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Influence of Heat Treatment Parameters on Structure and Mechanical Properties of an HSLA-100 Steel

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Summary(Abstract) :

An HSLA-100 steel received from the US Naval Research Laboratory has been characterised. The effects of heat treatment parameters such as austenitisation time and temperature, tempering time and temperature on mechanical properties have been studied. The microstructures resulted by different heat treatment conditions have been correlated with mechanical properties through SEM and TEM studies. Quantitative relationships have been developed between mechanical properties and the operational variables within a narrow range of variation of the variables by statistical design of experiments. A quantitative relationship has also been developed for the same for a wider experimental region through curve fitting technique. The best combination of strength and low-temperature toughness was obtained in the region of 700 °C tempering temperature and 0.3-0.4 hours tempering time.

Keywords :- HSLA-100 steel; Mechanical properties; Design of Experiments; Heat treatment.

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Introduction

Copper bearing HSLA 100 Steel developed by US Navy is used in Navy vessels since 1980 [1-3]. Several studies were made to correlate structure during tempering / ageing of copper bearing steels with mechanical properties, especially to understand the role of copper in these steels [4-7]. The alloy chemistry is so chosen in order to enhance properties by thermomechanical processing (TMP) coupled with judicious heat treatment parameters. While copper plays an important role in these steels, other alloying elements such as Mo, Cr, and Ni enhance hardenability of these steels. Addition of Nb enables the steel to be graded as HSLA and makes it responsive towards TMP [8]. The complex interactions of the processing variables with the alloy chemistry of the steel necessitate thorough investigation to understand the physical metallurgy of these steels.

In the present study attempts have been made to quantify the effects of the processing parameters on mechanical properties (hardness, yield strength, tensile strength and low-temperature impact property) of the steel. For the purpose of quantification statistical design of experiments [9-12] has been used where the operational variables are varied in a definite manner. The regression equation is obtained for the dependent variable Y, called the response (in this case a particular mechanical property), with the process variables X_1 and X_2 . The equation is generally of the form

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_{12} X_1 X_2 \quad \ldots\ldots \quad \ldots\ldots \quad \ldots\ldots(1)$$

where b_0, b_1, b_2 etc. are coefficients representing the effect of each factor X_1 and X_2, and b_{12} is the coefficient representing the effect of interaction between the process variables X_1 and X_2. The presence of the interaction coefficient makes the equation non-linear and suggests a curved response surface. However, if the ranges of variation of the process variables are sufficiently small, the value of the interaction coefficient becomes small and insignificant, and the response surface becomes almost planer in nature.

In the present investigation, regression equations have been developed for the mechanical properties and process variables. These equations are helpful to the users in determining the processing conditions for achieving a desired combination of properties. The equations further help in locating the zone for process variables where optimum combination of strength and toughness prevails.

Experimental

Steel received from US Naval Research Laboratory (see **table 1** for composition) was characterised with respect to mechanical properties (**table 2**), inclusion content, grain size, and microstructure (**figure 1**) in as-received condition. The Ac_1 and Ac_3 temperatures were determined using DTA/TMA apparatus. These were also calculated using empirical formulae [13-14] (**Table 3**). These temperatures were determined in order to decide the austenitising temperature for hardening as well as thermomechanical processing. In order to see the qualitative and quantitative effect of heat treatment parameters on mechanical properties, samples were austenitised at three different temperatures, i.e., 900, 950 and 1000 °C followed by quenching in water/cooling in air with subsequent tempering between 450-750 °C at an interval of 50 °C. The temperatures were maintained within an accuracy of ±3 °C.

Table 1. Chemical composition of the steel

C	Mn	P	S	Si	Cu	Ni	Cr	Mo	Al	Cb
0.04	0.86	0.004	0.002	0.27	1.58	3.55	0.57	0.60	0.032	0.030

Table 2. Mechanical Properties of the as-received material

YS, MPa	TS, MPa	YS/TS	%EL on 25mm GL	*n* value
746.2	804.8	0.93	28	0.04

Table 3. Ac_1 and Ac_3 values (Calculated and Experimental)

Ac_1 ,°C		Ac_3 ,°C	
Calculated	Experimental	Calculated	Experimental
671	660	878	845

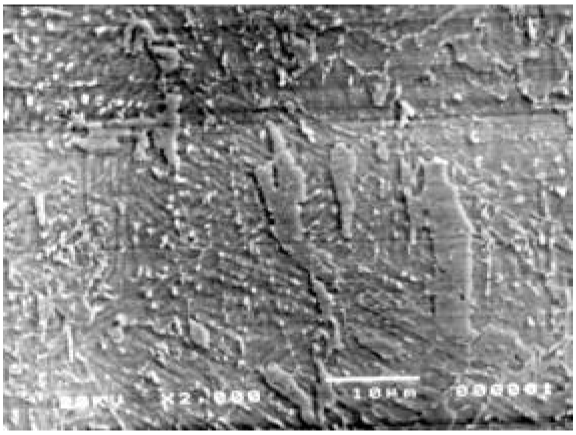


Fig. 1 - SEM of steel in as-received condition

For all the specimens microstructural investigations were carried out by optical microscopy, TEM and SEM studies. Vickers hardness tests, tensile tests (using Instron 1195 following ASTM E8-78 procedure) and Charpy V-notch impact tests (at -50 °C) were also carried out. Fracture surfaces of broken Charpy samples were examined in SEM.

