

An Analysis of the Amount of Retained Austenite in Manganese Alloyed Austempered Ductile Iron

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Austempered ductile iron (ADI) is a revolutionary material which has excellent mechanical properties with ease of manufacturing. Manganese addition to the ductile iron is a complex job as it may segregate at the grain boundary causing brittleness to the material. Hence, it is important to select the optimum level of manganese which may be added to the ADI without causing any adverse effect on the mechanical properties. In this study, the effect of manganese addition and heat treatment parameters on the amount of retained austenite is studied. Manganese is added in three different proportion. Microstructure and XRD analysis have shown that increasing the austempering temperature has resulted in the increase in volume fraction of retained austenite. Manganese addition up to 1 wt% did not cause any adverse effect on the amount of retained austenite. Higher volume fraction of retained austenite obtained at 420°C is found to be 36%.

Keywords: ADI, Retained austenite, Manganese, Austempering.

1. Introduction

Austempered ductile iron possesses remarkable mechanical properties owing to its microstructure which contains acicular ferrite and carbon rich austenite. During the austempering reaction, carbon gets deposited to the matrix of austenite till this phase becomes stable at room temperature. This phase, which is enriched with carbon is known as retained austenite. The amount of retained austenite phase affects the mechanical properties of the austempered ductile iron¹⁻³. Austempering time is an essential parameter which should be selected carefully in order to obtain the required microstructure of ausferrite in ADI. If the austempering time is too long, then the retained austenite phase may further transform to ferrite and carbide, which is not desirable because of the brittleness it induces in the material. Hence, it is important to obtain the required amount of retained austenite in the structure so that the material possesses the required combination of mechanical properties⁴⁻⁶. Sometimes, addition of alloying elements to the ductile iron is essential in order to increase the hardenability and other related properties. Manganese is one such element, which is added to improve the hardenability of the austempered ductile iron⁷. However, excess amount of manganese addition may result in its segregation at the grain boundaries, which may affect adversely on the mechanical properties of the material. Hence, it is essential to determine how the amount of retained austenite varies with the addition of manganese with different proportions.

Bosnjak et al.⁸ reported about the effect of nickel, molybdenum and copper addition on the volume fraction of retained austenite. It is reported that volume fraction of retained austenite increases with increase in the austempering

temperature for the alloyed ductile iron. Austempering time of 120 min has produced the optimum combination of mechanical properties. Aranzabal et al.⁹ reported about the improvement in ductile property with increase in the volume fraction of retained austenite. It is stated that, at the austempering temperature of 370°C and more, the volume fraction of retained austenite was found to be more than 25 per cent. Sellamuthu et al.¹⁰ investigated the effect of retained austenite on mechanical properties of austempered ductile iron. It is reported that, wear resistance deteriorates with increase in the amount of retained austenite, whereas impact energy improves with increase in the amount of retained austenite. Takahashi et al.¹¹ had reported the effect of silicon variation on the amount of retained austenite phase. It is reported that, decrease in the silicon content from 2.4 wt% to 1.4 wt%, had resulted in the considerable decrease in the amount of retained austenite. They have recommended to add more than 2 wt% silicon to get the minimum required amount of retained austenite. Takita and Ueda¹² have reported about the direct correlation with the amount of retained austenite and impact energy of the ADI.

In spite of the fact that many researchers have worked regarding the assessment of retained austenite, very few reports are available regarding the same study in manganese alloyed austempered ductile iron. Knowing the limit of manganese, which could be added without compromising the required mechanical properties could be beneficial, as it helps improving the hardenability. The authors have previously studied and published about the toughness and ductility of the manganese alloyed ADI¹³. Hence, in this study, an effort is made to assess the effect of manganese addition on the volume fraction of retained austenite. Mechanical properties

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such as tensile strength and impact energy are correlated to the varying volume fraction of retained austenite.

