Influence of Austempering Heat Treatment on Microstructure and Mechanical Properties of AISI 9255 High Silicon Steel

Maheshwar A. Hipparagi[1], Padmayya S. Naik[2], Ravishankar K. S.[3]

[1] Assistant Professor, Mechanical Engineering Department, Visvesvaraya Technological University. Belagavi, Karnataka, India

[2] Professor, Mechanical Engineering Department, Visvesvaraya Technological University. Belagavi, Karnataka, India

[3] Professor, Metallurgical and Materials Engineering Department, National Institute of Technology, Surathkal. Karnataka, India

Abstract

The present work is mainly centered on austempering of high silicon steel at different austempering conditions and its influence on microstructure and mechanical properties of AISI 9255 high silicon steel. The tensile specimens machined as per the ASTM standard were initially austenitized at three levels of temperatures namely 850°C, 900°C and 950°C for 30 minutes.Austempering was done in a molten salt bath at different levels of temperatures namely 280°C, 320°C, 360°C and 400°C for 90 minutes.

X-ray diffractometer, scanning electron microscope and optical microscope were used to characterize all heat-treated specimens. At lower austempering temperatures namely 280°C and 320°C the samples exhibit lower bainitic microstructure while the higher temperatures such as 360°C and 400°C exhibited upper bainitic microstructure. The specimen austempered at 280°C for 90 minutes after austenitizing at 850°C for 30 minutes showed highest UTS of 1680 MPa with a percentage elongation of 8.7 percent.

***Keywords**. Austenitizing, Austempering, Bainitic, High silicon steel, Tensile, Hardness.*

INTRODUCTION

The Austempering of Ductile Cast Iron and its mechanical property assessment has been the subject of a series of investigations around the world for the past 20 years. This is because of the unique microstructure which imparts excellent mechanical properties to the ductile iron such as strength and toughness with reasonably good ductility [1]. Additional advantages of ADI also comprise reduced manufacturing costs owing to its great machinability and outstanding castability, which results in more time tool lives [2]. Nevertheless, the ADI's graphite nodules frequently result in cracks, which lower the product's mechanical properties. Because of this, the austempered steel have attracted the researcher's interest particularly in automotive industry in recent years. Research on austempered high silicon steel with a comparable austempered composition has demonstrated the excellent mechanical properties like strength, hardness, and toughness in comparison to ADI, and can defeat the limitations of ADI [2]. An excellent ferrite strengthener is silicon. hardenability benefits from manganese content below 2%, which helps to strengthen solid solutions [3]. Steels that are AISI 9255 spring steel, having chemical arrangements of 0.56–0.61 carbon, 1.9–2.22 silicon, & 0.75–0.96 manganese, are among these steels. When these steels properly heat-treated have a high elastic limit and fatigue strength. Consequently, these steels work well to create knives for the manufacture of wood pellets, in addition to leaf springs, coiled springs, chisels, punches, and reversible shovels in cultivators. The most popular heat treatment is austenitizing, which is followed by austempering and oil quenching [4]. Numerous articles claim that adding pro-eutectoid ferrite to the finished structure will improve ADI's manufacturability [5].

It is well understood fact that ferrite and carbide are the microconstituents in an austempered steel. But according to research reports, because silicon severely inhibits the development of carbide, the high silicon steel's bainitic structure is composed of baiite plates and light coatings of preserved austenite that has been enhanced with carbon rather than carbide in the lath or inter lath [6]. Increased stability brought on by carbon-enriched austenite reduces the percentage of residual austenite that transforms into martensite. One class of bainitic steels that can be employed as high-performance alloys with good toughness and tribological qualities has strengths greater than 1200 MPa and an elongation of 13%. Superior mechanical properties were obtained when the specimens are subjected to 2 step heat treatment comparison with single-step process [7].

Despite the fact that work done on microstructural studies and mechanical behavior of high silicon steels, a thorough comprehension of the structure – property correlation is yet to be studied. This work's primary goal is to investigate how different austempering and austenitizing temperatures affect the tensile properties of high silicon steel. The investigation is also focused to arrive at an optimum austempering heat treatment to obtain optimum mechanical properties.

WORK IN EXPERIMENTATION

Material in proposed work.

The material for the present work is AISI 9255 steel and which is obtained from Mumbai, India, and prepared as cylinder-shaped rods with 14 mm diameter and 100 cm length. The chemical composition of the cylinder-shaped rods were resolved by optical emission spectrometer and the result is shown in Table 1.

Table 1. Chemical structure of AISI 9255 steel.

Constituent	C	Si	Mn	Cr	Ni	Mo	Cu	V	S	P
Wt.pct	0.61	1.95	0.82	0.14	0.11	0.02	0.17	0.18	0.01	0.02

Machined samples for tensile testing, hardness measurement and characterization purpose were machined from the above said cylindrical blocks.

Heat Treatment.

The austenitization of the samples was done using a heat resistance muffle furnace with an accuracy of + or – 5ºC whereas the austempering was done in bath of molten salt, which contained a combination of 56 percent KNO3 and 44 percent NaNO3 by weight. The salt mixture was selected to get a different working temperature ranging from 175°C to 540°C. The specimens were austenitized at three levels of temperatures namely 850°C, 900°C, and 950°C for 30 minutes. These temperatures were selected with expectation that higher amount carbon content that can be retained in the austenite after successful austempering process.

After complete austenitization of the samples at one of the above said temperatures, these were quickly transferred to a salt bath for the austempering reaction. The austempering was done at four levels of austempering temperatures namely 280°C, 320°C, 360ºC and 400°C for 90 minutes. These temperatures were so selected in such a way that the lower temperatures namely 280°C, 320°C results in lower bainitic microstructure while the higher temperatures such as 360°C and 400°C would impart upper bainitic microstructure in the AISI 9255 steel. It should be noted that the austenitizing time and austempering time were not considered as variables and kept constant.