Results and discussions

Effect of tempering time and temperature on the microstructure and mechanical properties of the steel. The hardness vs. tempering temperature curves for the steel austenitised at 900, 950, 1000 °C followed by quenching in water are shown in

figure 2. Superimposed on the same plots, hardness vs. tempering temperature curves for air-cooled samples austenitised at 900 and 950 °C are shown. The hardness values of the steel in as quenched condition are found to decrease with increasing austenitising temperature. This may be attributed to increase in the amount of retained austenite in quenched condition as a result of increasing austenitising temperature. This feature was less prominent in air-cooled samples. The microstructures reveal acicular structure for water quenched samples (**figure3**) whereas non-polygonal (Bainite) structure for those of air-cooled samples (**figure4**). The microstructure of as-received steel was found to be similar to the microstructure of the air-cooled steel (comparison of **figure 1** with **figure 4**).

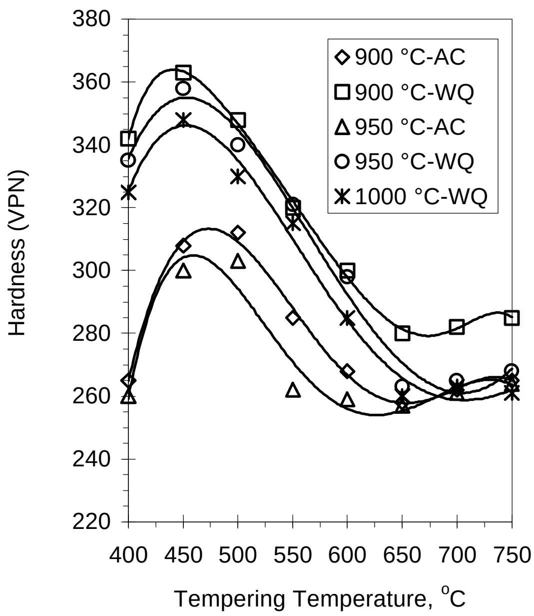


Fig. 2 - Tempering Temperature vs. Hardness curve

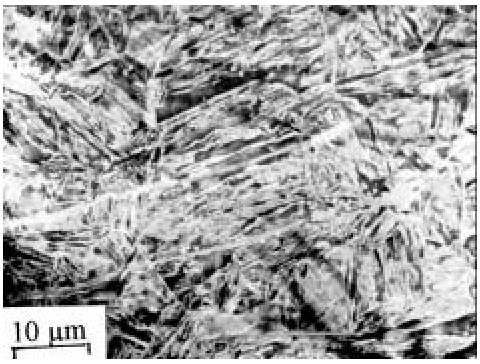


Fig. 3 - SEM of steel in WQ condition

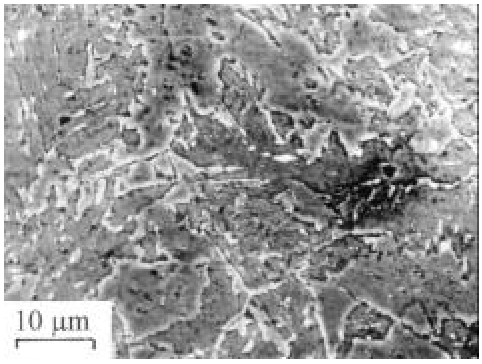


Fig.4 - SEM of steel in air-cooled condition

The effect of tempering temperatures on hardness values of the steel at each austenisation temperature, i.e., 900 / 950 / 1000°C, showed similar effect, i.e., peaks were observed in each hardness vs. tempering temperature curve. The first peak values were observed at 450°C for water quenched and tempered steel in comparison to peak value between 450°C - 480°C for air-cooled and tempered steel. Thus the peak shifted slightly to the right for air-cooled steel. The shift of peak for air-cooled samples is due to the retardation in the kinetics of precipitation in air-cooled samples [15]. Mujahid *et. al.* [16,17] showed following structural changes occur in stages during tempering (between 400-750 °C) in these types of steels:-

←--- Stage I --------- → ←---- Stage II -----→ ←-- Stage III ---→
450 °C 500 °C 500-650 °C
Cu rich α'→ Cu Cluster → BCC Cu rich→ Cu(FCC, Spherical)→ Rod shaped ppt →
Cluster (coherent)

←--- Stage IV ---→
650-700 °C
→ γ changing to
bainite / martensite on fast cooling

The occurrences of all the stages in the above sequences are functions of :
a) Solute Content in the Matrix, b) Tempering Temperature, c) Cooling Rate.