2. Materials and Methods

This section gives the details regarding the casting of material, heat treatment carried out and characterization methods. X-ray diffraction method is used to determine the volume fraction of retained austenite in the ADI

2.1. Material

Ductile iron is cast as per ASTM standard A897/A897M¹⁴. As it is needed to determine the effect of variation in manganese on retained austenite, 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 which is as follows:

Carbon- 3.3 wt%, Silicon- 2.6 wt%, Phosphorous- 0.015 wt%, Sulphur- 0.013 wt%, Cr- 0.017 wt%, Iron- balance.

Three different alloys are cast in which except the manganese content, all other elements are kept almost same as written above. The varying manganese content in three alloys are as follows

- Alloy 1, Mn- 0.26 wt%,
- Alloy 2, Mn- 0.64 wt%,
- Alloy 3, Mn- 1 wt%

2.2. Heat treatment and characterization

Austempering heat treatment is carried out on the test samples. Initially, the samples are heated in the muffle furnace to the pre-determined austenitization temperature. Samples are held in this furnace for the fixed amount of time followed by the rapid transfer of samples to the salt bath furnace maintained at the austempering temperature. After holding the samples at this temperature for the duration of austempering time, samples are taken out and cooled in air till it reaches room temperature. The values selected for the heat treatment parameters are based on the literature survey. The mechanical properties such as ductility and toughness are assessed and reported in the previously published report of the authors¹³

2.3. X-Ray diffraction analysis

The X-ray diffraction method is used to quantify the retained austenite, obtained in the heat treated samples. The samples are prepared by polishing to ensure smooth and defect free surface, which were then subjected to XRD analysis using chromium K α radiation. Scanning was done in the 2 θ range of 35-50° to get the austenite and ferrite peaks. The data of angle of reflection and the corresponding intensity of the X-ray are obtained from the XRD experiment. This data is plotted as a graph using the “Origin pro” software to determine the integrated intensity of the peaks. The austenite (111) and ferrite (110) peaks obtained within this range are used to calculate the amount of retained austenite in the structure. The selected range for the scanning angle is based on the literature review¹⁵. Based on the studies carried out on the ADI in the past, it is known that austenite and ferrite peaks are obtained within 40 to 50° of scan angle. The single

peak of austenite and ferrite is sufficient enough to calculate the amount of retained austenite in the structure. Hence, the scan angle was selected to be in the range of 35-50° in order to obtain the austenite and ferrite peak. This calculation was carried out as per the procedure specified in ASTM E975¹⁶. The amount of retained austenite is calculated using the Equation 1¹⁶.

$$V_{\gamma} = \frac{\frac{I_{\gamma}}{R_{\gamma}}}{\frac{I_{\alpha}}{R_{\alpha}} + \frac{I_{\gamma}}{R_{\gamma}}} \quad (1)$$

where,

V_{γ} - Volume fraction of retained austenite

I_{γ} - Integrated intensity of austenite peak (111)

I_{α} - Integrated intensity of ferrite peak (110)

R_{γ} - Theoretical intensity related to austenite phase

R_{α} - Theoretical intensity related to ferrite phase

Integrated intensities of the respective peaks are obtained from analyzing the graph in “Origin pro” software. Theoretical intensity values are directly obtained for the chromium K α radiation source from the ASTM E 975¹⁶.

3. Results and Discussions

3.1. Microstructure analysis

The scanning electron microscope (SEM) was used to obtain the microstructures for all the samples which are heat treated at various austempering temperatures and austenitization temperatures with different amount of manganese content.

Figures 1 to 3 show the microstructures of ADI samples which are heat treated at an austenitization temperature of 950°C and austempered at three different temperatures of 320, 370 and 420°C respectively. At lower austempering temperature of 320°C, the structure is finer, 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 ausferritic structure with relatively coarser austenite. Amount of retained austenite has increased with the increase in the austempering temperature from 320 to 420°C. This resulted in the lower hardness at the higher austempering temperature of 420°C¹⁷.