Metallography.

The austempered specimens under each heat-treated conditions were subjected to qualitative and quantitative analysis. The qualitative examination of the samples after polishing and etching with 3 percent nital was done using an optical microscope at different magnifications. This analysis helped in revealing the existence of quantity of retained austenite, bainitic ferrite and the way these phases are distributed inside the material's matrix. However, the exact amount of these phases was calculated by X-ray diffractometer. On a Jeol JDX-8P diffractometer, the samples were scanned using copper $K_\alpha$radiation in the angular 2θ, range of 40º – 50º at a scanning rate of 0.25 degrees per minute. Cullity [8] suggested the method to calculate the percentage of ferrite and retained austenite. A planimeter was used to measure the area under the peaks in order to determine the peaks for these samples. The following relationship was then utilized to calculate the percentage of retained austenite and ferrite.

$$\frac{I\gamma}{I\alpha} = (R\gamma * X\gamma)/(R\alpha * X\alpha) \quad \rightarrow (1)$$

where Iγ and Iα are the (111) peak of austenite and (110) peak of the ferrite respectively; Xα and Xγ are the ferrite and austenite volume fractions respectively. Rα and Rγ (Constants) are given by the following relationship

$$R = [\frac{1}{V2}]\,[F2.p(LP)]e^{-2m} \text{-------------} \rightarrow (2)$$

The carbon content of the preserved austenite was an additional significant microstructural characteristic that was determined by utilizing the diffraction profile. This was done by using the following relationship.

$$a\gamma = 0.35488 + 0.00844C\gamma \quad \rightarrow (3)$$

Hardness Measurement

Shimadzu MicroVicker's hardness with a load range from 10 gm to 1000 gm was used to study the variation of hardness imparted due to the variation in austempering conditions. Each heat-treated condition required at least five readings, and the values presented are the mean of the closer values.

Tensile Test

Tensile test was conducted as per ASTM E8 standard [9]. Figure 1. reveals dimensions of the tensile test specimen. Test was conducted on Shimadzu AG-X UTM with a 100 kN capacity and 1 mm/min crosshead speed. Three specimens were considered under each heat-treated condition and the tensile properties taken as the average values of these three values.

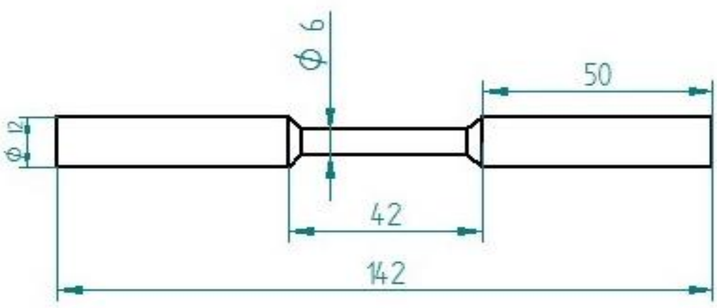

Figure 1. Dimensions of tensile test specimen in mm.

Fractography

The fractography of the tensile samples were analyzed by using a JEOL make analytical SEM at different magnifications to elicit the information regarding the type of fracture occurred in the steel under different austempering conditions. The instrument is also used to observe the microstructure at higher magnifications since the microstructural observation under ordinary light microscope is limited to 1000X.

RESULT AND DISCUSSION

Microstructural studies

Microstructure of AISI 9255 steel shown in Figure 2. that consists of pearlite and a very little percent of pro eutectoid ferrite in the matrix of the material. The microstructural studies of the specimens austempered at different temperature at three levels of austenitizing temperature are shown in Figure 3 (a) to (d), 4 (a) to (d) and 5 (a) to (d). In these Figures, the dark etched sheaves represent the bainitic ferrite while the white etched regions are the retained austenite. It can be observed that, for a given austenitizing temperature (say 850ºC), the lower bainitic ferrite occurred in the form of needles which are separated by thin films of austenite as given in Figure 3(a) and (b). With increasing austempering temperature to 360ºC and 400ºC, the quantity of retained austenite also increased considerably along with a change in the morphology of ferrite from needle to feathery as shown in Figure 3(c) and (d) respectively. A similar trend can also be observed for the other two austenitizing temperatures namely 900ºC and 950ºC with increasing austempering temperatures as shown in Figures 4 (a) to (d) and 5 (a) to (d) respectively.

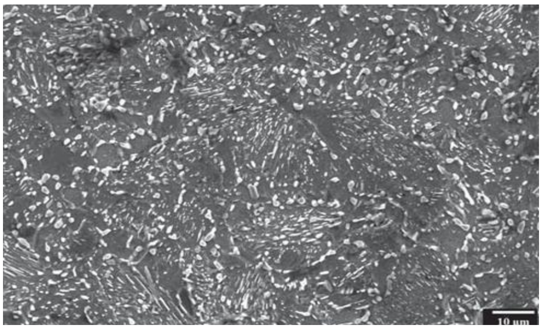

Figure 2. Microstructure of as-received AISI 9255 high-silicon steel.