The strengthening of the steel is due to combination of i) Formation of Low carbon martensite / bainite / acicular ferrite, with high dislocation densities, ii) Precipitation during tempering, iii) Finer grain size, iv) Solid solution.

Weakening of the steel at higher tempering temperature is due to changes occurring in the matrix during tempering, viz., a) Recovery / Recrystallisation, b) Coarsening of precipitates etc.

Combination of all the above processes during tempering at different regions of tempering temperature explains the hardness vs. tempering temperature curve, shown in **figure 2**. The slight delay in tempering reaction for air cooled samples showing alteration of kinetics of reaction may be attributed to the difference in the initial microstructure of air cooled samples in comparison to that of water quenched samples.

Microstructural Characterisation of heat treated samples through TEM studies. The TEM studies were carried out for the WQ and tempered steel. The WQ sample showed lath structure (vide figures 5(a) and (b)). At higher magnification, fine precipitates were observed within the lath, which may be due to undissolved Niobium-carbonitrides, figure 5(b). The laths were highly dislocated. There were some small bright regions which may be due to austenite as reported by Mujahid *et. al.* [16,17].

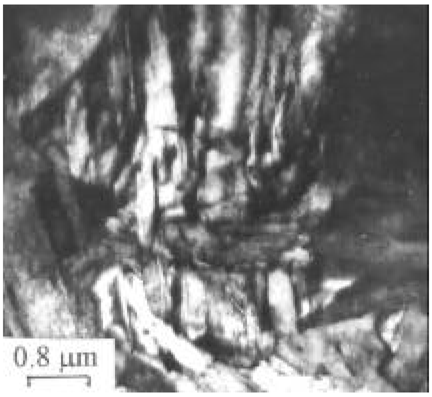


Fig. 5(a) - TEM of WQ steel showing lath structure

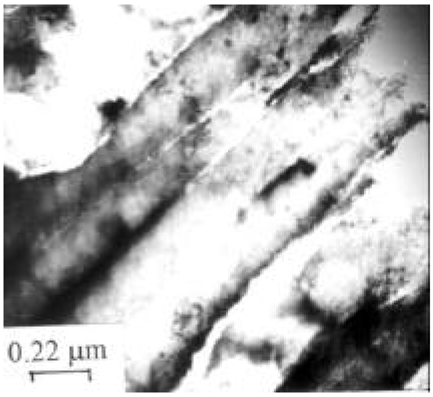


Fig. 5(b) - TEM of WQ steel showing precipitates within the laths. The white regions near lath boundaries are reported to be austenite.

Tempering steel at 450 °C shows persistence of lath structure (**figures 6(a)** and **(b)**). At this stage the microstructure showed blurred regions possibly indicating coherency strains due to Cu precipitates (**figure 6b**). The occurrence of Cu clusters of fine precipitates along with dislocations and coherency strains in the lath martensite without any recovery explains the highest hardness of the steel at this temperature (**figure 2**).

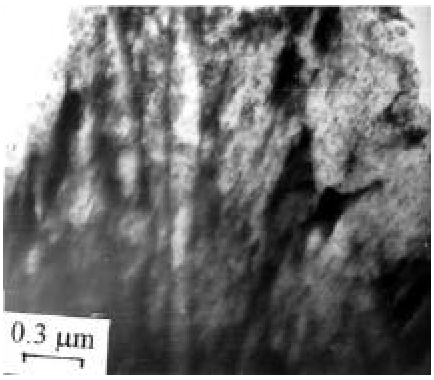


Fig. 6(a) - TEM of steel tempered at 450 °C (1 hour). shows persistence of lath structure.

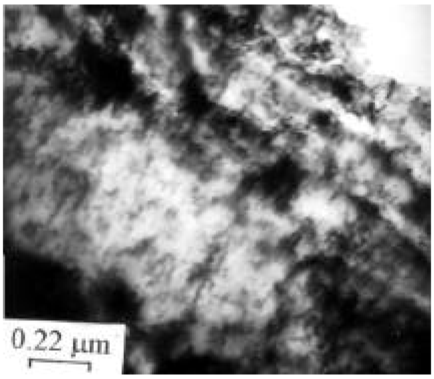


Fig. 6(b) - TEM of steel tempered at 450 °C (1 hour).The blurred regions indicate coherency strains due to Cu precipitates during the early stages of tempering.

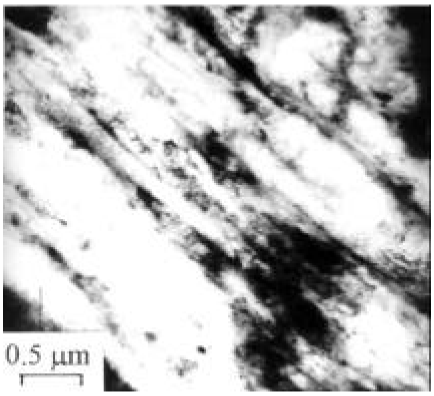


Fig. 7(a) - TEM of steel tempered at 600 °C (1 hour) shows cleaner ferrite with few precipitates.

