Effect of nickel content and austempering temperature on microstructure and mechanical properties of austempered ductile iron (ADI)

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Abstract. In this article, the effect of two variables namely nickel content and austempering heat treatment temperature on mechanical and microstructural properties of ADI were investigated. Four Nickel content levels of 0.0, 0.6, 0.8 and 1% and three austempering temperatures of 320, 340 and 360°C were selected. Prior to austempering, all the samples were austenitized for 30 min at 840°C. The microstructure of austempered alloys mainly consisted of a mixture of needle like acicular ferrite and carbon stabilized austenitic matrix. The increase in Ni content improved the strength and hardness and it reached its peak at 0.8%. The microstructures also became finer with increase in Ni content as confirmed by Impact energy and elongation. Nodularity was found to be above 90% for all alloys at all austempering temperatures. However, the nodule counts increased with increase in Ni content up to 0.8% and then dropped. XRD analysis was done to estimate the retained austenite content present in the ausferrite matrix and it was concluded that for any given Nickel content level, the retained austenite content increases with austempering temperature. Nickel of 0.8% was found to be optimum for all austempering temperatures.

1. Introduction
Austempered ductile iron (ADI) is emerging materials which is presently being used extensively in automobile, agricultural and mining applications due to its high strength, toughness and wear resistance [1-4]. Through suitable heat treatments or alloying additions, wear and mechanical properties of ADI could be improved by obtaining appropriate final matrix structure.

Additions of alloying elements to ADI enhance the mechanical properties and make it more suitable for commercial use. Unlike other alloying elements, nickel does not affect the carbon solubility in austenite during austenitization and in fact, aids in beneficial mechanical properties for castings [5]. Nickel was found to increase the impact toughness in ADI [6]. Previous researchers have observed that phase transformation temperature is changed by nickel addition. Nickel has the potential to eliminate the secondary carbides precipitation in the upper ausferritic range [7]. The present authors have reported elsewhere the influence of austempering temperature of 300, 320,340 and 360°C on
mechanical, wear properties and energy consumption [8]. Since not many published research articles are available on nickel, the current work is carried out at different Ni levels and different austempering temperature.

2. Experimental Procedure

2.1. Casting of ductile iron
Charging materials were calculated and melted in an induction furnace with a capacity of 1.5 MT. Figure 1 shows the dimensions of Y-blocks in which nodular iron was cast. The Y-blocks were cast along with the regular commercial products to make sure that the same treatment is given to ensure validation. The resultant chemical composition of all alloys were estimated using an optical emission spectrometer and is given in Table 1. Suitable Nickel additions to get Ni levels of 0.6%, 0.8% and 1.0% were added and they were given codes as Alloy A, Alloy B and Alloy C respectively.

Table 1. Base Chemical Composition for all Alloys.

|     | C  | Si | Mn  | S   | P   | Mg |
|-----|----|----|-----|-----|-----|----|
|     | 3.64| 2.56| 0.19| 0.002| 0.017| 0.050|

2.2. Austempering heat treatment of ductile iron
The heat treatment schedule for austempering of as-cast ductile iron samples is shown in the Figure 2. A preheating furnace was used to eliminate the impurities on the sample surface by heat treating the samples at 450°C for 10 min. In a closed austempering furnace, samples were heated to 840°C for 30 min to ensure complete austenitization. Finally austempering were carried out by quenching in a molten salt bath consisting of mixture of potassium and sodium nitrates maintained at three different temperatures of 320, 340 and 360°C. Rinsing the castings in a machine with hot water was used to wipe out the residual salt on the casting surface after austempering. The authors in their earlier study had concluded that though austempering at 300°C gives good tensile, it results in low elongation % and low impact strength and do not meet requirements of grade 1 ADI as per ASTM A897/A897M-16 specification and hence higher temperatures of 320, 340 and 360°C are selected for this study.

![Figure 1. Dimensions of Y-Block.](image1)

![Figure 2. ADI Heat Treatment Cycle.](image2)

2.3. Mechanical testing and optical microscopy
Hardness Test: The tests were done directly on the surface of the Y blocks after rough grinding. The load applied was 3000 kg and a steel ball with 10 mm diameter was used. Tensile Test: Samples with 14 mm gauge diameter and 70 mm gauge length were machined from the lower portion of the Y blocks. Impact Test: A Charpy type impact tester was used to find out the impact toughness. The rectangular samples were machined without any notch (unnotched) with dimensions of 55 x 10 x 10 mm. Metallography and Optical Microscopy: Samples were prepared for metallographic investigation by cutting, mounting, grinding, polishing and etching (using 2% Nital) to capture the images at a magnification of 100X (before etching) and 400X (after etching) using an inverted metallurgical microscope with Envision software.

2.4. X-Ray diffraction analysis
The phase analysis of samples was done in X-Pert PRO PANalytical goniometer. Samples were analysed using Cu-Kα radiation at 30 kV and 10 mA. The retained austenite content available in the matrix was calculated using direct comparison method.

3. Results and discussions

3.1. Effect of nickel on mechanical properties

The effect of different nickel content on mechanical properties of alloys austempered at 320°C is shown in Figure 3. There is an increase in yield and tensile strength when Ni is added and at 0.8% it reaches a peak and then there is a drop in value when Ni reaches 1%. Impact energy and elongation also gradually increases with increase in Ni content and reaches a maximum at 0.8% and then decreases. The same trend is observed in all alloys austempered at 340°C and 360°C as shown in Figure 4 and Figure 5 respectively.