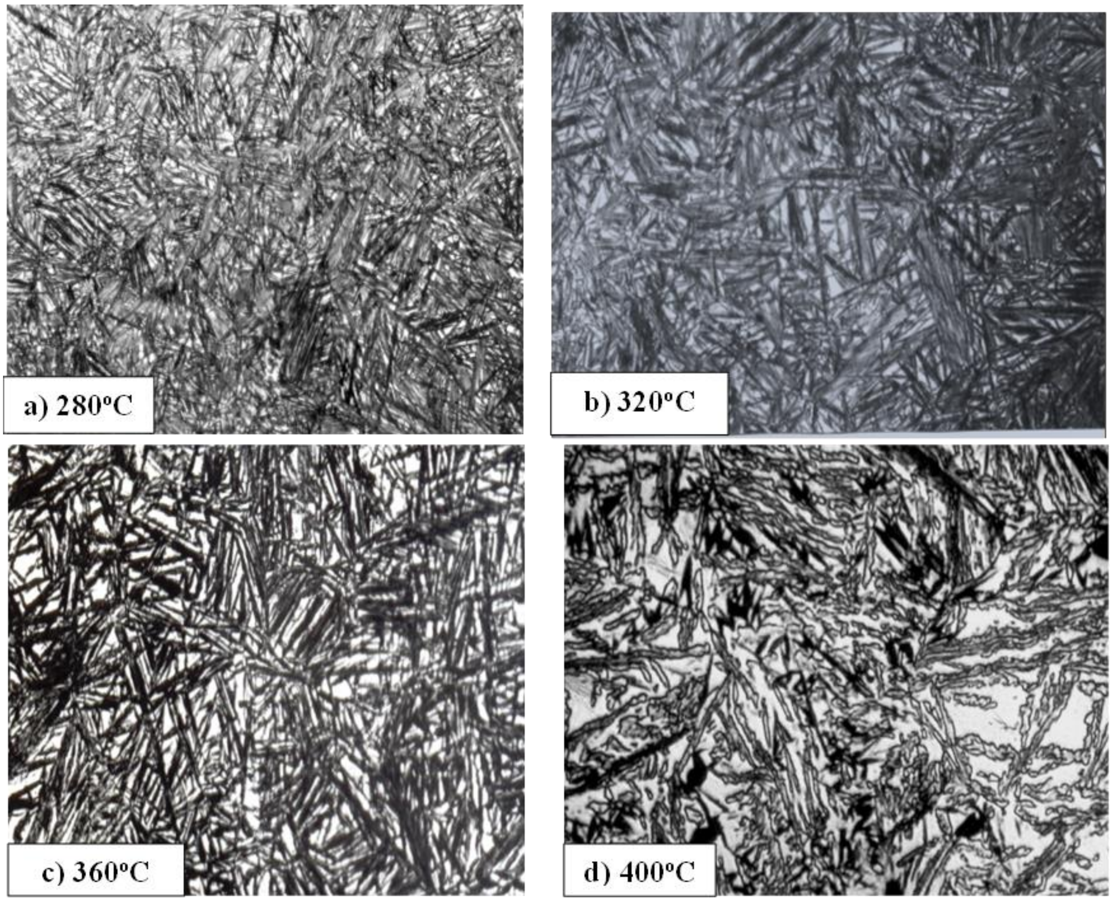

Figure 3. Change in microstructure with increasing austempering temperature for the austenitizing temperature of 850°C

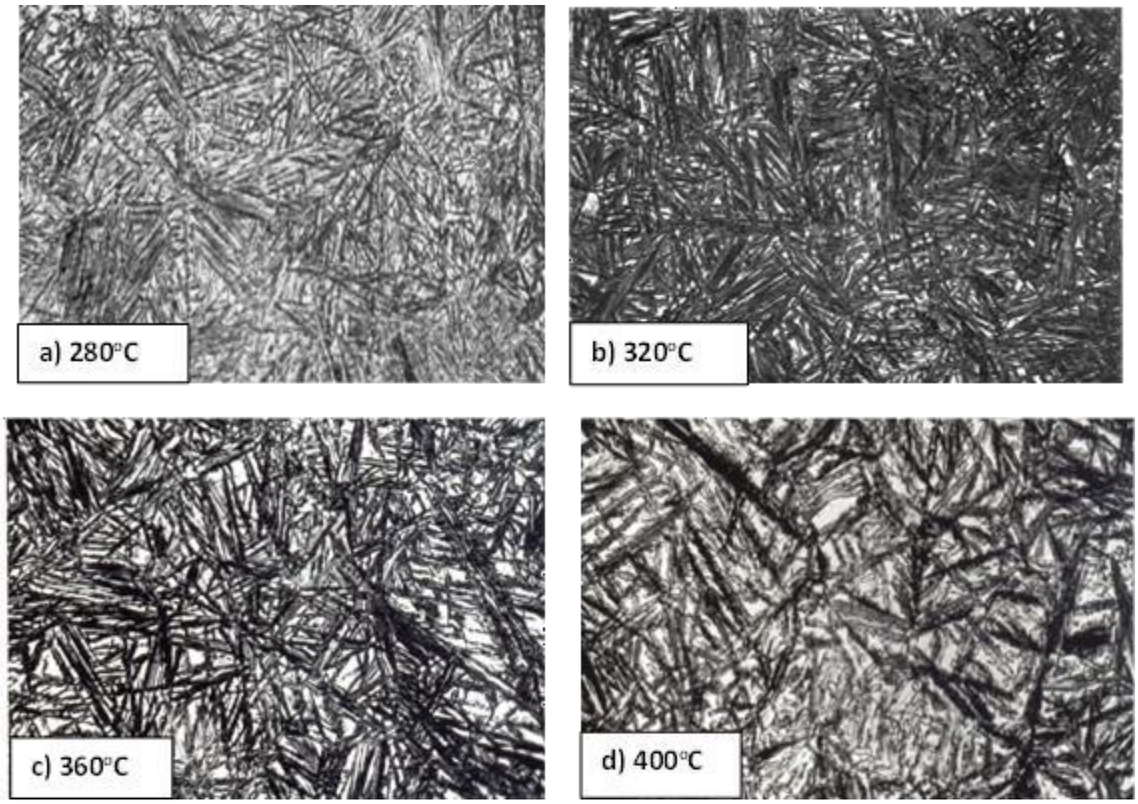

Figure 4. Change in microstructure with increasing austempering temperature for the austenitizing temperature of 900^{0} C

