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Optimization of Mechanical Properties of a Heat-treated Cu-bearing HSLA-80 Steel

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Abstract :

HSLA-80 steel, a replacement of HY-80 steel, is widely used in Navy vessels, where the requirement is good weldability, high strength and high low-temperature toughness. However, an increase in strength usually causes a decrease in toughness. Hence there is a need for optimization of the properties. The present work attempts to optimize the mechanical properties of an HSLA-80 steel through control of heat-treatment variables. The heat treatment region, where the optimum combination of properties is likely to be obtained, is determined by carrying out single factor experiments over a wide range of tempering time-temperature combinations. Subsequently further experiments are done in this zone using statistical design of experiments. The present work involves quantification of properties by (i) classical curve fitting technique with data obtained from single factor experiments, and (ii) forming regression equations from 2^2 *factorial design of experiments. Finally optimum combination of properties of the present steel has been obtained by Grid Search technique with a constraint on yield strength. Transmission Electron Microscopy studies are done, wherever necessary, to understand and correlate the mechanical properties with the microstructures.*

Keywords : **HSLA steel; Heat-treatment; Optimization; Design of experiments; Mechanical properties.**

1. Introduction

In the past HY-series (High Yeild) steels were being used in the shipbuilding industry. A navy vessel has to withstand a complex spectrum of dynamic loadings, viz. shock due to sea waves slamming and slapping, vibration, weapon reactions, aircraft landings etc. In addition the structure may have to operate in tropical as well as arctic conditions. HY-series steels met all these requirements. The first of this series was HY-80 steel (the suffix representing yield strength in ksi). However, HY-series steels were not easily weldable. In shipbuilding industry, welding may constitute upto 20% of the total fabrication cost [1] which include welding materials, processing (pre-heating, post-weld soaking etc.), non-destructive testing (NDT) and labour cost. In order to increase productivity it was necessary to have steels that are easily weldable and at the same time possess high strength as well as good low-temperature impact toughness. HSLA steel programme grew out of this need of the navy.

HSLA-80 steel is a replacement of HY-80 grade steel. To improve weldability, the steel has a very low carbon content (≤0.06 wt%). Hence other alloying elements are added (upto 4.8 wt.%) to compensate the loss of strength due to reduced carbon content as well as to improve other mechanical properties [2]. Addition of Cu, apart from increasing weathering resistance, makes the steel responsive to heat-treatment. It helps in increasing strength by formation of fine Cu-precipitates during tempering [3]. Cu also lowers the martensite/bainite transformation temperature, and retards recovery and recrystallization of as-quenched steel [4,5]. The relative positions of HY and HSLA steels in the weldability diagram are illustrated in Fig.1 [4,5].

Since this grade of steel (HSLA-80) is primarily used in naval hull construction [6], the requirement is high strength together with a high value of low-temperature toughness property. However, it is a known fact that an increase in strength is gained at the cost of toughness. Hence there is a need for optimization so that the process variables (in this case heat-treatment parameters) can be defined to achieve the best combination of properties (strength and toughness).

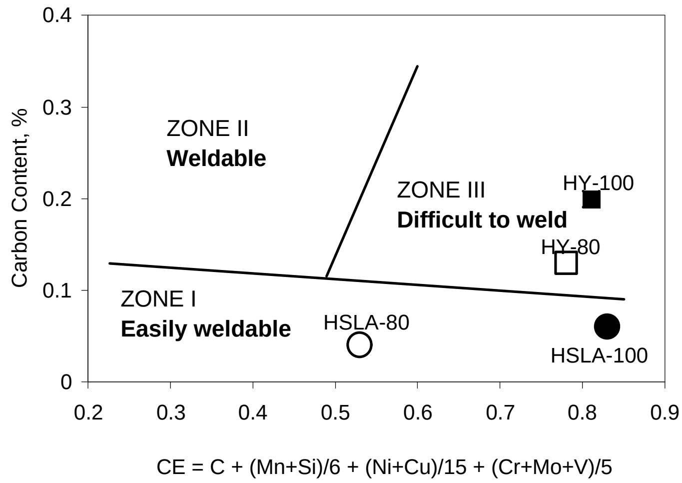

Fig. 1 - Relative positions of HY and HSLA steels in weldability diagram [4,5].

A number of literature [3-10] are available on the structure-property correlation of HSLA steels. There were also attempts [4,5,7] to define the zone of optimum combination of properties through single variable technique, where the effect of variation of one variable is studied when the other variables are kept at constant levels. Using this method Foley and Fine [7] defined the zone of optimum combination of properties. The drawback of this technique is that it does not account for the complex interaction effect of the variables when they change simultaneously. This difficulty can be overcome by applying statistical design of experiments where all the variables are varied simultaneously in a planned manner [11-14].