![Figure 3. Effect of Nickel Content on Mechanical Properties of Alloys Austempered at 320°C.](image)

![Figure 4. Effect of Nickel Content on Mechanical Properties of Alloys Austempered at 340°C.](image)

3.2. Effect of nickel on microstructure

The effect of nickel content on microstructure of alloys austempered at 320°C, 340°C and 360°C is shown in Figures 6, 7 and 8 respectively. The microstructure of all alloys consists of a mixture of acicular ferrite in a carbon stabilized/enriched austenite matrix and this mixture is called as ausferrite. From the micrographs, it is known that the structure is changing from coarse to fine with increasing Ni content when austempering temperature is kept constant. However, with increase in austempering temperature, the microstructure becomes finer with more amount of ausferrite. The nodularity and nodule count of all alloys austempered at three different temperatures are shown in Table 2.
**Figure 5.** Effect of Nickel Content on Mechanical Properties of Alloys Austempered at 360°C.

**Figure 6.** Effect of Nickel Content on Microstructures (400X) of Alloys Austempered at 320°C.

**Figure 7.** Effect of Nickel Content on Microstructures (400X) of Alloys Austempered at 340°C.
Figure 8. Effect of Nickel Content on Microstructures (400X) of Alloys Austempered at 360°C.

Table 2. Nodularity and Nodule Count.

| Nickel (Wt %) | Nodularity (%) Austempered at | Nodule Count (/mm²) Austempered at |
|--------------|-------------------------------|-----------------------------------|
|              | 320°C | 340°C | 360°C | 320°C | 340°C | 360°C |
| 0.0 [Alloy X]| 92.7  | 96.2  | 94.6  | 141   | 139   | 130   |
| 0.6 [Alloy A]| 95.5  | 93.3  | 90.6  | 159   | 156   | 145   |
| 0.8 [Alloy B]| 95.4  | 95.1  | 95.6  | 237   | 208   | 200   |
| 1.0 [Alloy C]| 97.3  | 93.1  | 97.9  | 178   | 174   | 133   |

In the case of nodularity%, no trend is observed except that it is constantly above 91% for all alloys austempered at various temperatures. However, the nodule counts decreases with increase in austempering temperature and the value is maximum for 0.8% Ni addition as shown in Figure 9. This is reflected in the mechanical properties of all alloys.

Figure 9. Effects of Nickel Content and Austempering Temperature on Nodule Count.

3.3. Effect of Nickel on Retained Austenite

The XRD patterns were studied to identify the intensities and peak positions of austenite \{111\} plane and ferrite \{110\} plane at different nickel content as shown in Figure 10, 11 and 12. The retained austenite content were estimated using the intensities of the above planes using the equation (1) [9],

\[
\frac{I_y(hkl)}{I_a(hkl)} = \frac{R_y(hkl) X_y}{R_a(hkl) X_a}
\]

(1)
Where $I_\gamma(hkl)$ is the integrated intensity of a plane in austenite ($\gamma$) phase; $I_\alpha(hkl)$ is the integrated intensity of a plane in ferrite ($\alpha$) phase; $R_\gamma(hkl)$ and $R_\alpha(hkl)$ are constants.

(a) 0.6%  
(b) 0.8%  
(c) 1.0%

**Figure 10.** XRD patterns of samples with different nickel content austempered at 320°C.

(a) 0.6%  
(b) 0.8%  
(c) 1.0%

**Figure 11.** XRD patterns of samples with different nickel content austempered at 340°C.

(a) 0.6%  
(b) 0.8%  
(c) 1.0%

**Figure 12.** XRD patterns of samples with different nickel content austempered at 360°C.

Figure 13 shows a plot of the retained austenite content against nickel content at different austempering temperature. The retained austenite content was found to reach a maximum value at 0.8% Ni irrespective of austempering temperature.

**Figure 13.** Effect of nickel content on retained austenite at different austempering temperature.
From the results, it is evident that all properties are increasing with nickel content up to 0.8% and then it decreases which is reflected in both nodule count and amount of retained austenite present in the matrix. This could be due to the fact that nickel, as a solute element, dissolved in the matrix interacts with dislocations and elements segregated near dislocations by forming Cottrell atmosphere. This Cottrell atmosphere increases the dislocation motion resistance and pinning constant which reflects in the brittleness of the ductile iron with nickel content above 0.8% [10].

4. Conclusions
- The strength and hardness of alloys increases with increase in Ni content and it is maximum at 0.8% and then decreases. Impact energy and elongation also exhibited the same behavior at all austempering temperatures.
- The microstructure of austempered alloys were a mixture of acicular ferrite and carbon stabilized austenite. The structure becomes finer with respect to increase in Ni content.
- The nodularity is above 90% for all alloys irrespective of austempering temperature. The nodule counts increase with increase in Ni content up to 0.8% and then it decreases with increase in austempering temperature.
- Without affecting impact, elongation and hardness, Ni addition of 0.8% is optimum and results in giving 20% increase in yield strength and 7% increase in tensile strength.
- The study of X-ray diffraction of austempered samples reveal that the retained austenite reaches maximum at 0.8%Ni in all 3 austempering temperatures studied and then it reduces on further increase of Nickel.
- Among the three austempering temperatures, 340°C gives optimum properties with respect to yield and tensile strength without affecting impact and elongation.

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