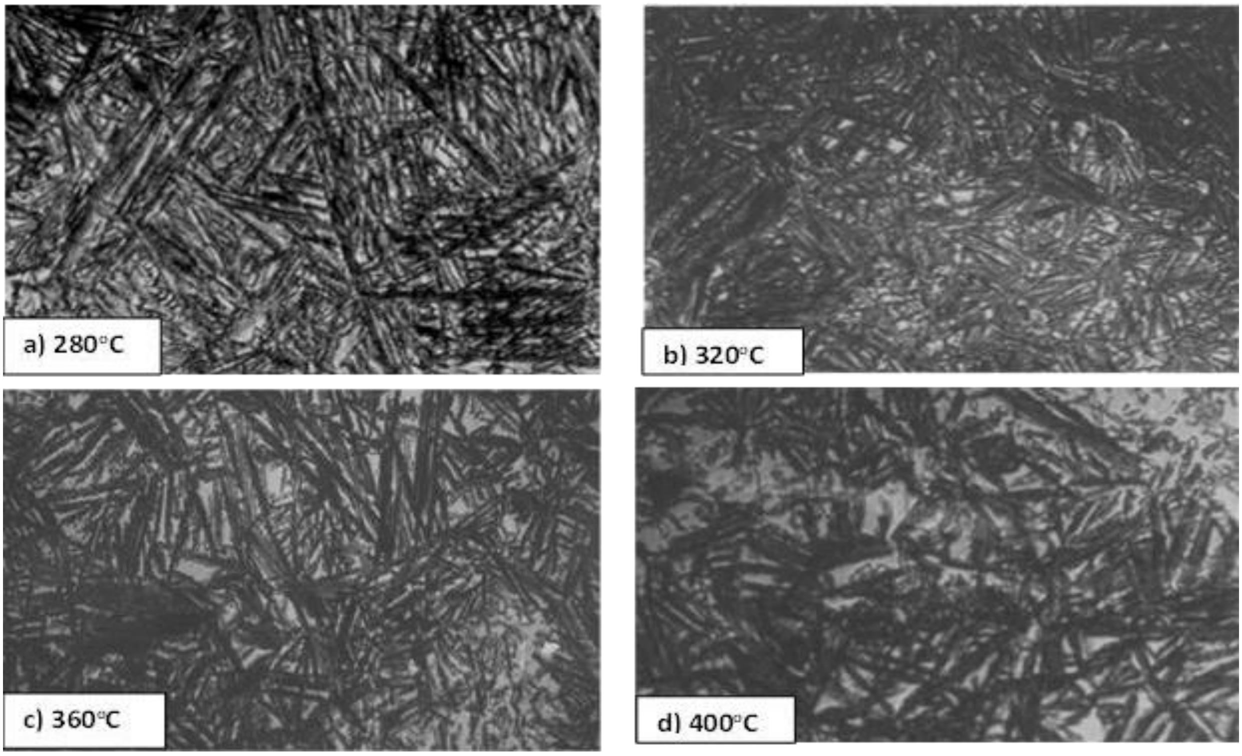

Figure 5. Change in microstructure with increasing austemp8ering temperature for the austenitizing temperature of 950ºC

The qualitative study of the austempered specimens at different temperatures for 90 minutes at the three levels of austenitizing temperatures was carried out using optical microscope. The figures 3,4 and 5 show some of the representative areas taken at the magnification of 500X. The black needle shape like structure represents the ferrite and white background is the retained austenite. It is clear from the figure 7 that as the temperature of the austempering process was raised, more retained austenite was also present. Apart from this, the fine bainitic needle shape ferrite has changed to feather type with increasing austempering temperature for a given austenitizing temperature. It should be mentioned that the variation in microstructure should correspond to the variation in austempering and austenitizing temperatures since the austempering and austenitizing times were kept constant within this investigation. While at 280ºC, the retained austenite is observed as fine sheath in between needle shaped fine ferrite laths; the 400ºC temperature has resulted in blocky austenite along with broad feathery bainitic ferrite in the microstructure.

The diffraction profiles obtained for different austempering conditions were obtained using X-ray diffractometer are shown in figure 6. There was observed considerable variation in intensity of austenite peak with varying austempering conditions

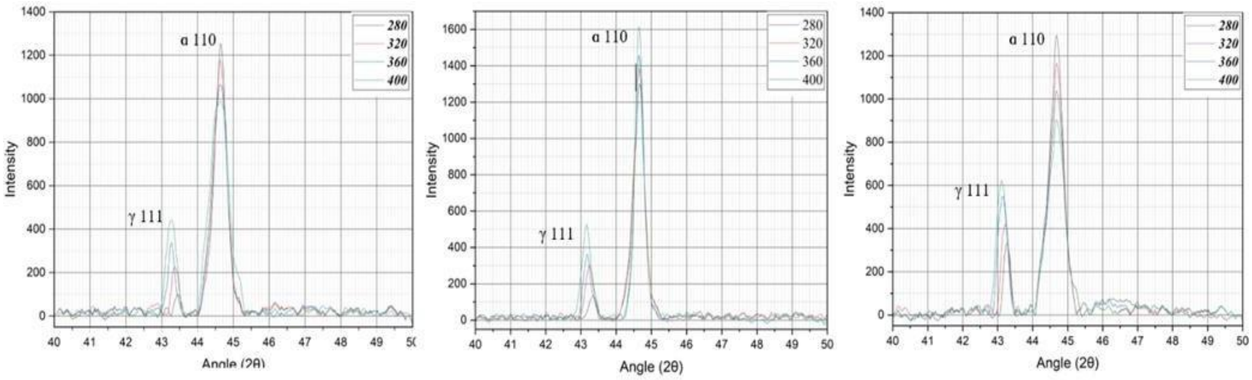

Figure 6. XRD plots for specimens austenitized at (a) 850°C (b) 900°C and (c) 950°C for different austempering temperatures.