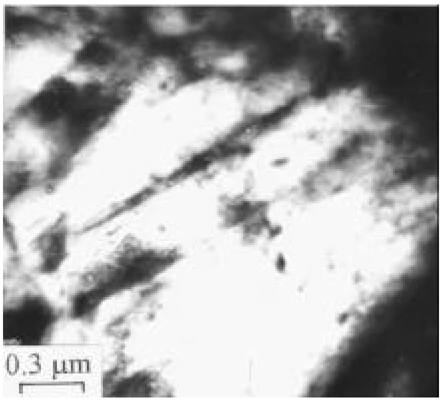
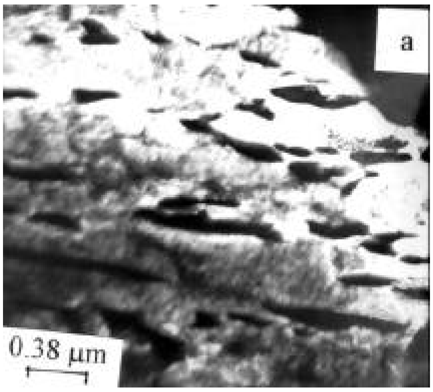


Fig. 7(b) - TEM of steel tempered at 600 °C (1 hour) shows occurrence of recovery in lath structure with distinct precipitates in the matrix.

The specimen aged at 600 °C shows clean ferrite with a few precipitates (**figures 7(a)** and **(b)**). The matrix seems to have recovered, without recrystallisation. However, no attempt was made to analyse the dark precipitates observed in the microstructure. The coarsening of the precipitates with recovery in the matrix explains the decrease in hardness due to tempering at 600 °C for one hour.



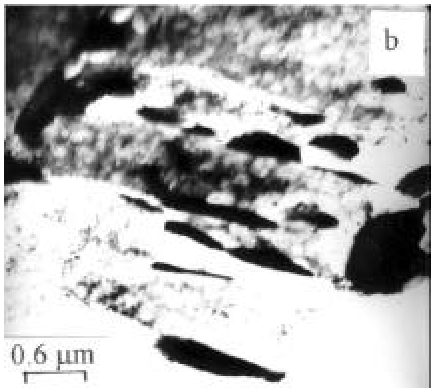


Fig. 8(a), (b) - TEM of steel tempered at 700 °C (1 hour) shows presence of new generation austenite at prior lath boundaries.

Tempering at still higher temperature, i.e., 700 °C, shows presence of new generation austenite, formed at the prior lath boundary [16-18] (dark small islands in **figures 8(a), (b)**). Some coarse precipitates (spherical / rod shaped) are observed in **figure 8(c).** The small islands at the boundaries have transformed to lath martensite from austenite (**figure 8(c)**) due to quenching of the steel after tempering at 700 °C for 1 hour, which causes the second peak in the hardness curves. Similar observations are made by other researchers [16-18].

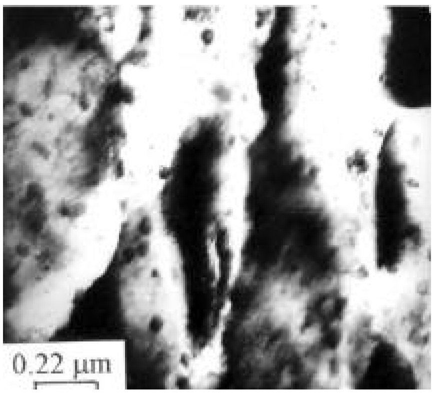


Fig. 8(c) - TEM of steel tempered at 700 °C (1 hour) shows spherical / rod shaped precipitates.

Studies on Quantitative Effect of the Process Variables on the Hardness and Charpy values. The effects of the temperature and time of ageing of samples austenitised at 950 °C, on hardness and Charpy values were quantified using statistical design of experiments [9-12]. The experiments were planned in three stages by varying temperature and time of tempering as shown in **tables 4(a), (b)** and **(c)**. The ranges of variation of tempering temperature and time for each design matrix were kept small in order to quantify the effect of variables within a narrow range where the mechanism of tempering remains the same.

Table 4(a). 2^2 Design Matrix along with response (steel austenitised at 950 °C - 1hr)

TREATMENT VARIABLES				RESPONSE
Tempering Temp.		Tempering Time		Hardness, VPN using 10kg load
Coded value X_1	Decoded form, °C	Coded value X_2	Decoded form, hr.	
+1	510	+1	8	317
-1	450	+1	8	363
+1	510	-1	4	334
-1	450	-1	4	373

Table 4(b). 2^2 Design Matrix along with response (steel austenitised at 950 °C - 1 hr)

TREATMENT VARIABLES				RESPONSE
Tempering Temp.		Tempering Time		Hardness, VPN using 10kg load
Coded value X_1	Decoded value, °C	Coded value X_2	Decoded value, hr	
+1	560	+1	8	285
-1	510	+1	8	317
+1	560	-1	4	301
-1	510	-1	4	334

Table 4(c). 2^2 Design Matrix with response (steel austenitised at 950 °C - 1 hr)

TREATMENT VARIABLES				RESPONSE
Tempering Temp.		Tempering Time		Hardness, VPN using 10kg load
Coded value X_1	Decoded value, °C	Coded value X_2	Decoded value, hr.	
+1	680	+1	8	264
-1	620	+1	8	251
+1	680	-1	4	236
-1	620	-1	4	271

Using the data from **tables 4(a), 4(b)** and **4(c),** the following regression equations were developed.