Figures 4 and 5 show the microstructures of samples which are austenitized and austempered at different conditions. It may be noticed that structure appears coarser with increase in austenitization as well as austempering temperature. However, there is no much difference in the structure with the increase in manganese content from 0.2 to 1 wt%. Amount of retained austenite is observed to be increasing with increase in heat treatment temperatures, which is confirmed with the XRD analysis.

Retained austenite without carbide provides favorable mechanical properties to ADI. The higher amount of stabilized retained austenite has positive effect on the impact properties

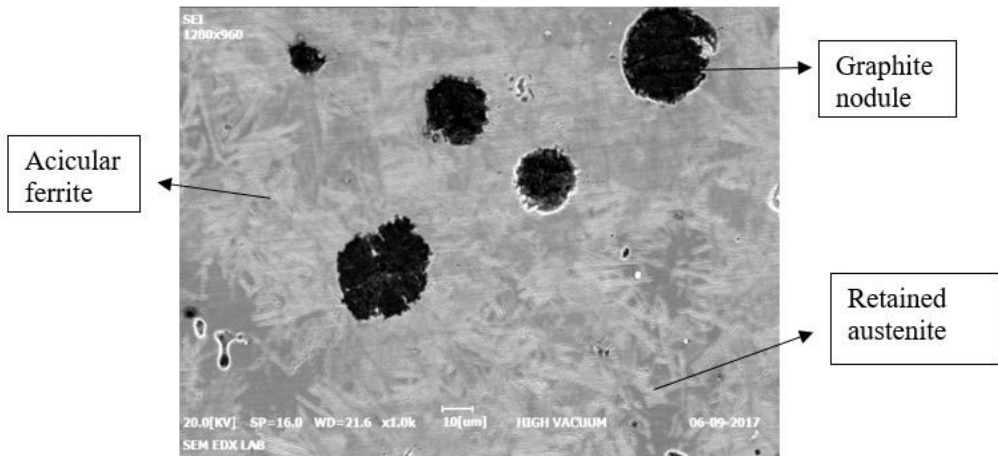


Figure 1. SEM image of ADI austenitized at 950°C and austempered at 320°C with 0.64 wt% Mn.

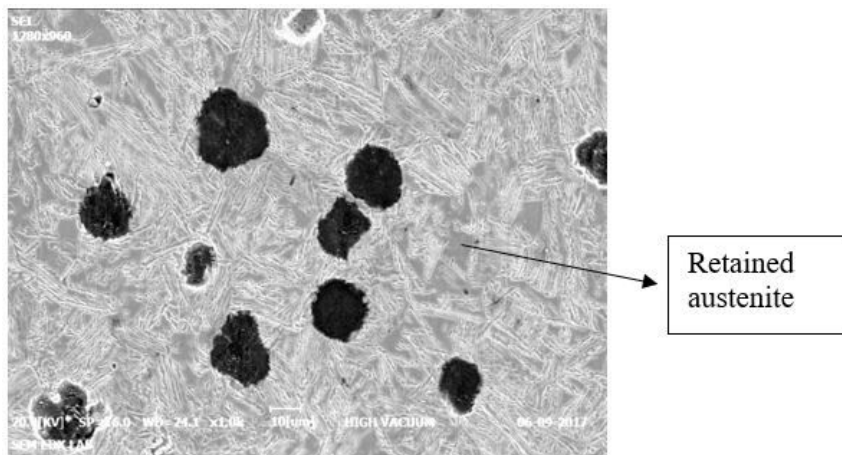


Figure 2. SEM image of ADI austenitized at 950°C and austempered at 370°C with 0.64 wt% Mn.