The XRD plots above display has 2 peaks in the 40–50° region of the 2θ angle. Details regarding the quantity of retained austenite are provided by the first peak from the (111) austenite planes (γ). The quantity of bainitic ferrite present in the matrix is shown by the second peak (α) from the (110) ferrite planes. The XRD pattern shows a shift in the austenite peak towards lower 2θ, indicating an increase in carbon content in the retained austenite as the austenitizing temperature rises. Figure 7. shows the retained austenite variation with rise in austempering temperatures for the three levels of austenitizing temperatures. There can be observed a systematic shift in the percentage of retained austenite with increasing austempering temperature for a given austenitizing temperature. It is possible to explain this rise in retained austenite with rising austempering temperatures by taking into account the different transformation mechanisms at different austempering temperatures.

At lower austempering temperatures such as 280ºC, there will be more nucleation of ferrite because of large undercooling; but the growth of these already nucleated ferrite is very slow due to very slow diffusion of the carbon from the ferrite at low temperatures. However, at higher austempering temperatures like 400ºC, even though nucleation rate is low but its growth rate is high because of the quick diffusion. Therefore, at lower austempering temperatures; one can observe smaller percentage of austenite compared to that of higher temperatures.

In the current study, the retained austenite increases from a low value of 5 volume percent at 280ºC to 24 volume percent at 400ºC for the austenitizing temperature of 850ºC whereas it has increased from 14 volume percent at 280ºC to 38 volume percent at 400ºC for the austenitizing temperature of 950ºC. It can also seen that the curve has shifted to upwards with increasing austenitizing temperature.

Another important parameter that was estimated using XRD is carbon content in the retained austenite, which decides its stability and ability to provide the resistance to dislocation movement during deformation. A major alteration in the position of the austenite peak was observed with changing austempering parameters. It is recognized that [10] increase in the content of carbon in austenite by 0.1 wt. % will increase the lattice parameter of FCC structure by 0.0044Aº.

Figure 8. gives the change of content of carbon in retained austenite with rise in austempering temperatures at three levels of austenitizing temperatures. It can be observed that the carbon content raised from 1.32 weight percent to 1.68 weight percent as austempering temperature was raised from 280ºC to 400ºC respectively for the austenitizing temperature of 850ºC. Similar increasing trend can be observed for the other austenitizing temperatures too.

The bainitic transformation of steels provides an explanation for the retained austenite and increased carbon content. The steel is the fully austenitic state during austenitization and bainitic transformation starts when it is transferred to the salt bath. The transformation is mainly dependent on the temperature. At low austempering temperature (say 280 ºC), the ferrite nucleates at the grain boundaries with the rejection of carbon. Since the silicon content is high, it suppresses the carbide precipitation and the rejected carbon will be absorbed by the surrounding austenite. But, due to the low temperature, the diffusion of carbon to the surrounding austenite is sluggish and results in low carbon content. But at higher austempering temperature such as 400ºC, there will be faster diffusion of carbon and as a result of this, more austenite will be stabilized on completion of austempering process.

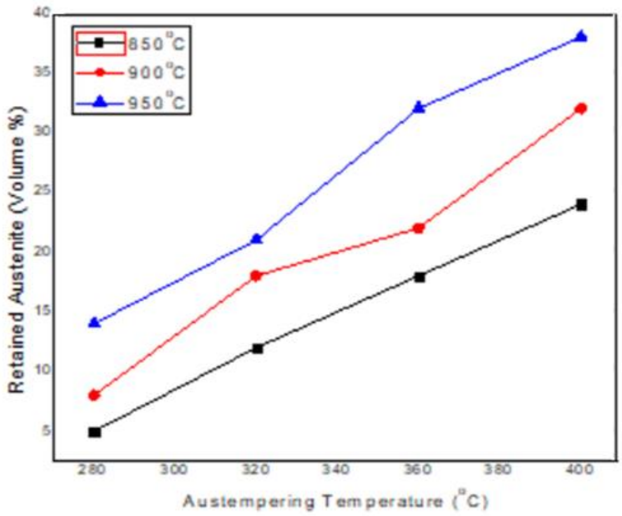

Figure 7. Differing austenitizing temperatures at various austempering temperatures result in divergence of retained austenite.

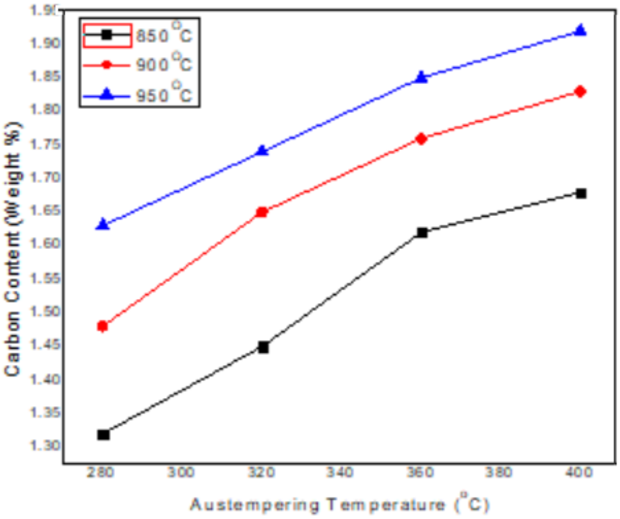

Figure 8. Differing austenitizing temperatures at various austempering temperatures result in divergence of carbon content

Mechanical properties

Hardness:

Hardness measurements are made using Shimadzu MicroVickers hardness tester for the samples austempered at different temperatures are shown in Figure 9. which shows that as the austemepring temperature increases, hardness decreases. The decreasein hardness with austempering temperature is attributed to the increased volume faction of retained austenite along with changeover of the bainitic ferrite from needle shape to feathery. With increasing austenitizing temperature, the decreasing trend of hardness with increasing austempering temperature remained same but the curve has shifted to lower values because of the more percentage of retained austenite occurring at the end of austempering after austenitizing at higher temperatures.

