Enhancement of ductility and strength in 410 stainless steel through cyclic heat treatment

Manoj Samson R, Harshavardhana N, Nirmal R and Ranjith R
Department of Mechanical Engineering, SRM Institute of Science and Technology, Kattankulathur, Chennai, Tamilnadu, India
E-mail: manojsar@srmist.edu.in

Abstract. The 410 stainless steel alloy is used to manufacture small components like nuts and bolts and also larger components like gas turbines. The work done here focuses on tempering the 410 stainless steel to increase the ductility and strength. Heat treatment is generally done in order to get the desired mechanical properties by heating the material above its recrystallization temperature and cooling it back. The material is subjected to annealing, normalizing, quenching and tempering process in this experiment. On conducting mechanical test and metallurgical observation on the heat treated sample, we could observe change in material properties like wear characteristics, hardness, tensile and impact strength of the specimen when compared to the as-received material. And on studying the obtained results of the specimens after each heat treatment process, we can understand how each process affects the properties of the material. On quenching, the sample becomes more brittle and it can’t be used to most applications, hence tempering is done to add toughness to the material.

1. Introduction
The material used in this work for heat treatment is 410 stainless steel alloy specimen to improve the mechanical properties [1]. The different heat treatment techniques include normalizing, annealing, quenching and tempering. The temperature, to which the specimen is heated, should be more than half of the recrystallization temperature. The temperature taken was 900ºC and a soaking period of one hour. The works aims to increase the ductility and strength of the material through heat treatment process. It results in altering microstructure of the sample which can be observed under optical microscope. ASTM E8 standard was followed to perform the tensile test in the heat treated samples of dimension 75X12.5mm. And sample for impact test has a dimension of 75mmX10mmX10mm with 2 mm deep groove cut at a distance of 25mm from one end of the sample. The pin-on test specimen has a dimension of 30X10mm (diameter). Here, we have used XRD to observe the residual stress [6&7]. And UTM was used to perform the tensile test on the specimens. In order to meet the objective of this work, the samples are quenched to increase the strength and hardness thereby making the sample more brittle which is not suitable for most of the applications, hence tempering is done to reduce the brittleness and simultaneously increasing the toughness of the material. [2-3]. 410 Stainless Steel is composed of carbon, manganese, phosphorous, sulphur, silicon, and chromium. The presence of chromium constitutes the corrosion resistant properties of stainless steel. The compositions of 410 stainless steel are given in Table 1.
Table 1. Composition of 410 Stainless Steel.

| Material   | Carbon (C) | Manganese (Mn) | Phosphorous (P) | Sulphur (S) | Silicon (Si) | Chromium (Cr) |
|------------|------------|----------------|-----------------|-------------|--------------|---------------|
| Wt.%       | 0.117      | 0.720          | 0.027           | 0.026       | 0.400        | 12.77         |

Following are the literature survey done. Rakesh Rajan et al [1] carried out the pin-on test on 410 stainless steel. The wear coefficients for the different conditions were calculated using the Archard formula. Scanning Electron Microscope (SEM) analysis was done on the worn out specimens. The disc used was manufactured from EN24 steel. Zou et al [2] performed tempering on 00Cr13Ni4Mo super martensitic stainless steel. The specimens were tempered at 520°C, 560°C, 600°C, 640°C, 680°C and 720°C for a soaking period of 3 hours. The microstructures of these tempered specimens were then obtained and compared. It was found that as the tempering temperature goes higher, the retained austenite content increases gradually and at 600°C it attains the maximum value. I.I.Ahmed et al [3] investigated surface residual stress of martensitic stainless steels using X-Ray Diffraction. The sin²Ψ method of X-ray diffraction is used to observe the residual stress. Compressive residual and tensile stresses in the parts of HAZ and in the parent material respectively were indicated by the residual stress levels. Qin et al [4] studied changes in mechanical properties on tempering of 00Cr16Ni5Mo stainless steel. The temperatures to which the specimens were heated to are 525, 550, 575, 600 and 625°C. It was concluded that on lowering the tempering temperatures, the impact energy is less. Yuan et al [5] observed martensite and retained austenite phase in the microstructure of tempered 0Cr16Ni5Mo. Based on the literature survey’s, very limited work was carried out in the 410 Stainless steel. Hence an attempt has been made to in this material to increase its strength and ductility through the heat treatment processes.