Y_H = 346.75 - 21.25 X_1 - 6.75 X_2 - 1.75 X_1 X_2 …… ….. ……(2)

where,

X_1 = (T - 480) / 30, and X_2 = (t - 6) / 2

The range of variation of tempering temperature (T) is 450-510 °C, the base level being 480 °C; and the same for tempering time is 4-8 hours, the base level being 6 hours. Hence in coded form the ranges are $-1 \leq X_1 \leq +1$ and $-1 \leq X_2 \leq +1$.

Y_H = 309.25 - 16.25 X_1 - 8.25 X_2 + 0.25 X_1 X_2 …… …… ….. (3)

where,

X_1 =(T - 535) / 25 and X_2 = (t - 6) / 2

The range of variation of tempering temperature (T) is 510-560 °C, the base level being 480 °C; and the same for tempering time is 4-8 hours, the base level being 6 hours. Hence in coded form the ranges are $-1 \leq X_1 \leq +1$ and $-1 \leq X_2 \leq +1$.

Y_H = 255.5 - 5.5 X_1 + 2X_2 + 12 X_1 X_2 …… …… …… ……. (4)

where,

X_1 =(T - 650) / 30 and X_2 = (t - 6) / 2

The range of variation of tempering temperature (T) is 620-680 °C, the base level being 480 °C; and the same for tempering time is 4-8 hours, the base level being 6 hours. Hence in coded form the ranges are $-1 \leq X_1 \leq +1$ and $-1 \leq X_2 \leq +1$.

The above equations are strictly valid in the respective ranges of variations of tempering temperature and time.

Regression eqn. (2) is formulated by using the response from the matrix of **table 4(a).** Above 480 °C the second stage of ageing process occurs where Cu clusters tend to change to Cu precipitate thereby loosing the hardness values. Some recovery of dislocations occurs. The coherency strain in the matrix is reduced. This explains the -ive coefficients attached to X_1 and X_2 of eqn. (2). The coefficient attached to X_1 (tempering temperature) is 3 times higher than the coefficient of X_2 (tempering time) indicating that the temperature influences the kinetics far more than the tempering time. The coefficient attached to $X_1 X_2$ of eqn. (2) is much less pronounced in this range.

Regression eqn. (3) is formulated by using the data of the matrix of **table 4(b).** Here the tempering temperature was varied between 510-560 °C keeping the base level at 535 °C and tempering time at 6 hour. The eqns. (2) and (3) are similar except the coefficients attached to X_1 X_2 , i.e., the combined effect of time and temperature of tempering is positive and very insignificant. The decrease of hardness values with increase in temperature above 535 °C is due to recovery of the matrix and formation of incoherent and coarsened Cu precipitates (**figures 7(a) and (b)**).

Regression eqn. (4) is formulated by using the data of the matrix of **table 4(c).** The individual effects of temperature and time of ageing have been reduced substantially compared to their effects in eqns. (2) and (3) though the combined effect of time and temperature (i.e., the coefficient attached to $X_1 X_2$ in eqn. (4)) resulted a very significant value. Thus, the positive interaction between temperature and time (i.e., coefficient of X_1 X_2) shows some distinct changes in mechanism of reaction above 650 °C. Since the Ac_1 temperature determined for this steel is above 650 °C , it is possible that austenites formed in the higher temperature range of tempering subsequently changed to Bainite on quenching after tempering the steel at the highest temperature and time used in the design matrix (**figures 8(a), (b) and (c)**). The occurrence of the second small peak in hardness vs. tempering temperature curve (**figure 2**) justifies the above statement.

Table 5 shows Charpy values (at -50 °C) for the steel quenched and tempered at 450 / 600 / 700 °C (1 hour). Charpy value was minimum at tempering temperature of 450 °C and increased substantially between 600 to 700 °C. The fracture surface of the Charpy sample aged at 450 °C for 1 hour indicates typical brittle fracture (**figure 9**). The fractrograph of broken Charpy sample tempered at 700 °C shows typical ductile fracture (**figure 10**).