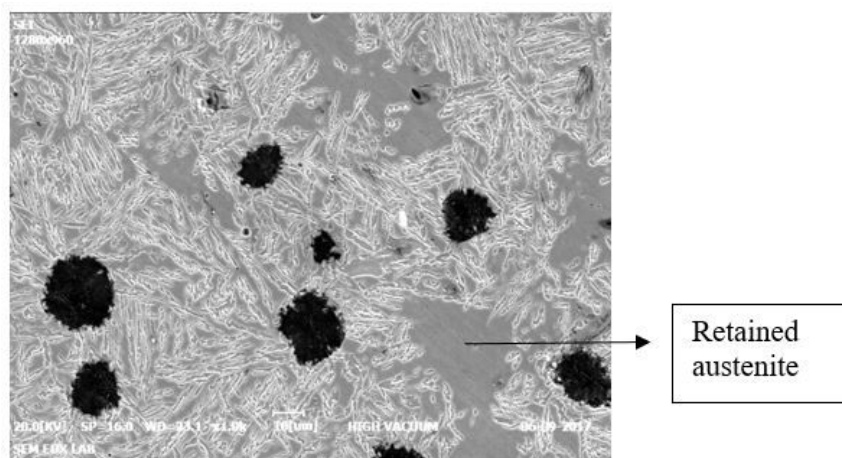


Figure 3. SEM image of ADI austenitized at 950°C and austempered at 420°C with 0.64 wt% Mn.

of ADI¹¹. However, if the austempering time is too long, the retained austenite may not be stable at that condition. It may further get transformed to bainitic ferrite and carbide, which may affect adversely on the mechanical properties

of ADI. Hence, the amount of retained austenite plays an important role in deciding the mechanical properties of ADI. This mainly depends on the chemical composition and austempering parameters. Hence, obtaining the higher

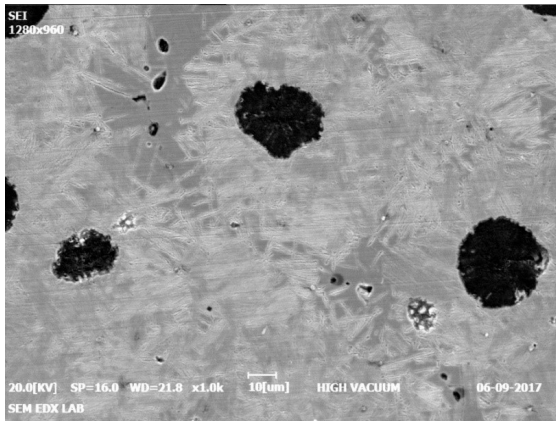


Figure 4. SEM image for ADI sample austenitized at 850°C and austempered at 320°C with 1.01 wt% Mn.

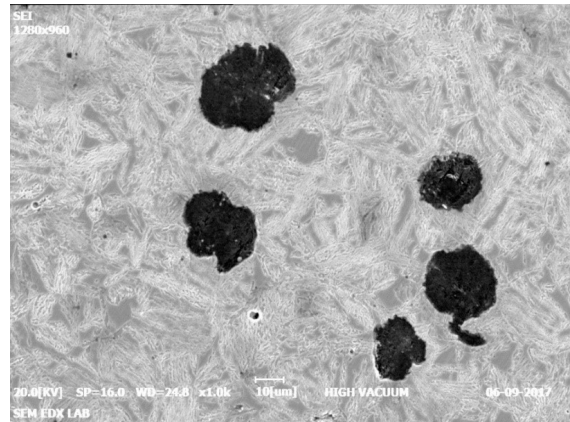
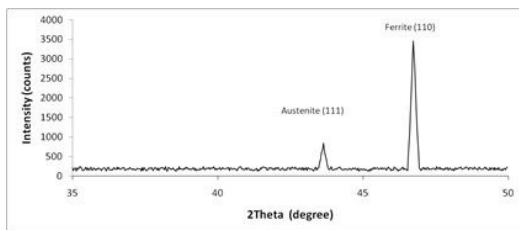
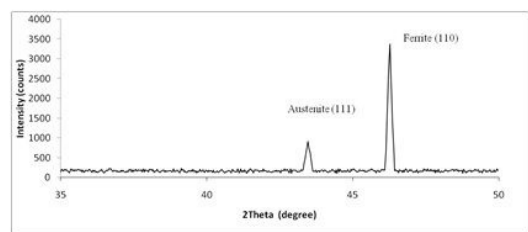