2. Experimental work

2.1. Optical Emission Spectroscopy

The optical emission spectroscopy (OES) is done in order to find the composition of the constituents of the material, in this case, 410 stainless steel. In this process, the atoms are vaporized and excited to higher energy level by introducing a spark between the electrode and the specimen using the electrical energy.

2.2. Heat Treatment Method

2.2.1. Normalizing process. In normalizing the material heated above the recrystallization temperature of the material and soaked inside the furnace for particular period and it is allowed to cool in the ambient temperature. In our experiment, the sample is taken above 900°C, soaked in furnace for 90mins and allowed to cool in ambient temperature which leads to the formation of fine grains in the specimen.

2.2.2. Annealing process. Annealing and normalizing process doesn’t have much differentiation in their process. Instead of cooling the samples in the ambient temperature, the samples are cooled in the furnace itself. The temperature and soaking period is same as that of the normalizing process. A coarse grain structure is formed when the specimens are annealed. This decreases the strength of the material [14].

2.2.3. Quenching process. After annealing the samples are quenched in either water, oil or brine solution to increase the hardness of the material. In this experiment the specimens are rapidly quenched in the SAE 40 oil. The time taken for the specimen to be immersed in the oil from the furnace should be less than 2 seconds [16]. It is found that the quenched specimen is the strongest and
the specimen with the least hardness among the tested specimens. This is because the cooling rate is faster in quenching than the other treatment methods.

2.2.4. Tempering process. As explained earlier, when the material is quenched, the material becomes hard and brittle due to the formation of fine grains as in martensitic structure, which is not desired in most of the application. So, tempering was done for three different temperatures as 400, 500 and 600°C. The Tempering is a controlled heat treatment processes usually performed on ferrous materials after quenching process. The ferrous material is heated below austenitic temperature (typical in the range of 910°C) for the certain period of time and allowing it to cool in still air. This results increase in ductility, toughness with the slight reduction in hardness, strength and internal stresses of the materials compared to the quenched sample. This Tempering process is extensively used in steel industries for recovering the ductility and toughness of the steel. In our case, the tempered samples where then soaked in furnace for 90mins and cooled at ambient temperature.

The microstructure of the sample was analysed after paper polishing with 400, 800, 1200 and 2500 grade paper followed by diamond polishing with 1μm diamond paste. The etchant used for optical microstructure is nital with 10ml nitric acid and 100ml distilled water. The specimen (a) untreated, (b) annealed, (c) normalized, (d) quenched and Tempered to (e) 400°C, (f) 500°C, (g) 600°C are denoted as sample A,B,C,D,E,F and G respectively. The tensile test was performed on the dog bone sample as per ASTM E8 standard in the strain rate of 10^{-3} mm/min. The Hardness measurements were performed on the universal hardness tester at the load of 150kgf for the time period of 10 seconds.

3. Results and discussions

3.1. Microstructural Analysis

Figure 1 represents the microstructure of the material where no heat treatment is done on it. Sample A which is untreated, shows the elongated grains with numerous microstructural defects. This sample has received in the deformed condition and the thermo mechanical history of this sample A was unknown.
The microstructure of annealed sample heated to 900°C is shown in figure 2. The microstructure of the Sample B shows coarse equiaxed grains. The average grains size was found to be 25µm. The quenched sample is tempered at 400, 500 and 600°C followed by air cooling. Figure 5, 6 and 7 shows the microstructure of quenched sample followed by tempered at 400, 500 and 600°C respectively and followed by cooled in air. During normalizing, the formation of coarse grains occurs as shown in figure 3. Some defects also appear in the sample. The grains are laminar with the aspect ratio of width to length is 20/100. The sample D is heat treated to 900°C followed by quenching in SAE 40 oil medium. Subsequently, the sample is tempered at 400°C and cooled to ambient temperature. The microstructure shows lamellar structure with lot of defects. The defects are generated during quenching process as in figure 4. The defects during quenching persist even after tempering at 400, 500 and 600°C. Laminar grains with lot of defect are seen in figure 5- 7.