In the present study, initially single variable experiments were conducted in a broad range of tempering time and temperature combinations to locate the zone where the optimum combination of properties lies. Regression equations for mechanical properties have been obtained in this range of variations of heat-treatment parameters. Because of the complexity of the equations 3-D plots have been made to visualize and understand the nature of the response surfaces. In the next stage, experiments were planned in a shorter range (i.e. in the zone where the optima lies) using 2^2 factorial design of experiments. The regression equations obtained in this shorter zone are used to determine the heat-treatment parameters for obtaining the best combination of properties. In this study the optimization of properties has been done by Grid Search Method. The computer program developed for this purpose searched for the highest value of one property (in this case low-temperature toughness) with a constraint on the other (yield strength). It was found that the best combination of properties (CVN = 219 joule, YS = 635 MPa) occurred when the steel was tempered at a temperature of 670 °C for a time duration of 0.5 hour.

2. Experimental

The steel for this study was received from the US Naval Research Laboratory in the form of plate having dimensions 250 mm x 300 mm x 50 mm. It was examined for inclusion contents and the grain sizes were measured using Quantimet-570 image analysis system.

Initially heat-treatment was carried out at three different austinitization temperatures (900, 950, 1000 °C – 1 hour) followed by quenching in water, and then tempering at different temperatures ranging from 450-700 °C at intervals of 50 °C. Temperatures during heat-treatment were maintained within an accuracy of ±3 °C. The preliminary investigations revealed that the strength increased at tempering temperature between 600-700 °C where the toughness value was also high. Therefore further experiments were conducted in this tempering temperature region using 2^2 factorial design of experiments.

All the samples for tensile and low-temperature Charpy tests were prepared in LT direction. The tensile tests were done as per ASTM E-8-78 methodology in an Instron-1195 machine at a crosshead speed of 5 mm/min. For determining low-temperature toughness property Charpy impact tests were done at –50 °C. The surfaces of broken Charpy samples were examined in a JEOL scanning electron microscope. For characterization of heat-treated samples, transmission electron microscopy was performed using thin foils prepared from tested Charpy samples. The thin foils were examined in a JEOL transmission electron microscope at an operating voltage of 100 kV.

3. Results and Discussions

The chemical composition of the steel is given in Table 1. The inclusion content was found to be less than 10^{-4} area fraction. The average grain size was found to be in the range of 7-8 μm. The tensile properties of the steel in as-received condition are given in Table 2.

Table 1. Chemical composition of the steel

Element	C	Mn	P	S	Si	Cu	Ni	Cr	Mo	Al	Cb	V	Ti
Wt %	0.05	1.00	0.009	0.001	0.34	1.23	1.77	0.61	0.51	0.025	0.037	0.004	0.003

Table 2. Mechanical Properties of as-received steel

	Supplied*	Experimental
YS (MPa)	658.0	636.7
TS (MPa)	757.9	708.2
YS/TS	0.87	0.90
%EL on 25 mm GL	32	30

*** -** value supplied by the US agency.

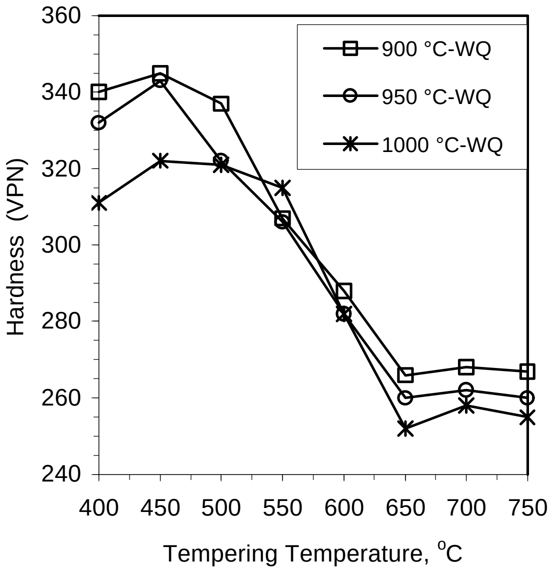

Fig. 2 - Tempering temperature vs. hardness curves.

The tempering temperatures vs. hardness curves are shown in Fig.2. It is seen that there is an increase in hardness with increase in tempering temperature for all austinitization temperatures (900, 950 and 1000 °C) and a peak is reached at a tempering temperature of about 450 °C. The hardness then sharply decreases till a tempering temperature of ~600 °C, whereupon it again increases and a second peak is obtained between 640-700 °C.