Table 5. Charpy values (test temperature = -50 °C)

Treatment Condition	Impact Values, J
As-received	194
(950°C 1hr. - WQ)	115
Air Cooled	146
WQ + tempered at 450°C for 1hr.	79
WQ + tempered at 600°C for 1hr.	176
WQ + tempered at 700°C for 1hr.	230

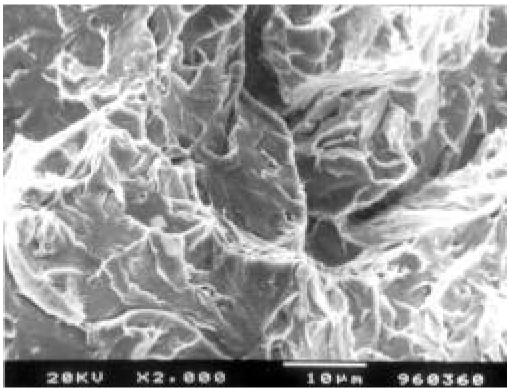


Fig. 9 - Fractograph of Charpy specimen tempered at 450 °C-1hour shows quasi-cleavage fracture (test temperature = 50 °C).

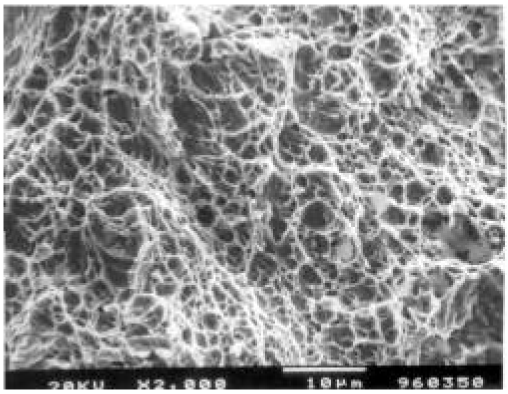


Fig. 10 - Fractograph of Charpy specimen tempered at 700 °C-1hour shows dimples on the fracture surface (test temperature = 50°C).

It is concluded from the above discussion that the impact values of the steel increased by tempering between 650-700 °C (1 hour). Similarly hardness values also increased by tempering the steel between 650-700 °C (**figure 2**). It was therefore decided to construct a design matrix around tempering temperature and time of 690 °C and 7.5 hr. respectively. **Table 6** shows treatment combinations with responses (Charpy values at -50 °C). Treating the responses obtained from various treatment combinations eqn. (5) was developed. The equation is valid within the range of variation of the variables.

$$Y_{charpy} = 126 + 15.5\, X_1 + 20\, X_2 \quad \ldots\ldots \ldots\ldots \ldots\ldots \ldots\ldots (5)$$

where $X_1 = T - 690 / 10$, $X_2 = t - 7.5 / 2.5$

T and t are natural values of temperature in °C and time of tempering in hour. Y_{charpy} is the Charpy impact value in Joule at -50 °C. X_1 and X_2 are the coded values. Natural values of temperature and time of tempering can be obtained by decoding, using equation (5).

Table 6. 2^2 Design Matrix with response

TREATMENT VARIABLES				RESPONSE
Tempering temp.		Tempering Time		Charpy value, J
Coded value X_1	Decoded value, °C	Coded value X_2	Decoded value, hr	
+1	700	+1	10	221
-1	680	+1	10	175
+1	700	-1	5	163
-1	680	-1	5	125

Eqn. (5) shows that if the tempering time and temperature are increased beyond 7.5 hour and 690 °C the Charpy value increases. Earlier the hardness value also showed a peak in the vicinity of 680-700 °C. Therefore it is expected that strength and impact value can be put at optimum level if the steels are quenched and tempered between 650-700 °C.

Considering the above results it was decided to find out the effects of temperature and time of tempering on the tensile properties of the steel in the broader range i.e., tempering temperature of 600-700 °C and tempering time of 0.33-80 hours.

Quantitative effects of heat treatment variables on the tensile properties.

Table 7 shows strength properties obtained by austenitising at 950 °C for 1 hour and tempering at different combinations of tempering temperature and time. It is observed that the steel quenched from 950°C-1hr. followed by tempering at 700°C for the time period ranging between 0.33 hrs. to 80 hrs. resulted in the reduction YS/TS ratio from 0.88 to 0.64. The *n* (strain hardening exponent) values increased to 0.24 at 12 hrs and subsequently dropped to 0.2 at 80 hrs.

Table 7. Tensile Properties of the steel after tempering at different

time and temperature combinations

Temp.,°C / Time, hr.	YS, MPa	TS, MPa	YS/TS	*n*
700 / 0.33	733.8	831.9	0.88	0.20
700 / 2	544.5	750.5	0.73	0.21
700 / 12	436.5	652.4	0.67	0.24
700 / 80	348.3	543.5	0.64	0.20
600 / 0.33	846.6	863.3	0.98	0.069
600 / 2	716.1	735.8	0.97	0.12
600 / 12	670.0	680.8	0.98	0.13
AQ	861.3	943.7	0.91	----

However, tempering at 600°C for the same period of time did not change the YS/TS ratio (0.98). The *n* values increased from 0.069 to 0.13. No attempt was made to temper the steel beyond 80hrs.

Significant observations can be made from the stress - strain diagrams. Tempering at 600°C always produced discontinuous yielding and occurrence of sharp yield point. However, tempering at 700°C resulted in continuous yielding behaviour (**figure11**), typical of the behaviour of Dual phase steel [19].

