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Mechanical characterization and optimization of heat treatment parameters of manganese alloyed austempered ductile iron

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ABSTRACT

Austempered Ductile Iron (ADI) belongs to the family of cast irons whose mechanical properties are altered using austempering heat treatment process. The objective of this paper is to study the effects of heat treatment parameters on manganese alloyed ADI. Hence, austenitization temperature, austempering temperature and austempering time are taken as the control variables along with the manganese content in the material. The effects of heat treatment are studied by measuring the ultimate tensile strength and the hardness of the material. The regression equations are developed to relate the various parameters under study. The microstructures of the specimen reveal that retained austenite content increases with increase in manganese and results in decrease in hardness of the material. The statistical analyses indicate that the austempering temperature is the major factor affecting the variation in hardness and tensile strength with 74.5 % of contribution within the range of values whereas, variation in manganese content does not have significant effect on hardness within the investigated composition range in the material.

Keywords: Optimization; Analysis of variance; Austempered ductile iron; Austenitization ; Austempering.

INTRODUCTION

Austempered ductile iron (ADI) is a heat treated ductile cast iron with a unique microstructure known as “ausferrite” which offers superior combination of various mechanical properties [1]. The microstructure of ADI consists of acicular ferrite and carbon rich austenite (known as “ausferrite”) with graphite nodules. ADI offers excellent strength, toughness and fatigue characteristics. It has high strength to weight ratio and excellent wear resistance. High austempering temperature (400˚C) produces ADI with high ductility, yield strength in the range of 500 MPa with good fatigue and impact strength. A lower austempering temperature results in ADI with very high yield strength, high hardness and excellent wear resistance. The properties can be controlled and altered suitably by varying the heat treatment parameters [2-4]. The farm machinery industry has taken a keen interest in ADI for its excellent wear characteristics. The heavy truck industry has recognised the potential benefits of austempering solutions many years ago. Manufacturers took advantages of the versatility of ADI to introduce innovative light weight; high performance parts. Typical examples include diesel engine timing gear, suspension brackets, gear housings, wheel hubs, and sprockets [5-6].

Many authors have reported the effect of heat treatment parameters on the mechanical properties of ADI [7-10]. Various methods to produce the ADI have also been reported by few researchers. Olawale et al. [11] reported about the forced air cooling quenching to produce austempered ductile iron. It was reported that ADI of section thickness up to 25mm can be produced with the use of forced air cooling quenching method. Arft et al. [12] reported about the machinability aspects of austempered ductile iron. The study revealed about the possible adoption of machining strategies to counteract the obstacles faced during the machining of ADI due to its high hardness. Basso and Sikora [13] reported about the production of dual phase austempered ductile iron. The study described the methodology used to produce dual phase ADI which is capable of replacing steel castings and forgings in many engineering applications. Zanardi et al. [14] reported about the grading system of ADI. The study includes the mechanical properties of ADI and how these properties vary with the different grades of ADI. Mendez et al. [15] compared the properties of conventionally heat treated austempered ductile iron with nodular iron having ferritic –pearlitic grades. Murthy et al. [16] reported the abrasion behaviour of manganese alloyed permanent moulded ADI. Bayati and Elliott [17] reported about the austempering process in the ADI containing 0.67 wt % manganese. It was reported that addition of high amount of manganese delays the stage I austempering reaction and also delays stage II transformation. Not much information is available regarding how the variation of manganese within the range of 0.3 wt % to 1 wt % affects the mechanical properties of the ADI. Machinability of any material is an important aspect to consider while selecting the material [18]. It is very essential to optimize the process parameters in order to obtain optimum mechanical properties and machinability.

The present study focuses on optimizing the heat treatment parameters in manganese alloyed ADI in order to obtain the optimum hardness. The present investigation selected four independent variables which are called as factors: austenitization temperature, austempering temperature, austempering time and manganese content. The hardness and tensile strength are the mechanical properties considered to be important to study the effects of changes in the factors. Manganese is varied in the lower range so as to ensure that it is fully dissolved in the matrix. A regression model is developed using Response Surface Methodology (RSM) to relate mechanical properties with the heat treatment parameters and manganese content.