3.2. Tensile Test

Tensile test was performed according to ASTM E8 standard in the UTM machine to find the tensile strength, peak load, maximum displacement and elongation percentage of the samples. Figure 8 shows sample having a bone structure of dimension 70mmX12.5mm (diameter) as in. The tensile test sample after breakage is shown in figure 9. The breakage of the tensile test sample happens at the middle region of the sample and not at the ends which shows the prefect failure of the sample.

Table 2. Tensile strength, Yield stress and load at peak values for the specimen undergone various heat treatment process.

| Specimen | Tensile strength (N/mm²) | Yield strength (N/mm²) |
|----------|-------------------------|-----------------------|
| A        | 568.707                 | 366.695               |
| B        | 598.523                 | 310.127               |
| C        | 1047.819                | 921.249               |
| D        | 1176.282                | 938.595               |
| E        | 804.158                 | 635.334               |
| F        | 1031.389                | 822.293               |
| G        | 986.697                 | 788.184               |

Table 2 shows the tensile test and yield strength data for various specimens. By performing quenching in SAE oil we get high tensile strength and yield strength. Moreover, it is evident from figure 10 that the yield and ultimate stress of the quenched specimen D is found to be high and the annealed sample B is found to be low. The as received sample A also shows similar tensile strength as annealed sample B which indicated the as received sample in the defect free conditions. But when tempering is done on the specimen, there is a fall in both yield and ultimate stress (refer E, F, and G) as compared to the quenched sample D.
3.3. Hardness Test

Rockwell hardness testing machine as shown in the figure 11 was used for measuring the hardness value of the samples. The sample is placed in the holder. Diamond cone indenter with a load of 150kgf and for a dwell period of 10seconds was used for the stainless steel samples. In order to get the accurate results, the hardness is measured in 4 places in a particular sample and the average value is taken and noted down from the digital display that is shown in figure 12.

Figure 13 shows that the hardness plot in which of the quenched specimen D is found to be high hardness and the annealed sample B is found to have low hardness. The as received sample A also shows similar tensile strength as annealed sample.
3.4. Impact Test
Impact test was done to measure the toughness and the impact strength of the heat treated samples. The test was conducted in AIT 300N Izod impact testing machine manufactured by FASNE Pvt Ltd as shown in figure 14(b). Sample for impact test has a dimension of 75mmX10mmX10mm with 2mm deep groove cut at a distance of 25mm from one end of the sample as shown in figure 14(a). The pendulum of the impact testing machine can produce maximum impact energy of 168J and weighs about 250kg. The test is done by taking the pendulum to maximum height and released, so that it hits the specimen with high impact energy that is placed in the sample holder thereby causing a fracture to the specimen. Dial indicator is use to indicate the energy absorbed by the samples. The impact value is found by finding the ratio of energy absorbed to the C.S.A of the specimen. For accurate results the procedure is repeated thrice and the average is value is tabulated as in Table 3. The normalized sample shows high impact load than other heat treated samples that were tested.

![Figure 13. Hardness plot of the treated samples.](image)

![Figure 14. a) Izod specimen dimension. b) Izod Impact Testing Machine. c) Specimens after Impact Test.](image)
Table 3. Impact energy of specimens

| Specimen | Impact energy (joules) |
|----------|------------------------|
| A        | 118                    |
| B        | 76                     |
| C        | 152                    |
| D        | 24                     |
| E        | 40                     |
| F        | 38                     |
| G        | 30                     |

3.5. Pin-On Test

The pin-on test is performed in pin-On disk apparatus figure 16 to find the wear rate of the material. To conduct the test, the G99 ASTM Standard Pin-On test apparatus was used. An EN31 disc of diameter 100mm and 7mm thickness was used. The software used to translate the data onto the computer was WINDUCOM 2008 figure 15. A load of 3kgf was applied with a sliding velocity of 100RPM. The total sliding time was fixed at 40 minutes and the track diameter was set to 60mm. Also, the surface roughness and weight of the specimens were measured before and after the pin-on test[9].