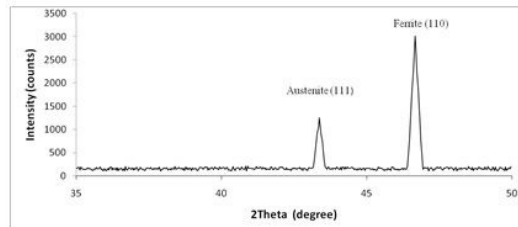
Figure 5. SEM image for samples austenitized at 950°C and austempered at 420°C with 0.26 wt% Mn.



(a)



(b)



(c)

Figure 6. XRD graphs for ADI samples with 0.64 wt% Mn, austenitized at 950°C and austempered at (a) 320, (b) 370, and (c) 420°C.

amount of retained austenite without compromising in the hardness value, helps achieving the superior combination of mechanical properties of ADI.

3.2. XRD analysis

X-ray diffraction analysis was carried out on the heat treated samples. The scan was carried out for the 2 theta range of 35 to 50 degree in order to obtain the austenite (111) and ferrite (110) peaks. Integrated intensities of the respective peaks are obtained from the “Origin pro” software. The volume fraction of retained austenite was calculated from the equations as explained in the methodology. Figure 6 shows the XRD graphs for the austempered ductile iron produced by conventional heat treatment using different austempering temperatures.

Figure 6 provides the details of XRD analysis for the conventionally austempered samples at different austempering temperatures. The volume fraction of retained austenite was calculated and the results are provided in the Table 1.

Table 1. Variation of amount of retained austenite with austempering temperature.

Austenite temperature (°C)	Volume fraction of retained austenite (%)
320	25
370	31.5
420	33

The XRD analysis shows that, increase in the austempering temperature, with austenitization temperature and manganese content at the constant level, has resulted in the increase in amount of retained austenite. The austempering temperature of 420°C has produced the ADI with 33% volume fraction of retained austenite. Also, at the higher austempering temperature, the structure obtained was coarser in nature which was evident in the microstructure analysis.

At the lower austempering temperature of 320°C, finer grains were observed. However, the amount of retained

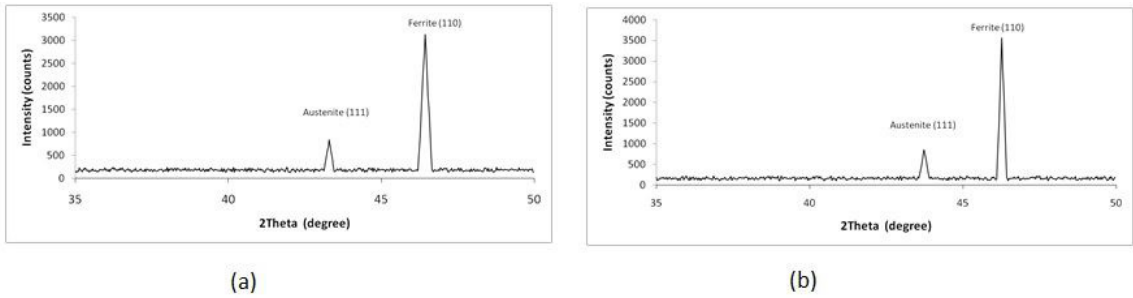


Figure 7. XRD graphs for ADI samples austenitized at 850°C and austempered at 320°C with (a) 0.268 wt% Mn, (b) 1.01 wt% Mn.