MATERIALS AND METHODS

Design of Experiment

In the current study, austenitization temperature, austempering temperature, austempering time and manganese content are selected as independent variables or control factors. Austenitization time is kept constant at 2h in order to focus on the effect of manganese on mechanical properties. Generally, addition of high amount of manganese forms the carbide at the intercellular region. Manganese tends to segregate at the grain boundary which produces precipitates. This adversely affects on the mechanical properties of ADI. Hence, the addition of manganese is restricted to 1 wt %. The dependent variables are hardness and tensile strength. Mixed level design is employed and full factorial design is employed to analyze the various control factors and hence a total of 36 trails are conducted. The details about the design of experiments are given in the Table 1.

Table 1. Details of control factors with various levels

Control factors	Level 1	Level 2	Level 3
Austenitization temperature	850˚C	950˚C	---
Austempering temperature	320˚C	370˚C	420˚C
Austempering time	1 h	2 h	----
Manganese content	0.268 wt %	0.64 wt %	1.01 wt %

Material

Ductile iron was cast as per the ASTM standard A897/A897M - 15 [19]. The raw materials were melted in the medium frequency induction furnace. The required alloying elements were added to the melt followed by nodulization and inoculation. The melt was poured to the Y block mold at temperature range of 1490˚C to 1520˚C. The manganese content was varied in three levels as low (0.268 wt%), medium (0.64 wt%) and high (1.01 %). The wt% of manganese is restricted because the maximum solubility of manganese in ferrite phase is 3 wt %, so that the precipitation of hard manganese carbide phase is avoided during heat treatment. The composition of all the castings and the different melts are maintained close to the composition specified in Table 2 as per the experiment design requirements. Care is taken to ensure that only manganese content is varied while keeping all other elements at the same amount so that effect of manganese on the mechanical properties can be analyzed. Tensile test specimens were machined out from the Y block casting according to ASTM standard E8/E8M [20]. For micro structure analyses and hardness test, the specimens were machined out from the round bars.

Table 2. Chemical composition of ductile iron casting

Type of casting\ Element in wt%	C	Si	Mn	P	S	Cr	Mg	Fe
Low Mn	3.70	2.60	0.268	0.015	0.013	0.017	0.0360	93.35
Medium Mn	3.71	2.60	0.64	0.015	0.013	0.017	0.0380	92.96
High Mn	3.71	2.59	1.01	0.015	0.013	0.016	0.0370	92.6

Heat treatment and Characterization

The samples are subjected to austempering heat treatment. The samples were heated in a muffle furnace to the predetermined austenitization temperature and then held at that temperature for a predetermined time of 2h and this holding time period is called austenitization time. The specimens were then transferred quickly to a salt bath comprising of $NaNO_2$ and $NaNO_3$ maintained at the required austempering temperature. The specimens were held at this austempering temperature for a time period called austempering time followed by the air cooling of the samples to room temperature. Tensile tests of these heat treated samples were carried out using a computer controlled tensometer. Hardness of all the specimens was determined using Brinell hardness method. Metallurgical microscope is used to study the microstructure of the samples which are prepared by polishing and etching the specimen using nital.

RESULTS AND DISCUSSIONS

Tensile test and Hardness test have been conducted as per the ASTM standards E8/E8M on all the test specimens prepared as per the design of experiments. The results of the same are provided in Table 3 and Table 4. The average of three test results is considered as the result for a given experiment condition and it has been found that the variation of results for a given experiment design is less than 1% in all cases. The close values of the results can be attributed to the sample preparation methods and control of process parameters.

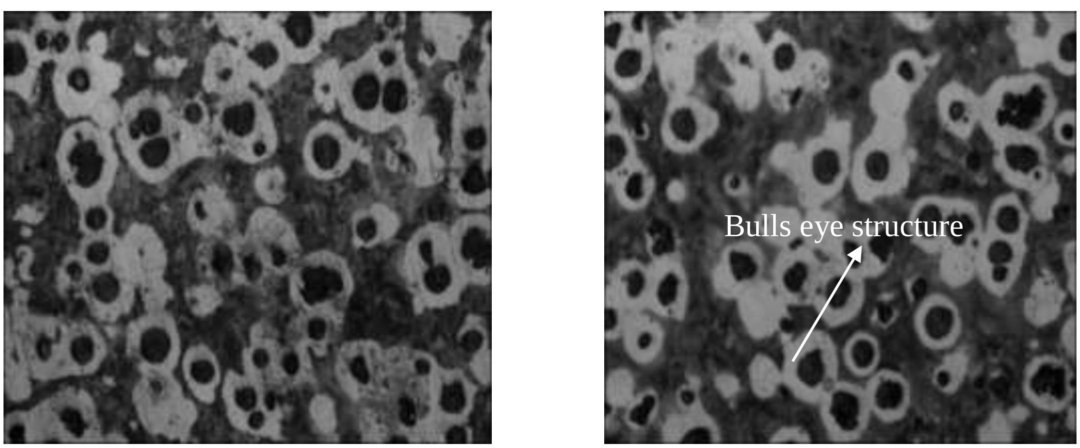
Table 3. Hardness and tensile strength of heat treated samples at austenitization temperature 950˚C

