Influence of Austempering Heat Treatment on Microstructure and Mechanical Properties of Medium Carbon High Silicon Steel

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Abstract. In the present investigation, the influence of austempering heat treatment on the microstructure and mechanical properties of medium carbon high silicon steel was evaluated. The test specimens were machined from the as-received steel and were first austenitised at 900 °C for 45 minutes, followed by austempering heat treatment in salt bath at various temperatures 300 °C, 350 °C and 400 °C for a fixed duration of two hours, after that those specimens were air-cooled to room temperature. The characterization studies were carried out using optical microscope, scanning electron microscope (SEM) and x-ray diffractometer (XRD) and then correlated to the hardness and tensile properties. Results indicate that, the specimens austempered at lower temperature i.e. at 300 °C, which offered high hardness, tensile strength and lower ductility (1857 MPa and 13.3 %) due to the presence of acicular bainite i.e. lower bainite and also some martensite in the microstructure. At 350 °C, reduction in the tensile strength and hardness was observed, but comparatively higher ductility, which was favored by the presence of bainite laths i.e. upper bainitic structure along with higher retained austenite content. Finally at 400 °C, reduction in both ductility and tensile strength was observed, which is due to the precipitation of carbides between the bainite laths, however good strain hardening response was observed at austempering temperatures of 350 °C and 400 °C.

1. Introduction
Steels with high strength and ductility are generally preferred in railways and automotive industries, inorder to fulfill the above prerequisite. Hence researches have been engaged towards evolution of advanced high strength steels. Austempered high silicon steels are developing as an exceptionally encouraging class of steels, as they offer predominant blend of strength and ductility, excellent wear resistance and formability at low costs [1]. The mechanical properties of austempered high silicon steel was found to override those of hardened and tempered steel of same hardness basically as a result of its microstructure that comprises of fine bainite laths and retained austenite, which was achieved by isothermal holding technique. The high strength is because of the fineness of bainite laths and retained austenite as films was most alluring keeping in mind the end goal to accomplish great blend of strength and ductility [2]. Addition of manganese advanced the development of balanced out retained austenite and the presence of silicon (at least 1.5 %) has poor dissolvability in cementite, in this manner prompts hindrance of cementite in the matrix [3].
The present study aims to establish microstructure-mechanical property correlation and also investigate the strain hardening response of AISI 9260 high silicon steel subjected to varying austempering temperatures.

2. Experimental procedure

The test material used for the present research work was AISI 9260 steel in the form of hot rolled bars of 15 mm diameter and 1000 mm long. The chemical composition of the as-received steel was checked using optical emission spectrometer, with composition details shown in table 1.

Table 1. Chemical composition of the steel (weight %)

| C  | Si  | Mn  | Cr  | Ni  | Mo  | Cu  | V   | S   | P   |
|----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 0.58 | 2.01 | 0.82 | 0.12 | 0.019 | 0.007 | 0.009 | 0.002 | 0.006 | 0.012 |

Tensile specimens were machined from the as-received steel bars with a gauge length of 30 mm and gauge diameter of 6 mm, as per ASTM-E8M standards [4].

To perform austempering heat treatment, both tensile samples and dummy samples (mainly used for microstructural analysis) were first austenitized at 900 °C for 45 minutes in a high temperature muffle furnace, followed by isothermal treatment carried out in a resistance heated salt bath furnace. The salt blend comprises of 55% KNO₃ and 45% NaNO₃ by weight. This composition has a wide working temperature range varying from 222 °C to 540 °C. Accordingly austempering temperatures of 300 °C, 350 °C and 400 °C were selected and heat treatment was carried out for a fixed duration of 2 hours.

Microstructural analysis of the austempered specimens after etching with 2% nital was carried out using Zeiss AXIO Lab.A1™ optical microscope and JEOL JSM-6380LA scanning electron microscope (SEM).

X-ray diffractometer (XRD) analysis was performed using JEOL-JDX-8P XRD (operated with Cu-Kα radiation at 30KV and 20mA). The 2θ range was selected between 40° to 50° at a scan speed of 1°/min. The volume fraction of retained austenite was determined using equation (1) mentioned below [5]

\[ V_\gamma = \left( \frac{I_\gamma}{I_\alpha} \right) + 1.4 \]  

Where \( I_\gamma \) and \( I_\alpha \) are the integrated intensities of retained austenite and ferrite peaks respectively.

