn content (wt%) | Austempering temperature (°C) | Impact energy (J) | Ductility (% elongation) | Ultimate tensile strength (MPa) |
|------------------|-------------------------------|-------------------|--------------------------|-------------------------------|
| 0.268            | 320                           | 50                | 3                        | 1105                          |
| 0.268            | 370                           | 76                | 7                        | 835                           |
| 0.268            | 420                           | 96                | 10                       | 610                           |
| 0.64             | 320                           | 54                | 3.2                      | 987                           |
| 0.64             | 370                           | 78                | 7.2                      | 765                           |
| 0.64             | 420                           | 98                | 10.3                     | 548                           |
| 1.01             | 320                           | 48                | 2.8                      | 904                           |
| 1.01             | 370                           | 72                | 6.9                      | 687                           |
| 1.01             | 420                           | 93                | 9.7                      | 470                           |

from the Y block casting. The specimen was prepared according to the shape and dimensions specified in ASTM E8 (ASTM E8/E8M, 2016). The microstructures are obtained using scanning electron microscopy (SEM) which was operated with accelerating voltage of 15 kv.

3. Results and discussions
The results obtained for the tensile properties and impact energy at various conditions are provided in Table 3.

From Table 3, it is seen that impact energy was increased with the increase in the austempering temperature. However, increase in the manganese content from 0.268 to 1.01 wt%, has not produced the significant variation in the impact energy. Similarly, high ductility was obtained for the high austempering temperature of 420°C. This may be because of the higher amount of retained austenite obtained at this austempering temperature (Olawale & Oluwasegun, 2016). The ultimate tensile strength data is taken from the previously reported results from the same project by the same author (Hegade et al., 2019) for the comparison purpose. It may be seen that tensile strength obtained at the higher austempering temperature of 420°C is lower because of the coarser structure. These results are used to obtain the combined optimization parameters based on the approach of maximizing impact energy and tensile strength.

3.1. Microstructure analysis

(b) 420°C with 0.64 wt% Mn.

Figure 1(a,b) shows the microstructures of ADI specimen which are austempered at 320 and 420°C respectively. Typical ausferrite structure is observed with graphite nodules. At the higher austempering temperature of 420°C, the structure is coarser with higher amount of retained austenite. Finer structure is obtained at the lower austempering temperature of 320°C, which is the reason for higher tensile strength at this temperature. The increased amount of retained austenite may be the reason for the higher impact energy obtained at this temperature (Pereiraa et al., 2020).

3.2. Statistical analysis
The results obtained for impact energy and ductility are subjected to statistical analysis. The effect of each factors on the impact energy and ductility are determined using Analysis of Variance test.

From the ANOVA results provided in Tables 4 and 5, it is seen that austempering temperature is the major contributing factor on the impact energy and ductility within the range of selected values for current study. Also, from the ANOVA, it is seen that manganese has no significant effect
on ductility within the range of values selected for the current study. The effect of these parameters on impact energy and ductility are shown graphically in the following figures.

Figures 2 and 3 show the effect of austempering temperature on impact energy and ductility respectively. As it seen, the increase in the austempering temperature has resulted in increase of impact energy and ductility. The same pattern is observed with varying manganese content. However, the relative contribution of manganese on the results is very less compared to the effect of austempering

| Table 4. ANOVA results for impact energy of ADI |
|-----------------|--------|----------------|--------|----------------|----------------|
| Factors         | Df     | Sum of squares | Adjusted MS | P value | Relative contribution |
| Austempering Temperature | 2     | 3046.89         | 1523.44  | <0.001 | 98               |
| Mn Content      | 2     | 48.22           | 24.1     | 0.001  | 1.5              |
| Error           | 4     | 1.78            | 0.44     |        |                  |
| Total           | 8     | 3096.8          |          |        |                  |
As the austempering temperature is increased, the carbon content in the austenite also increases because of the higher rate of carbon atom diffusion. This may be the reason for the higher impact energy obtained at the austempering temperature of 420°C.

Regression equations are obtained based on the results available for impact energy and ductility. The regression equations are fit in order to predict the impact energy and ductility involving the factors in the range of values considered for this study. Regression equations for impact energy and ductility are given in the Equations (1) and (2), respectively.

\[
\text{Impact Ennery}(J) = -90.03 + 0.45(a) - 4.03(b)
\]

\[
R^2 = 98.5\%
\]

\[
R-Sq (Adj) = 98.02\%
\]

\[
\text{Ductility} \ (% ) = -19.5 + 0.07 \ (a) - 0.269 \ (b)
\]

\[
R-Squared = 98.9\%
\]
Figure 3. Effect of austempering temperature on ductility.

Effect of austempering temperature and manganese on ductility

![Graph showing the effect of austempering temperature and manganese on ductility.]

\[ R\text{-Sq} \text{ (Adj)} = 98.54\% \]

where,

\[ a \text{— Austempering temperature (°C)} \]
\[ b \text{— Manganese content (wt%)} \]

The high R square values obtained indicate that, regression fit obtained is good. These equations may be used to predict the impact energy and ductility for any intermediate values of the control variables.

In order to obtain the superior combination of impact energy and tensile strength, combined optimization is carried out using response surface methodology. "Maximize" approach is used for both impact energy and tensile strength for the optimization. The following optimized values are obtained for austempering temperature and manganese content from RSM optimization.

Values obtained from RSM optimization

Austempering temperature = 377°C
Manganese content = 0.268 wt%

4. Conclusions
In this study, various tests are carried out to understand the effects of austempering temperature and manganese content on impact energy and ductility of ADI produced by conventional austempering process. Based on the results and analyses, following conclusions are obtained.
• Increase in the austempering temperature has resulted in the significant improvement in impact energy and ductility. Higher amount of retained austenite is observed at the higher austempering temperature of 420°C. However, ultimate tensile strength obtained at this temperature is lower.

• From the statistical analysis, it is determined that the effect of manganese on the impact energy is minimum, whereas, effect of the same on ductility is not significant within the range of values selected for the study.

• The regression equations are obtained for predicting the impact energy and ductility. The high R squared values obtained for the regression model indicates a good fit and the same may be used to predict the result for any intermediate values of austempering temperature and manganese content.

• Austempering temperature and manganese content are optimized to obtain the superior combination of impact energy and ultimate tensile strength.

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