![Figure 15. WINDUCOM 2008.](image1)

![Figure 16. Pin-On Disk Apparatus.](image2)

3.5.1. Surface Roughness Test. The surface roughness is the presence of micro irregularities in the real surface which deviates from the ideal surface. Surfcom 1400G Surface Roughness Tester as in figure 17 was used for surface roughness measurement of heat treated samples. The value obtained from results are plotted as in figure 18.

![Figure 17. Surface Roughness Tester.](image3)
3.5.2. Weight of the specimens. In order to calculate the wear rate of the specimens after conducting the pin-on test, the weight of the specimens is required. The weights before and after the test are shown in figure 19.

3.5.3. Wear Rate Of Specimens. Wear rate of the specimens were calculated using the formula:-

\[
\text{Wear Rate} = \frac{\text{Original Wt. of Specimen} - \text{Wt. of Specimen after Pin-On Test}}{\text{Density of the Specimen} \times \text{Time taken}}
\]

Table 4. Coefficient of Friction and Wear Rate of Specimens.

| Specimen | Coefficient of friction | Rate of wear  |
|----------|-------------------------|--------------|
| A        | 0.273                   | 6.41 x 10^{-6} |
| B        | 0.607                   | 1.2 x 10^{-4}  |
| C        | 0.582                   | 2.1 x 10^{-4}  |
| D        | 0.312                   | 9.8 x 10^{-5}  |
| E        | 0.504                   | 6.2 x 10^{-4}  |
| F        | 0.318                   | 6.3 x 10^{-4}  |
| G        | 0.340                   | 6.6 x 10^{-4}  |

3.6. X-Ray Diffraction

X-Ray diffraction was done on all specimens to know more about the intermetallic compounds which are formed during various heat treatment methods. All the sample shows the characteristic peak of BCC. All the samples show the peaks at 44, 65, and 82.5° which represent the presence of BCC structure only and there is no other phases present.

Table 5. XRD Analysis results.

| Specimen | Pos. [°2Th.] | Height [cts] | FWHM Left [°2Th.] | d-spacing [Å] | Rel. Int. [%] |
|----------|--------------|--------------|-------------------|---------------|---------------|
| A        | 45.5243      | 93707.31     | 0.5077            | 1.9925        | 100.00        |
|          | 65.5216      | 6708.51      | 0.5077            | 1.4189        | 7.16          |
|          | 83.0434      | 14530.37     | 0.6192            | 1.1620        | 15.51         |
|          | 45.0978      | 106410.40    | 0.4231            | 2.0104        | 100.00        |
| B        | 65.2483      | 9816.32      | 0.4231            | 1.4299        | 9.22          |
|  |  |  |  |  |
|---|---|---|---|---|
| 82.4993 | 23311.78 | 0.5160 | 1.1682 | 21.91 |
| 44.5829 | 131597.30 | 0.3385 | 2.0324 | 100.00 |
| 64.7657 | 10097.13 | 0.4231 | 1.4394 | 7.67 |
| 82.0741 | 20036.65 | 0.5160 | 1.1732 | 15.23 |
| 44.3573 | 71780.33 | 0.4231 | 2.0422 | 100.00 |
| 64.8164 | 3188.58 | 0.5077 | 1.4384 | 4.44 |
| 82.0535 | 10376.80 | 0.7224 | 1.1735 | 14.46 |
| 44.8368 | 77936.30 | 0.3385 | 2.0215 | 100.00 |
| 65.0694 | 4444.61 | 0.5924 | 1.4334 | 5.70 |
| 82.4271 | 10511.74 | 0.6192 | 1.1691 | 13.49 |
| 45.5385 | 55506.73 | 0.3808 | 1.9919 | 100.00 |
| 65.8452 | 2607.05 | 0.3385 | 1.4184 | 4.70 |
| 82.9553 | 7305.95 | 0.3096 | 1.1630 | 13.16 |
| 44.4858 | 56024.23 | 0.5924 | 2.0366 | 100.00 |
| 64.8786 | 3317.13 | 0.5924 | 1.4372 | 5.92 |
| 82.1482 | 9390.30 | 0.6192 | 1.1723 | 16.76 |