Experiment No	Austempering Temperature **˚C**	Austempering Time **Hour**	Mn Content **wt %**	Hardness **BHN**	Ultimate Tensile Strength **N/mm²**
1	320	1	0.268	331	1131
2	320	2	0.268	322	1105
3	370	1	0.268	277	890
4	370	2	0.268	271	835
5	420	1	0.268	240	650
6	420	2	0.268	233	610
7	320	1	0.64	341	1050
8	320	2	0.64	331	987
9	370	1	0.64	276	815
10	370	2	0.64	278	765
11	420	1	0.64	240	580
12	420	2	0.64	233	548
13	320	1	1.01	330	950
14	320	2	1.01	322	904
15	370	1	1.01	264	715
16	370	2	1.01	265	687
17	420	1	1.01	233	490
18	420	2	1.01	229	470

Table 4. Hardness and tensile strength of heat treated samples at austenitization temperature 850˚C

Experiment No	Austempering Temperature ˚C	Austempering Time Hour	Mn Content wt %	Hardness BHN	Ultimate Tensile Strength N/mm^2
19	320	1	0.268	392	1410
20	320	2	0.268	382	1377
21	370	1	0.268	326	1179
22	370	2	0.268	322	1120
23	420	1	0.268	271	910
24	420	2	0.268	265	875
25	320	1	0.64	388	1310
26	320	2	0.64	386	1272
27	370	1	0.64	322	1086
28	370	2	0.64	331	1050
29	420	1	0.64	277	855
30	420	2	0.64	270	810
31	320	1	1.01	375	1210
32	320	2	1.01	373	1175
33	370	1	1.01	322	975
34	370	2	1.01	308	930
35	420	1	1.01	262	745
36	420	2	1.01	264	724

Microstructure analysis



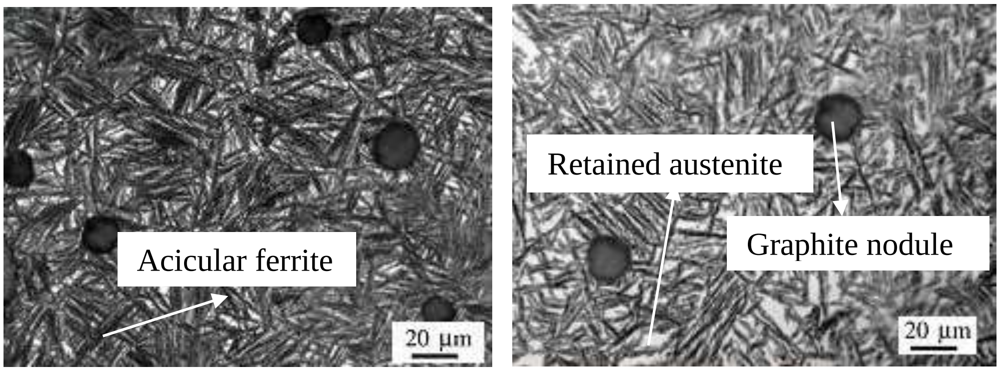
(a) (b)

Figure 1. Microstructures of as cast ductile iron with (a) 0.64 wt % Mn, (b) 1.01 wt % Mn

Figure 1 shows the microstructures of as cast ductile iron samples with different proportion of manganese content. It is observed that graphite nodules are surrounded by ferrite in the bull's eye structure. The nodule count of the samples are determined as 354 number/mm^2 for 0.64 wt % Mn sample, and 380 number/ mm^2 for 1.01 wt % Mn sample respectively.