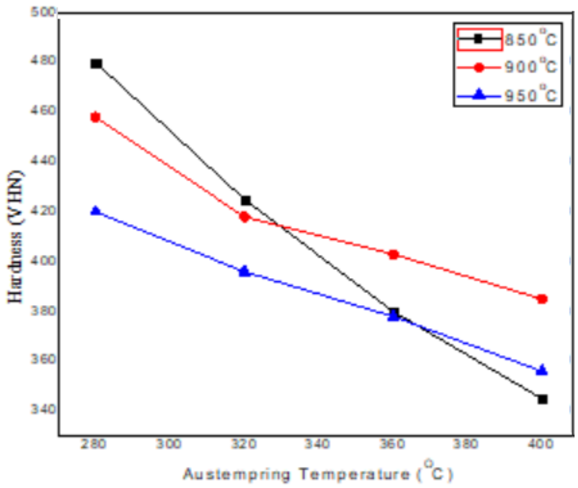

Figure 9. Differing austenitizing temperatures at various austempering temperatures result in divergence of micro hardness

Tensile properties:

The dependence of tensile properties namely UTS, YS and % elongation on austempering temperature at the three levels of austenitizing temperature is shown in Figures 10 to 12 respectively. An increasing trend of percentage elongation with a decreasing trend of strength is evident from these figures. The strength decreases with increasing austempering temperature up to 360°C and remained more or less a constant except for the austempering temperature of 400°C at the austenitizing temperature of 850°C. When the temperature of austempering rises to 360°C, it is observed that the percentage elongation increases. and thereafter the increase in only marginal. The variation of these tensile properties can be explained by considering the variation in microstructures imparted to the material at different austempering and austenitizing temperatures. Here it is worth noting that the number of phases present, its allocation and stability of the phases play an significant role in deciding the properties of the steel. As already explained earlier, at low austempering temperatures such as 280ºC at any given austenitizing temperature, the microstructure contained very fine needle like ferrite in between thin film of retained austenite while at higher austempering temperatures such as at 400ºC, the microstructure is characterized by feathery bainitic ferrite along with considerable amount of blocky austenite. In austempered ductile iron, the lower bainitic microstructure usually imparts higher strength with lower percentage elongation compared to upper bainitic microstructure [11]. In the present investigation also, this seems to be true. The presence of retained austenite as a thin film between the fine acicular like ferrites will provide higher resistance to the dislocation movement during uniaxial tensile loading. But due to the appearance of feathery bainitic ferrite and blocky austenite would help in improving the percentage elongation rather than strength. Due to this reason, one can observe increase in percentage elongation with increasing austempering temperature for a given austenitizing temperature. However, UTS and YS of the austempered steel was increased when it was austempered for 2 hours at 400ºC irrespective of the austenitizing temperature. This seems to be some exceptional case. Here it should be worth noting that the UTS and YS values from 360ºC to 400ºC remained more or less a constant or marginal increase for the austenitizing temperature of 900ºC and 950ºC, while these values increased for the austenitizing temperature of 850ºC. Since the mechanical properties depend on the microstructure of a given material, in the present case also, it should be attributed to the microstructure. One of the possibility to have an increase in the UTS and YS may be due to the strain induced martensite in the matrix of the material when the material during tensile loading. The quantity of austenite retained at the end of austempering is mainly decided by the austenitizing temperature. Increase in austenitizing temperature leads to increase the amount of retained austenite, as one can see in the present work. The amount of carbon in the retained austenite, its size, shape, and distribution within the microstructure are some of the variables that affect austenite's stability. If the carbon distribution is uneven within the blocky austenite, then during deformation it may get transformed into martensite which in turn may result in an increase in strength. In the present investigation, it is seen that the retained austenite value increases when the austempering temperature was increased from 360ºC to 400ºC with a marginal increase in carbon content. Therefore, retained austenite is not enriching with carbon and its stability is less and might have transformed into martensite.

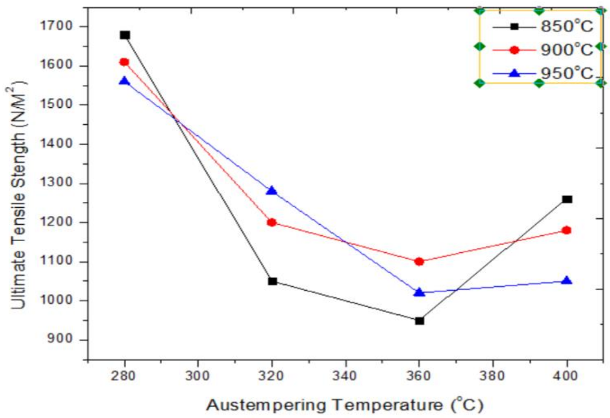

Figure 10. Differing austenitizing temperatures at various austempering temperatures result in divergence of ultimate tensile strength

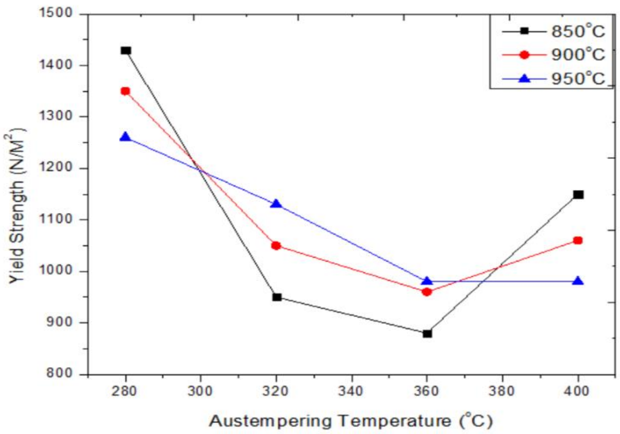

Figure 11. Differing austenitizing temperatures at various austempering temperatures result in divergence of yield strength