**Figure 20.** XRD of Untreated specimen.

**Figure 21.** XRD of Annealed specimen.

**Figure 22.** XRD of Normalized specimen.

**Figure 23.** XRD of Quenched specimen.

**Figure 24.** XRD of 400°C Tempered Specimen.

**Figure 25.** XRD of 500°C Tempered Specimen.
4. Conclusion

Annealing, normalizing, quenching and tempering heat treatment process of 410 stainless steel are compared for its tensile strength, hardness, impact strength and wear rate in this work.

- On comparing the tensile test results, the quenched specimen has highest tensile strength of $1176.28\text{N/mm}^2$ because of the formation of fine grains and internal stresses and annealed specimen with a lowest tensile strength of $568.70\text{N/mm}^2$ because of the formation of coarse recrystallized grain structure at higher temperature. There is 31.63% decrease in tensile strength for $400°C$ tempered specimen followed by 12.31% and 16.11% decrease in tensile strength for $500°C$ and $600°C$ tempered specimen respectively. This reflects the reduction of internal stresses during tempering.

- Hardness test reveals similar trends as tensile test value. Higher hardness value is achieved in quenched specimen because of the rapid cooling rate and low hardness value in annealed sample due to its coarse grain structure. On comparing the quenched and tempered specimen there is a fall of 60.28%, 45.42% and 27.42% for sample E, F and G respectively.

- Impact test results shows that the normalized sample has better impact energy followed by the untreated one, while the sample D has the least value. There is 40%, 36.84% and 20% increase in impact strength for sample E, F and G respectively as compared to quenched sample which follows typical tempering conditions.

- It is also found that the highest wear rate is obtained from the sample tempered at $600°C$ and the as-received sample has the lowest wear rate. On considering the quenched and tempered samples there is increase in wear rate by 84.19%, 84.44% and 85.15% for sample E, F and G respectively.

- XRD graphs reveal the formation BCC structure for all heat treatment conditions. This confirms that there is formation of BCC structure only and no intermediate compounds and martensitic formed during the heat treatment process.

5. References

[1] Rakesh. K. Rajan 2014 Sliding Friction and Wear Characteristics of Grade 410 Martensitic Stainless Steel. *Applied Mechanics and Materials* **592** 1346-1351

[2] Zou De-Ning, Ying H, Zhang W and Fang X D 2015 Influence of Tempering Process on Mechanical Properties of 00Cr13Ni4Mo Super martensitic Stainless Steel *Journal of Iron and Steel Research, International* **17** 50-54