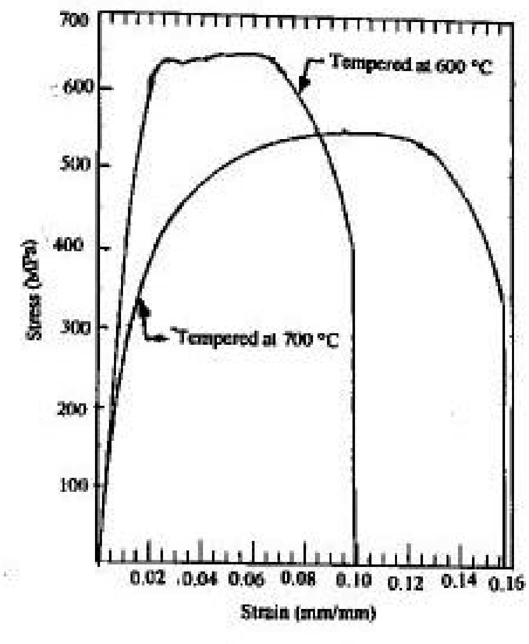


Fig. 11 - Stress-Strain curves for steel tempered at 600 °C and 700 °C

Figures 12 and 13 show yield and tensile stresses against Hollomon-Jaffe temperature normalised time parameter respectively. Fairly good agreement with this parameter is obtained for the yield stress for all tempering conditions; however, tensile stress values deviate significantly at higher temperature ranges [18]. This is because of the precipitation of austenite (**figures 8(a) and (b)**) which causes change in YS/TS ratio at higher temperature ranges.

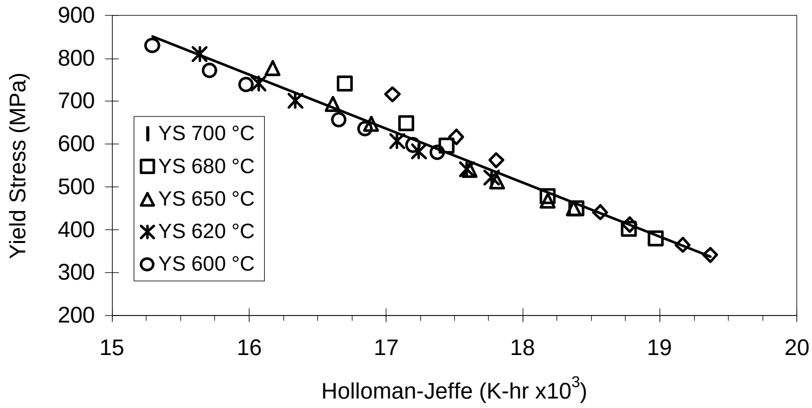


Fig. 12 - Yield stress vs. Hollomon-Jaffe parameter curve

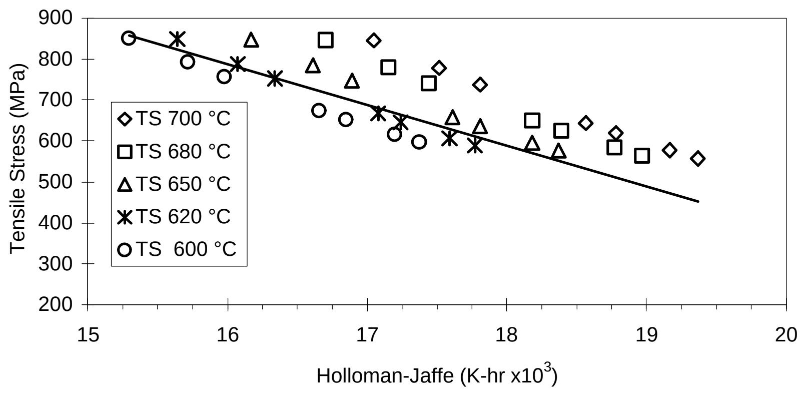


Fig. 13 - Tensile stress vs. Hollomon-Jaffe parameter curve

In order to obtain a single equation for a particular mechanical property in the entire experimental region, the following equations are developed using least square curve fitting technique.

$$YS\ (MPa) = (173.5 - 0.158\ T).\ t^{(2.8107 - 0.4496 \ln T)} \quad \ldots\ldots\ \ldots\ldots\ \ldots\ldots\ \ldots\ (6)$$

$$TS\ (MPa) = (89.3 - 0.01435\ T).\ t^{(0.4153 - 0.075 \ln T)} \quad \ldots\ldots\ \ldots\ldots\ \ldots\ldots\ \ldots\ldots\ (7)$$

where T is the tempering temperature in °C and t is the tempering time in hour. The above equations are valid in the temperature range 600-700 °C and time range 0.33-80 hours. All the above equations are helpful for deciding the heat treatment conditions to produce a desired a property in the steel.