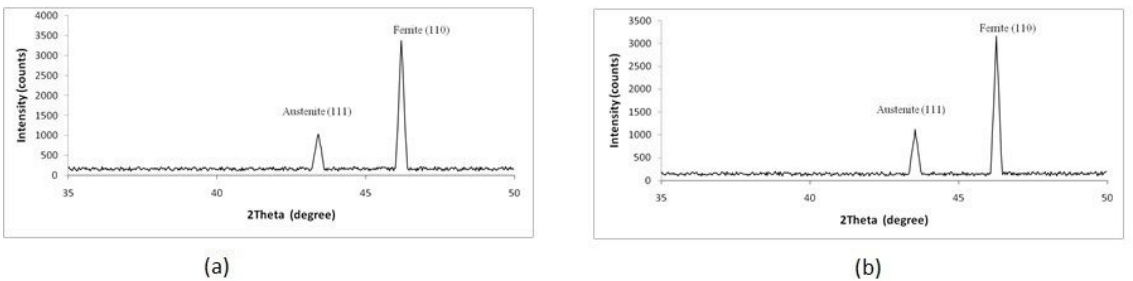


Figure 8. XRD graphs for samples austenitized at 950°C and austempered at 420°C with (a) 0.268 wt% Mn, (b) 1.01 wt% Mn.

Table 2. Amount of retained austenite at different conditions.

Austenitization temperature (°C)	Austempering temperature (°C)	Manganese content (wt%)	Volume fraction of retained austenite (%)
850	320	0.268	24
850	320	1.010	28
950	420	0.268	34
950	420	1.010	36

austenite is found to be 25% at this temperature. This considerable amount of retained austenite confirms that, austempering time is sufficient enough to initiate the austenite transformation. This is helpful in obtaining the optimum combination of strength and impact properties even at the lower austempering temperature of 320°C.

Figures 7 and 8 provide the XRD graphs for the samples with different manganese content and heat treated at different temperatures.

Figures 7 and 8 show the XRD analysis carried out for the samples at various heat treatment parameters with different manganese content and amount of retained austenite was calculated for all the cases. The results obtained for the amount of retained austenite are provided in the Table 2.

From the Table 2, it is seen that amount of retained austenite increases with the increase in austenitization temperature. Increase in the manganese content has resulted in the marginal increase in the amount of retained austenite. This justifies the variation in mechanical properties with the change in heat treatment parameters. Hardness and tensile strength values have been found to be decreasing with increasing austempering temperature¹⁷⁻¹⁹. These reduction in hardness and strength may be attributed to the coarser

grains obtained at higher austempering temperature. From the microstructure study and XRD analysis, it may be seen that, increase in the amount of retained austenite has resulted in the improved impact energy and ductility which is previously reported by the authors¹³. Also, it is seen that by selecting the appropriate austempering parameters, amount of retained austenite obtained at the lower austempering temperature of 320°C is nearly 24 vol %. This helps in obtaining the relatively higher impact properties. Further, this analysis is proven to be helpful to achieve the superior combination of strength and toughness by having high amount of retained austenite with finer grains^{13,17}.

4. Conclusions

- Microstructure study has confirmed the “ausferrite” structure for the manganese alloyed ADI produced by conventional austempering. Higher austempering temperature of 420°C has produced coarser and feathery structure, whereas lower austempering temperature of 320°C has produced finer and acicular structure.
- The amount of retained austenite was increased from 25 to 33% (volume fraction), with the increase in

the austempering temperature from 320 to 420°C. The increase in manganese content from 0.26 to 1.01 wt% has resulted in the marginal increase of retained austenite.

- As the austenitization temperature was increased from 850 to 950°C, slight increase in the amount of retained austenite is observed.
- Higher quantity of retained austenite (36 vol %) is obtained at an austempering temperature of 420 °C.
- The authors confirm that the data supporting the findings of this study are available within the article.

5. References

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