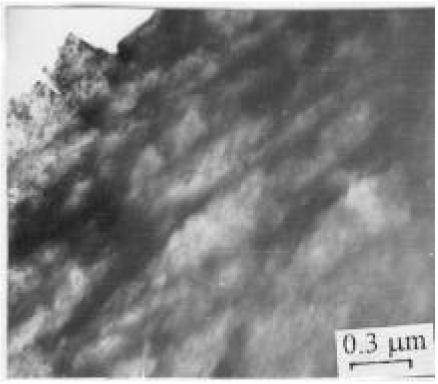

Fig. 3a - TEM of Quenched and tempered steel (450 °C, 1 hour) showing lath structure with dislocations.

Fig. 3b - TEM of Quenched and tempered steel (450 °C, 1 hour) showing occurrence of precipitates due to tempering.

Figs.3a,b show transmission electron micrographs of steel tempered at 450 °C (1 hour). The transmission electron micrographs reveal lath structure with coherent Cu-rich clusters [4,5]. The blurred regions (Fig.3b) indicate coherency strains due to the Cu precipitates. The precipitation of fine coherent Cu-precipitates along with dislocation and coherency strains without recovery explains the increase in strength and hardness, and low value of toughness at this tempering temperature.

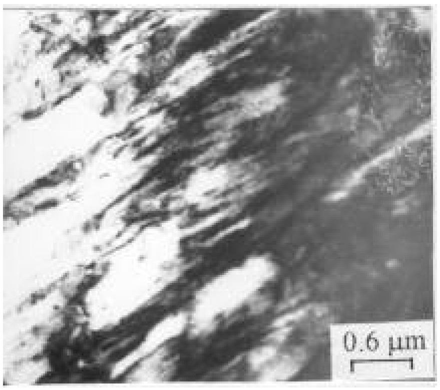

Fig. 4 - TEM of Quenched and tempered steel (600 °C, 1 hour) showing recovered structure.

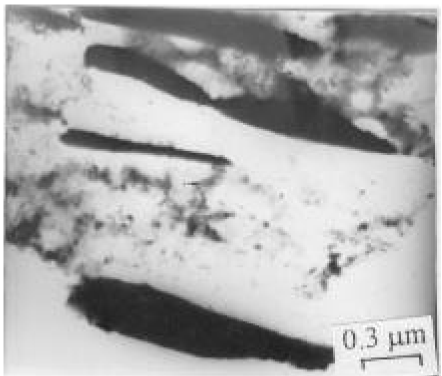

Fig. 5 - TEM of Quenched and tempered steel (700 °C, 1 hour) showing second generation austenite (dark regions) at lath boundaries.

The transmission electron micrograph of steel tempered at 600 °C shows some recovery with clean ferrite and few precipitates (Fig.4). The coarsening of the precipitates and recovery of the matrix explain the decrease in hardness at this temperature.

The transmission electron micrograph of samples tempered at 700 °C shows formation of new generation austenite at the prior lath boundary [4,5], in a direction parallel to the lath (Fig.5). In the early stages of formation, the new austenite is rich in solutes such as Ni, Cu, Mn and Cr. This austenite is highly stable due to the presence of austenite stabilizing agents like Ni and Cu [4,5,7] and is retained on cooling. However, at higher tempering temperature stability is reduced since the growth of the austenite causes a dilution in the volume fraction of the stabilizing agents [4,5] and the newly formed austenite changes to martensite/bainite structure on quenching. This is why the hardness again increases and there is a second peak in the hardness curve (Fig.2) in the higher tempering temperature range (650-700 °C). The thermally stable new austenite also causes an improvement in the toughness of the steel [7].

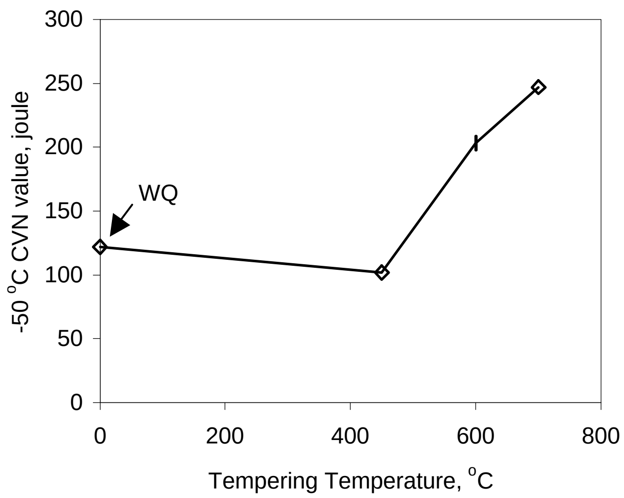

Fig. 6 - Variation of –50 °C CVN values with tempering temperature.