[3] I.I. Ahmed 2016 Investigation of surface residual stress profile on martensitic stainless steel weldment with X-ray diffraction *Journal of King Saud University - Engineering Sciences* **30** 183-187
[4] B. Qin and Sun Q S 2017 Effect of tempering temperatures on properties of 00Cr16Ni5Mo Stainless steel Materials Characterization 59 1096-1100
[5] Yuan, Wu-hua, Xue-hui Gong, Yong-qing Sun, and Jian-xiong Liang 2016 Microstructure evolution and precipitation behavior of 0Cr16Ni5Mo martensitic stainless steel during tempering process Journal of Iron and Steel Research, International 23 401-408
[6] Pandey C and Mahapatra M M 2016 Effect of groove design and post-weld heat treatment on microstructure and mechanical properties of P91 steel weld Journal of Materials Engineering and Performance 25 2761-2777
[7] Gao G, An B, Zhang H, Guo H, Gui X and Bai B 2017 Concurrent enhancement of ductility and toughness in an ultrahigh strength lean alloy steel treated by bainite-based quenching-partitioning-tempering process Materials Science and Engineering: A 702 104-112
[8] Saha, D C, Biro E, Gerlich A P and Zhou Y 2016 Effects of tempering mode on the structural changes of martensite Materials Science and Engineering: A 67 467-475
[9] Shrestha, Triratna, Sultan F, Alsaegabi, Indrajit Charit, Gabriel P. Potirmiche, and Michael V. Glazoff 2015 Effect of heat treatment on microstructure and hardness of Grade 91 steel Metals 5, 1 131-149
[10] Mitra, Abhishek, Siva Prasad N and Janaki Ram GD 2016 Estimation of residual stresses in an 800 mm thick steel submerged arc weldment Journal of Materials Processing Technology 229 181-190
[11] Salimianrizi A, Foroozmehr E, Badrossamay M, Farrokhpour H 2016 Effect of laser shock peening on surface properties and residual stress of Al6061-T6. "Optics and Lasers in Engineering 77 112-117
[12] Okonkwo P C, Kelly G., Rolfe B F and Pereira M P 2016 The effect of sliding speed on the wear of steel–tool steel pairs Tribology International 97 218-227
[13] Yadollahi, Aref, NimaShamsaei, Scott M. Thompson, AlaElwany and LinkanBian 2017 Effects of building orientation and heat treatment on fatigue behavior of selective laser melted 17-4 PH stainless steel International Journal of Fatigue 94 218-235
[14] Pandey C, Giri A and Mahapatra MM 2016 Effect of normalizing temperature on microstructural stability and mechanical properties of creep strength enhanced ferritic P91 steel Materials Science and Engineering: A. 657 173-84
[15] Pandey C, Mahapatra MM, Kumar P and Saini N 2018 Effect of strain rate and notch geometry on tensile properties and fracture mechanism of creep strength enhanced ferritic P91 steel Journal of Nuclear Materials 498 176-86
[16] Wang, Zhuqing, Todd A. Palmer, and Allison M. Beese 2016 Effect of processing parameters on microstructure and tensile properties of austenitic stainless steel 304L made by directed energy deposition additive manufacturing ActaMaterialia 110 226-235
[17] Odnobokova M, Belyakov A, Enikeev N, Molodov D A and Kaibyshev R 2017 Annealing behavior of a 304L stainless steel processed by large strain cold and warm rolling Materials Science and Engineering: A 689 370-383
[18] Zheng Z J, Liu J W and Gao Y 2017 Achieving high strength and high ductility in 304 stainless steel through bi-modal microstructure prepared by post-ECAP annealing Materials Science and Engineering: A. 680 426-432
[19] Seo EJ, Cho L, Estrin Y, De Cooman BC 2016 Microstructure-mechanical properties relationships for quenching and partitioning (Q&P) processed steel ActaMaterialia 113 124-139
[20] Zhao ZZ, Liang JH, Zhao AM, Liang JT, Tang D, Gao YP 2017 Effects of the austenitizing temperature on the mechanical properties of cold-rolled medium-Mn steel system Journal of Alloys and Compounds 691 51-59
[21] Ziętała M, Durejkó T, Polański M, Kunce I, Płociński T, Zieliński W, Łazińska M, Stępniewski W, Czyżko T, Kurzydłowski KJ, Bojar Z 2016 The microstructure, mechanical properties
and corrosion resistance of 316 L stainless steel fabricated using laser engineered net shaping *Materials Science and Engineering: A* **677** 1-10

[22] Zambrano OA, Aguilar Y, Valdés J, Rodríguez SA, Coronado JJ 2016 Effect of normal load on abrasive wear resistance and wear micromechanisms in FeMnAlC alloy and other austenitic steels *Wear* **348** 61-68