The tensile specimens were machined as per ASTM E8M standards and were tested in a Shimadzu AG-X plus™ tensile tester of 10 ton capacity and with a fixed crosshead speed of 1mm/min. The hardness tests of all the austempered samples was measured using a Shimadzu HMV-G 20ST Vickers hardness tester with a 1 kg load for dwell time of 15 seconds. Average of at least six readings was reported as hardness values.

3. Results and Discussion

3.1 Microstructural studies

Figure 1 demonstrates the microstructure of austempered test specimens comprising of dark etched bainitic ferrite, retained austenite films (white areas) and furthermore nearness of some martensite (Figure 2(a)). The figure 1(a) shows a very fine needle like bainitic ferrite structure and less retained austenite content for specimens austempered at 300 °C due to excessive rate of nucleation. Microstructure gains coarseness with increasing austempering temperature (Figure 1(b & c)).
Figure 1. Optical micrographs of specimens austempered for fixed duration of 2 hours at various temperatures (a) 300 °C (b) 350 °C (c) 400 °C.

Figure 2. Scanning electron micrographs of specimens austempered for fixed duration of 2 hours at various temperatures (a) 300 °C (b) 350 °C (c) 400 °C.

While increasing the austempering temperature from 300 °C to 350 °C (Figure 2(b)), were the carbon diffusion rates are increasing and leads to increased growth rates, which prompts the extending of bainitic ferrite laths and the retained austenite (film shaped and blocky) between the adjoining bainite thickens and furthermore some acicular bainitic ferrite was seen in the microstructure. The microstructural features of high silicon steel sample austempered at higher temperature of 400 °C for duration of two hours, which has extremely high carbon diffusion rates, due to which the growth rates are likewise high, thereby more extensive acicular ferrite and retained austenite in blocky form are observed as shown in figure (1 and 2)(c)). The more extensive acicular ferrite (lath morphology) is otherwise called upper bainite. Moreover, isothermal holding for a longer time may trigger the secondary reaction in which fine carbides would be precipitated in retained austenite, there by prompts the diminishment in the retained austenite content, which was affirmed from XRD examination appeared in figure 3(b) beneath.

3.2 X-ray diffraction (XRD) studies
XRD analysis was adopted to quantitatively estimate retained austenite and bainitic ferrite contents.

Figure 3. (a) XRD profiles of the specimens austempered for 2 hours at 300 °C, 350 °C and 400 °C (b) Retained austenite content as a function of varying austempering temperatures.
Figure 3(a) shows the XRD patterns for specimens austempered at various temperatures, the first peak corresponds to retained austenite i.e. γ(111) and the very next peak belongs to ferrite α(110). The XRD pattern shows a slight shift in austenite peak towards lower 2θ with varying temperatures, which indicates the carbon enrichment in retained austenite.

From figure 3(b) it was observed that austempering at lower temperature of 300 °C, contains reduced amount of retained austenite, which is because of more prominent supercooling of bainitic transformation and in this way prompts nucleation of abundance bainitic ferrite [6]. Increasing the temperature to 350 °C, here maximum amount of retained austenite was observed, which is due to lower supercooling and nucleation rates being lower, which results in fewer acicular ferrite each growing to larger size due to increased carbon diffusion rates at higher temperatures.

At 400 °C for duration of 2 hours, drastic reduction in the retained austenite content was detected, since the carbon diffusion rates are to a great degree high in order to deal with the request of phase transformation, therefore retained austenite decreases, which apparently indicates second stage reaction has set in i.e. carbide precipitation [7].

3.3 Mechanical properties

Representative engineering stress-strain plots are shown in the figure 4(a) for austempered specimens and as-received steel.