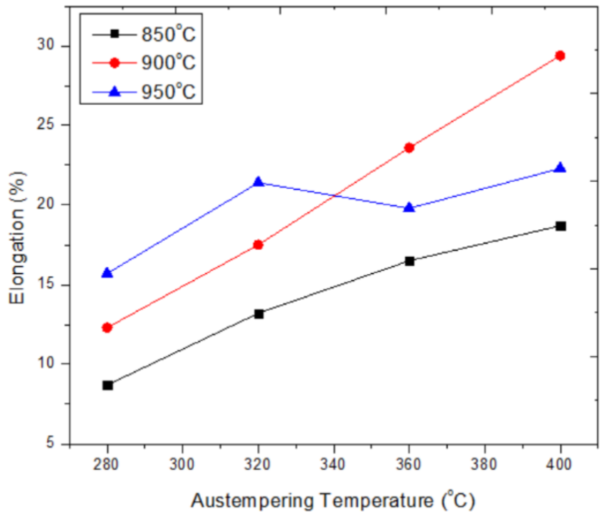

Figure 12. Differing austenitizing temperatures at various austempering temperatures result in divergence of % elongation.

Fractography:

The fractured tensile samples were studied under JEOL scanning electron microscope and some representative areas are shown in Figure 13. The type of fracture surfaces observed for these austempered samples supported the obtained mechanical properties. The specimens austempered at lower temperatures such as 280°C and 320°C is characterized by considerable amount of cleavage facets which is the indicative of brittle type fracture. These cleavage facets occurred in the form discrete packets which resists the dislocation movement to a greater extent. The fracture surfaces at higher austempering temperatures such as 360°C and 400°C, the presence of dimples clearly show that the material has failed with considerable amount of plastic deformation and helped in increasing the ductility of the material.

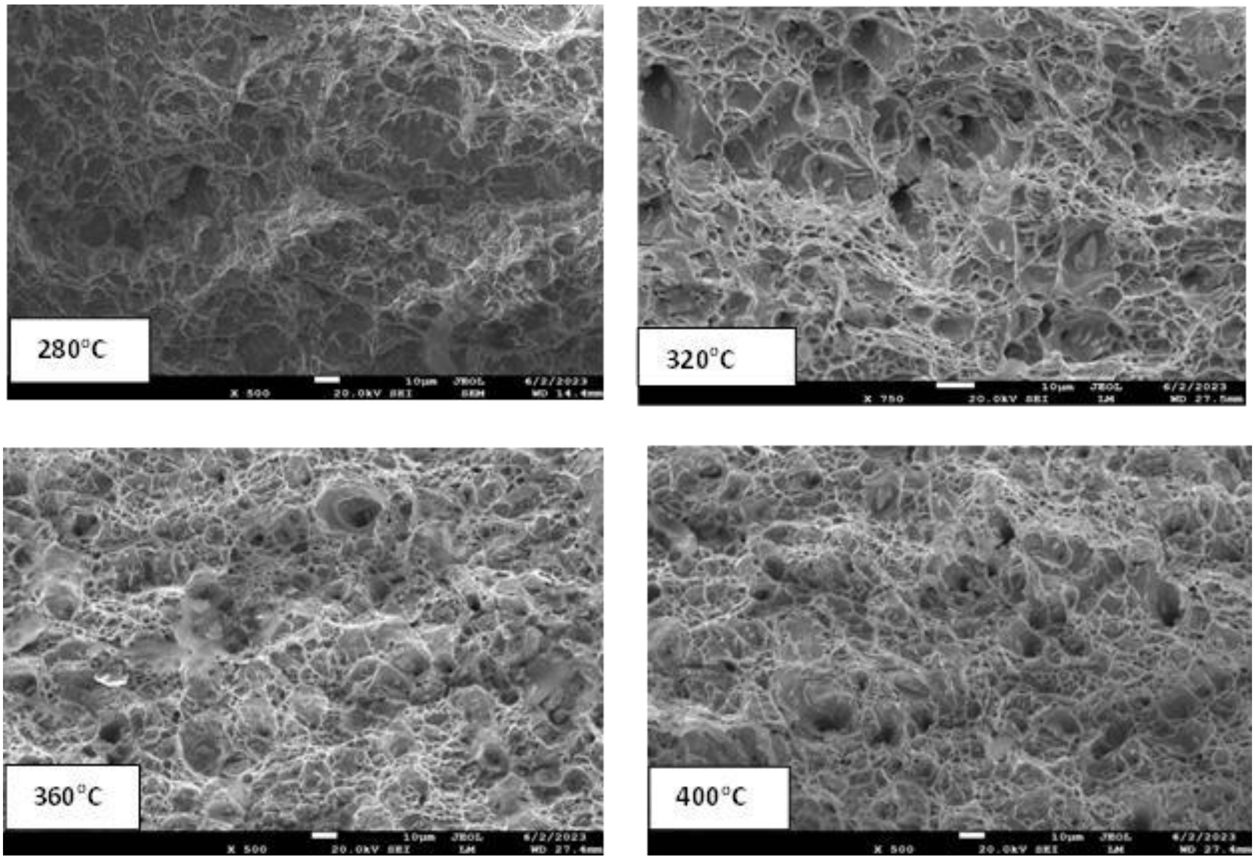

Figure 13. Fractographs of the fractured tensile samples at the austenitizing temperature of 900°C with austempering temperatures indicated

CONCLUSIONS

The present investigation is able to draw the following conclusions:

1. The lower austempering temperatures results in very fine needle shaped ferrite which are well separated by thin preserved austenite films, where as those austempered at higher austempering temperatures results in broad feathery bainitic ferrite along with blocky austenite regions.
2. The percentage of retained austenite and carbon content increase as the austempering temperature rises for a given austenitizing temperature. Furthermore, for a given austempering temperature, an increase in the retained austenite is the result of boosting the austenitizing temperature.
3. The UTS and YS decreases with increasing asutempering temperature for a given austenitizing temperature while percentage elongation shows an opposite effect also when the austenitizing temperature rises for a given austempering temperature, the same pattern holds true.
4. Increase in the hardness of the steel achieved with lower austempering and austenitizing temperatures and it decreases with increase in temperature.
5. The lower bainitic microstructure exhibits brittle type of fracture while upper bainitic microstructure results in ductile fracture. This kind of behavior is mostly caused by the top bainitic microstructure having a higher concentration of austenite than the lower one.