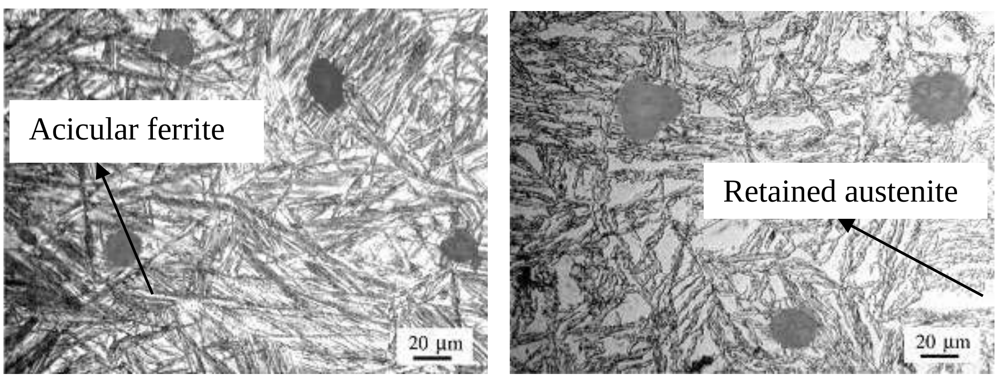
The microstructures of ADI specimen which are austempered for 1h duration are taken for analysis since there was no difference in the mechanical properties due to change in austempering time. It is observed that most of the microstructures consisted a mixture of dark needle shaped ferrite along with retained austenite. Graphite nodules were found to be dispersed in the matrix. At lower austempering temperature of 320˚C, the very fine structure is found and the ferrite is more acicular. As the austempering temperature is increased to 420˚C, the structure became coarser with feathery ferrite. This is the upper bainitic structure with relatively coarse structure. Thus, increasing the austempering temperature resulted in coarsening of the ferrite, as well as, an increase in the retained austenite content which would lead to reduced hardness. This is in accordance with the microstructure study which has been published many authors [21-24].

The figure 2 and 3 show the microstructures of samples which had been austenitized at 850˚C and 950˚C respectively with different austempering temperatures and manganese content. The amount of retained austenite was measured using the metallurgical image analysis software as per the ASTM standard E1245-03 [25]. The results are shown in the table 5. From the results of the image analysis, it is observed that, as the austempering temperature and austenitization temperatures are increased, the amount of retained austenite is increased significantly. Also, there was a slight increase in the amount of retained austenite as the manganese content is varied to 1.01 wt% from 0.268 wt%. This explains the very little variation in the hardness value as the manganese content is varied and hence the predominant factor affecting the hardness is austempering temperature. The optimum hardness and strength obtained even with the high amount of manganese addition may be attributed to the high nodule count. It was also evident that most of the carbides got dissolved in the matrix.
At the austempering temperature of 320˚C and austenitization temperature of 850^{0}C, a very fine structure is observed in the microstructure and the ferrite was found to be more acicular as shown in Figures 2 a and 2 b. When the austempering temperature is 420˚C and austenitization temperature is 950˚C, an upper bainitic structure is noticed which is a coarse structure with feathery ferrite as shown in figures 3 a and 3 b. At the high austempering temperature of 420˚C, lower hardness was resulted. Also, at the austenitization temperature of 950˚C, hardness value is slightly lower compared to the hardness at the austenitization temperature of 850˚C due to the coarsening of the grains. This explains the moderate contribution of austenitisation temperature on the hardness. This is in accordance with the various reports which have been published [26-29].



(a) (b)

Figure 2. Microstructures of ADI austenitized at 850˚C for 2 h and austempered at 320˚C for 1h with (a) 0.268 wt % Mn, (b) 1.01 wt % Mn



(a) (b)

Figure 3. Microstructures of ADI austenitized at 950˚C for 2h and austempered at 420˚C for 1h with (a) 0.268 wt % Mn, (b) 1.01 wt % Mn

Table 5. Amount of retained austenite by metallurgical image analysis

Austenitization Temperature ˚C	Austempering Temperature ˚C	Manganese Content wt %	Content of Retained Austenite Vol %
850	320	0.268	24
850	320	1.010	28
950	420	0.268	35
950	420	1.010	38

Analysis of variance (ANOVA) for hardness and tensile strength

ANOVA was carried out, initially with all the four terms together with their interactions and it was found that the linear terms had more than 99% contribution for tensile strength and hardness. Hence, the final ANOVA analysis is carried out at 5% significance level using only the linear terms to obtain the relative contribution of the four factors on the hardness and tensile strength. The results of ANOVA of the hardness are shown in table 6. From the results, it can be inferred that austempering temperature has the most significant effect on the hardness followed by austenitization temperature. These two factors contribute to 97.7% of the changes in the hardness in the rage of values under study. Austempering time and Manganese content do not contribute much to the variations in hardness of the material in the range of study.