![Figure 4](image)

**Figure 4.** (a) Engineering stress-strain plots for as-received and austempered specimens. (b) Tensile strength, % strain and modulus of toughness as a function of austempering temperatures. (c) Vickers Microhardness as a function of austempering temperatures.
Table 2. Mechanical properties of the as-received high silicon steel.

| Tensile strength (MPa) | % Strain | Modulus of toughness (MJ/m³) | Vickers Microhardness (HV) |
|------------------------|----------|-----------------------------|---------------------------|
| 748.77                 | 18.2     | 108.1                       | 325                       |

The profiles presented in the figure 4 (b & c) reveals significant improvement in tensile properties and hardness for all the austempered specimens when compared to that of as-received steel (table 2). Here modulus of toughness is estimated by integrating the engineering stress-strain curve (Figure 4(a)). At lower austempering temperature of 300 °C, the microstructure consists of acicular bainitic ferrite, retained austenite and some martensite as well, resulting in extremely high strength (1857 MPa) and hardness (575 HV) with reduced ductility (13.3%) and modulus of toughness (111 MJ/m³) on the other side. Further increase in temperature to 350 °C drastic reduction in strength and hardness, with increased ductility and modulus of toughness was observed on the other hand, which is due to the mixed morphology of bainitic ferrite in form of laths and also some acicular structure and higher amount of retained austenite (in the form of films and blocks), film morphology of retained austenite exhibits high mechanical stability that contributes for excellent strength and toughness. At elevated temperature of 400 °C further reduction in strength and hardness was observed, which is due to the excess coarseness of bainitic ferrite and reduced level of retained austenite, which is in the block form, this blocky morphology of retained austenite contains inhomogeneously distributed carbon, which leads to the mechanical instability of blocky austenite and thereby undergoes strain induced martensitic transformation while tensile loading [8]. Hence specimens austempered at 350 °C and 400 °C showed superior stain hardening response during tensile loading. Ductility and modulus of toughness at 400 °C was comparatively lower than that of 350 °C, which gives an indication of second stage reaction in austempered steels, where carbide precipitation occurs between bainitic ferrite lath and retained austenite, scanning electron micrographs slightly reveals the carbide presence (Figure 2(c)).

3.3.1 Strain hardening behavior

Figure 5 displays the plot of true stress vs true strain in a natural logarithmic scale, which was in turn generated from engineering stress-strain plot (Figure 4(a)) by considering datas from yield stress to ultimate tensile stress. The points indicated in figure 5 reveals a linear trend that persuades Holloman [9] relationship (equation (2)).

\[ \sigma = K\varepsilon^n \]  

Where \( \sigma \) is the true stress, \( \varepsilon \) is the true strain, \( K \) is the strength coefficient and the strain hardening exponent “n”, which is the slope of the linear trendline.

Figure 5. True stress as a function of true strain in logarithmic scale for as-received and austempered specimens
In figure 5 it can be observed that the strain hardening behavior plots for the as-received and specimen austempered at 300 °C for 2 hours showed a linear trend with a single “n” value of 0.08 and 0.04 respectively, which implies that there is no variation in the strain hardening behavior during tensile loading. For the specimens austempered at 350 °C and 400 °C for 2 hours shows variation in the slope, which shows multiple “n” values i.e. (0.18 and 0.08) for 350 °C and (0.09 and 0.16) for 400 °C respectively, which implies continuous phase transformation during tensile loading. In addition to this Vickers microhardness test (load= 15 grams and dwell time= 30 seconds) was also carried out on the polished and etched surface of the fractured tensile specimens and were found to have a maximum value of 873 HV, which confirms the transformation of block shaped retained austenite to strain-induced martensite.

4. Conclusions
The following conclusions can be drawn from the present study on microstructural aspects and mechanical behaviour of AISI 9260 high silicon steel

- High silicon steel austempered over a temperature range 300 °C to 400 °C generates bainitic structure mainly consisting of bainitic ferrite and retained austenite. Variation in the ferrite morphology from acicular to lath structure was observed at higher austempering temperatures of 350 °C and 400 °C.
- Retained austenite content initially increases from 300 °C upto 350 °C then decreases at 400 °C, which is due to reduction in degree of supercooling and also due to the second stage reaction (carbide precipitation) of bainitic transformation in steels.
- Tensile strength and hardness decreases with increasing austempering temperature from 300 °C to 400 °C and on the other hand ductility and modulus of toughness were found to be maximum at 350 °C.
- Strain hardening exponent values was found to be high at 350 °C and 400 °C, which implies that the transformation from block shaped retained austenite to strain induced martensite is more predominant at higher austempering temperatures.

5. References
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