REFERENCES

1. Je-Young, S., Kim, J.H., Kim, W.B., and Ye, B.J. (2010). "Effects of Austempering conditions on the Microstructures and Mechanical properties in Fe-0.9%C-2.3%Si-0.3%Mn Steel. "*Metals Materials International*", June 2010, Volume 16, No. 3, pp 357-361.
2. Putatunda, S.K. (2003). "Influence of austempering temperature on microstructure and fracture toughness of a high-carbon, high-silicon and high-manganese cast steel." "*Materials and Design*", April 2003, Volume 24, pp 435-443.
3. Mandal, D., Ghosh, M., Pal, J., De, P. K., Chowdhury, G.S., Das, S. K., Das, G., Ghosh, S. (2009). "Effect of austempering treatment on microstructure and mechanical properties of high-Si steel." *Journal of Material Science*, Jan 2009, Volume 4, pp 1069–1075.
4. Rajan, T.V., Sharma, C.P., Sharma, Asssswsweq. "Heat treatment Principles and Techniques." Second edition, *PHI Learning Private limited*, India.2014
5. Mona Hussein, Ragaie M. Rashad, Mostafa Ahmed, A. Nofal, Mohamed Soliman, Lamiaa Z. Mohamed, Heinz Palkowski (2023) "Influence of Austenite Stability on The Machinability of Different Grades of Austempered Ductile Iron (Adi) During Dry Turning Machining" *Journal of metal casting*, Volume 18, pages 744-758.
6. Bhadeshia H.K.D.K and Edmonds D.V (1983) "Bainite in Silicon Steels: new composition-property part 1" *Materials science*, Sept. 1983, volume 17, pp 411-41
7. Mattia Franceschi , Alvise MiottiBettanini, Luca Pezzato ,Manuele Dabalà, Pascal J. Jacques (2021) "Effect of Multi-Step Austempering Treatment on the Microstructure and Mechanical Properties of a High Silicon Carbide-Free Bainitic Steel with Bimodal Bainite Distribution" Metals 2021, Volume 11, Issue (12), 2055
8. B.D. Cullity-Elements of x-ray diffraction-secondedition.pdf
9. ASRM e8 "Standard Test Method for Tensile Testing of Metallic Materials", Annual book of ASTM Standards,3.01, ASTM (1993),pp 542-566
10. K.B. Rundman and R.C. Klug "An x-ray and metallographic study of austempered ductile cast iron" afs transaction, 90 (1982), PP:499-508
11. Susil K Putatunda, Arjun V Singar, Ronald Tackett & Gavin Lawes, (2009), "Development of a high strength high toughness ausferritic steel" *Materials Science and Engineering: A*, Volume 513-514, 15 July 2009, Pages 329-339.
12. Palaksha Pilar Acharya, Rajendra Udupa & Ravishankar Bhat, (2017), "Microstructure and mechanical properties of austempered AISI 9255 high-silicon steel" *Materials Science and Technology*, October 2017, Volume 34, Issue 3, pp 355-365.
13. P A Palaksha and K S Ravishankar (2017)" Influence of Austempering Heat Treatment on Microstructure and Mechanical Properties of Medium Carbon High Silicon Steel" *Materials Science and Engineering*, 2017, Volume 225, Conference 1, pp 1-6.
14. P A Palaksha, P Shyamkrishna, Ravishankar K S (2017)." Effect of Austempering Heat Treatment Parameters on the Microstructure and Dry Sliding Wear Behaviour of AISI 9255 High Silicon Steel" *Science Direct*, August 2017, Volume 4, Issue 10, pp 10757–10763.
15. Bagliani, E.P., Santofimia, M.J., Zhao, L., Sietsma, J., and Anelli E. (2013). "Microstructure, tensile and toughness

properties after quenching and partitioning treatments of a medium-carbon steel." *Materials Science & Engineering*, Sept 2012, A 559, pp 486–495.

16. Li, Y., and Chen, X. (2001). "Microstructure and mechanical properties of austempered high silicon cast steel." *Materials Science and Engineering*, 2001, A 308, pp 277–282.
17. Jose, A.D.C.J, Vilela, J.J., Gonzalez, B.M., and Santos, D.B. (2014). "Effect of Retained Austenite on impact toughness of Multi-Phase Bainitic-Martensitic Steel.", *Advanced Materials Research*, May 2014,Volume 922, pp 298-303.
18. Prabhukumar Sellamuthu, D. G Harris Samuel, D. Dinakaran, V. P. Premkumar, Zushu Li, Sridhar Seetharaman (2018), " Austempered Ductile Iron (ADI): Influence of Austempering Temperature on Microstructure, Mechanical and Wear Properties and Energy Consumption" *Metals* 8(1), Jan 2018, pp 1-12.
19. Akor, T Tuleun , L,T (2014) "Effect of Austempering time on the Mechanical Properties of Ductile Iron, Austempered in Rubber Seed Oil", *www.ijerd.com*, August 2014, Volume 10, Issue 8, pp 31-34.
20. Amir Abedi , Pirooz Marashi, K.Sohrabi, M.Marvastian (2011) " The Effect Of Heat Treatment Parameters on Microstructure and Toughness of Austempered Ductile Iron (ADI) " *Advanced Materials Research*, June 2011, Volume 264-265, pp 409-414.