Table 6. ANOVA results for hardness

Factors	Degrees of freedom	Seq sum of square	Adj MS	P	% Contribution
Austenitization Temperature	1	18678	18678	0.000	21.5
Austempering Temperarure	2	66113	33057	0.000	76.2
Austempering Time	1	187	187	0.031	0.2
Mn Content	2	688	344	0.001	0.8
Error	29	1059	37		
Total	35	86725			

The results of ANOVA for tensile strength are provided in table 7. Austempering temperature and austenitization temperature contribute for 90.5% of the variations in the tensile strength of the material. The effect of Manganese is more pronounced in case of tensile strength with a contribution of 8.6% of the variations in tensile strength while austempering time has very less contribution.

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Table 7. ANOVA results for tensile strength

Factors	Degrees of freedom	Seq sum of square	Adj MS	P	% Contribution
Austenitization Temperature	1	648293	648293	0.000	30
Austempering Temperarure	2	1313249	656624	0.000	60.5
Austempering Time	1	13885	13885	0.000	0.6
Mn Content	2	187233	93617	0.000	8.6
Error	29	4579	158		
Total	35	2167239			

Regression models and optimization for hardness and tensile strength

Response surface regression was used to fit an equation in order to optimize the hardness and tensile strength using the following factors: Austenitization temperature (a), austempering temperature (b), austempering time (c), and manganese content (d).

The regression equations for hardness and Tensile Strength are given in Equation (1) and Equation (2) respectively.

$$Hardness\ (BHN) = 1111.64 - 0.455556(a) - 1.04667(b) - 4.55556(c) - 9.5294(d) \quad (1)$$

$$Tensile\ strength = 5279.48 - 2.68389\ (a) - 4.67833\ (b) - 39.2778\ (c) - 237.739\ (d) \quad (2)$$

The R-Sq values for hardness and tensile strength are 97.89% and 99.76% respectively. The R-Sq (Adj) values for hardness and tensile strength are 97.62% and 99.73% respectively. The high R-Sq and R-Sq (Adj) values indicate that the regression equations possess a good fit to the actual experiment conducted. The equations could be used for optimization of process variables and for prediction of hardness and tensile strength at any of the intermediate values of the control variables. In Equations (1) and (2), the coefficients of all the terms are negative and hence the hardness and tensile strength will be minimum at the upper limits of the factors under consideration in the range of the study undertaken.

Optimization of the parameters using the Response Surface Optimizer based on “lower is the better” approach was carried out separately for hardness and tensile strength, as lesser hardness gives better machinability. The optimized parameters are as follows: Austenitization temperature = 950˚C, Austempering temperature = 420˚C, Austempering time= 2 h and Manganese content = 1.01 wt%. The optimal parameters are upper limits of the parameters considered in the study. The values of hardness and tensile strength at the optimal process parameters from the regression equations (1) and (2) are 220.6 BHN and 446 N/mm^2 respectively. The values of hardness and tensile strength at the optimal conditions from the experiments are 229 BHN and 470 N/mm^2. The percentage variation in hardness and tensile strength between the experimental values and the values from regression equations are 3.8% and 5.3% respectively which is very less. A Pearson's coefficient of correlation between the hardness and tensile strength is 0.955 and this indicates that the change in hardness is a strong indication of change in tensile strength in the same direction.

CONCLUSIONS

In this study, microstructure analysis is carried out to understand the effects of heat treatment on manganese alloyed ADI. Response surface methodology was used to optimize the process parameters and to fit a regression equation. ANOVA was carried out to determine the relative contribution of each factor on the responses. Based on the statistical and microstructure analysis, the following conclusions are made.

- Microstructure analysis has revealed the presence of typical "ausferrite" structure. At the high austempering temperature of 420^{0}C, a coarser and feathery structure was obtained with higher retained austenite content which resulted in reduced hardness and tensile strength.
- As the austenitization temperature has been increased from 850^{0}C to 950˚C, the amount of retained austenite has been increased, resulting in the marginal decrease of hardness and tensile strength.
- According to ANOVA results, the predominant factor affecting the hardness and tensile strength was austempering temperature with a contribution of 74.5 % followed by austenitization temperature with 23 % contribution.
- Based on the response optimization, using the "lower is the better" approach, the optimum level of process parameters for hardness was obtained as: austenitization temperature=950˚C; austempering temperature= 420˚C; austempering time= 2 h; manganese content= 1.01 wt %.
- Hardness values predicted using general linear regression analyses were in close agreement with experimental values with a high R squared value of 98 %.

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