Optimum values of Strength and Toughness. Charpy V-notch impact tests were carried out at -50°C for specimens tempered at 600°C and 700 °C for different time durations. The results are presented in **table 8.**

Table 8. -50 °C CVN Energy values of the steel at different tempering temperatures and time

Tempering temperature °C / hour	-50 °C CVN Energy, J
700 / 0.33	258
700 / 1.0	235
700 / 2.0	232
700 / 12	180
700 / 80	208
600 / 0.33	135
600 / 1.0	176
600 / 2.0	196
600 / 12	215

Figure 14 represents plots of Charpy impact values against yield stress at two different tempering temperatures, i.e., 600 °C and 700 °C. Each point on the curve

represents the properties obtained for a particular combination of tempering temperature and time. The dashed line shown in the figure represents the region where yield stress is above 685 MPa (~100 ksi) and Charpy value is more than 225 Joules.

Thus the best combination of properties is obtained in the region of tempering temperature of 700 °C and tempering time of 0.3-0.4 hours.

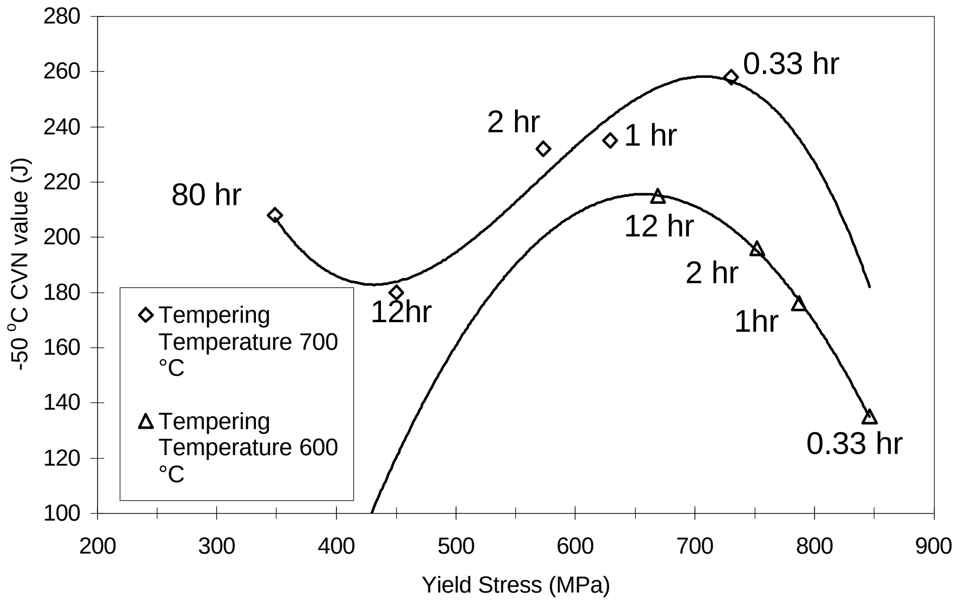


Fig. 14 - Charpy impact value vs. Yield Stress curve

Conclusions

1. The steel responds well to heat treatment, both in water-quenched condition as well as air-cooled condition.

2. On tempering at 700°C for different lengths of time, the steel showed lower YS/TS ratios in comparison with those obtained by tempering the steel at 600°C for different lengths time. \`*n*' values (strain hardening exponent) are higher (~0.20) for 700°C tempered steels in comparison with the \`*n*' values (~0.12) obtained by tempering at 600°C at all tempering times.

3. Continuous yielding was found to occur for the steel tempered at 700°C for different lengths of time. However, sharp yield points were obtained in the stress - strain diagram of the steel tempered at 600°C for different lengths of time. The stress - strain behaviour at 700°C resembles the stress strain diagram of typical Dual phase steel.

4. The quantitative effects of tempering parameters i.e. temperature and time of tempering o mechanical properties are shown in the form of regression equations. The equations are obtained by applying statistical Design of Experiments. These quantitative relations are strictly valid within the range of variation of the variables. The equations are helpful to the user in deciding the heat treatment condition that will produce the desired mechanical property in the steel.

5. The structural behaviours are fully characterised for the steel in the quenched, quenched and tempered conditions at various tempering temperatures. These structures explain stages of tempering as have been discussed in the literature by various authors [16,17,20]. The structures are also correlated with regression coefficients obtained through design of experiments.

6. In order to obtain quantitative effect in a larger range of the process variables, data were utilised to obtain numerical relations by adopting best-fit method. These equations can predict the strength values for the steel. Accuracy of the equations has been checked by comparing experiments carried out randomly.

7. The YS and TS values were plotted against Holloman-Jaffe temperature normalised parameters. The plot of Yield stress vs. Hollomon-Jaffe Parameter shows linearity within wider range of tempering temperature. However, similar plot for TS vs. Hollomon-Jaffe Parameter shows significant deviations from linearity. This is due to the formation of austenite at higher tempering temperature.

8. The best combination of YS and Charpy values at -50°C were obtained for the steel tempered at 700°C for 0.3-0.4 hours.

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