Fig.6 shows the variation of –50 °C CVN values with tempering temperatures. The toughness decreases during the initial stages of tempering reaching a minimum at peak hardening (102 joule at a tempering temperature of 450 °C). This is possibly due to the coherent Cu-precipitates along with dislocation and coherency strains without recovery [3]. The fracture surface of broken Charpy test samples tempered at 450 °C (and tested at –50 °C) shows (Fig.7) quasi-cleavage fracture, a characteristic of typical brittle fracture. The toughness subsequently increases with increasing tempering temperature (258 joule at 700 °C). The improvement in toughness may be attributed to recovery, incoherent Cu-precipitation and formation of new generation austenite[3,15]. The new austenite acts as a sink for detrimental grain boundary impurities and thus reduce intergranular fracture [4,5]. The fracture surface of Charpy test samples tempered at 700 °C shows dimples on the fracture surface (Fig.8) indicating a ductile fracture and hence an improvement in toughness.

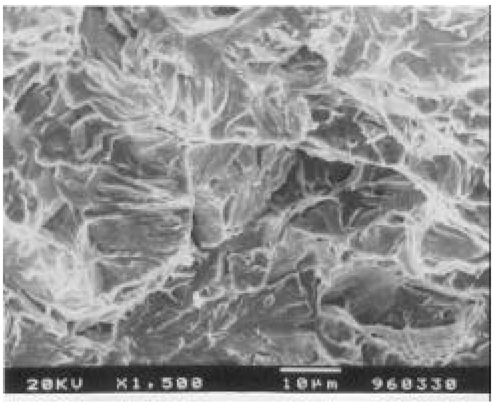

Fig. 7 - Fractograph of broken Charpy specimen (Quenched and tempered at 450 °C, 1 hour) shows quasicleavage nature.

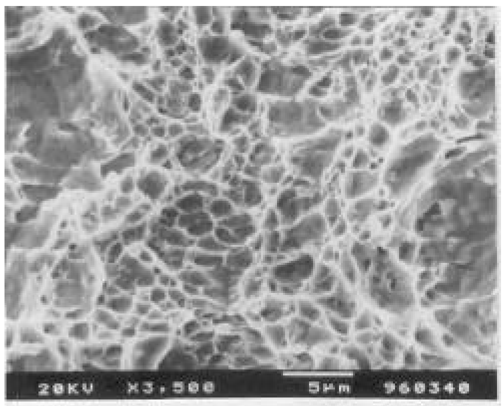

Fig. 8 - Fractograph of broken Charpy specimen (Quenched and tempered at 700 °C, 1 hour) shows dimple fracture.

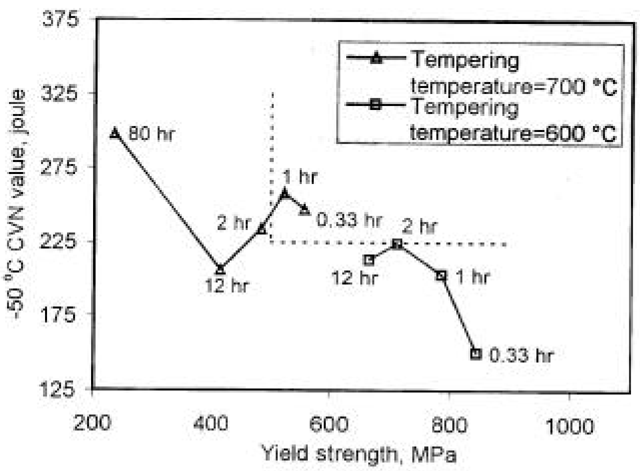

Fig. 9 - Yield stress vs. CVN value at different tempering temperature-time combinations.

It is clear from the above facts that the optimum combination of strength and toughness was likely to be obtained in the neighborhood of 650-700 °C. Hence further experiments were carried out by tempering the steel at 600 °C and 700 °C for different lengths of time. The results of these experiments are given in Table 3. These data have been used to plot Charpy values against yield stresses at the two different tempering temperatures, i.e. 600 °C and at 700 °C (Fig.9). Each point on the curve indicates the properties obtained for a particular combination of tempering time and temperature. The dashed line in the figure shows the region where the yield stress is high (above 500 MPa) and the low-temperature Charpy value is also high (more than 225 Joule).

Table 3. Mechanical properties after tempering the steel at different temperature and time combinations

Tempering temperature(°C) – time (hour)	YS (MPa)	TS (MPa)	YS/TS	–50 °C CVN Energy (J)
700-0.33	556.2	662.2	0.85	247
700-1.0	522.8	656.7	0.80	258
700-2.0	483.6	633.7	0.76	234
700-12	413.0	570.9	0.72	206
700-80	232.5	341.4	0.68	298
600-0.33	843.7	853.5	0.99	150
600-1.0	783.8	793.4	0.99	203
600-2.0	708.3	715.1	0.99	224
600-12	661.2	673.9	0.98	213

Using the data of Table 3 the properties have been quantified by classical least square method. The equations obtained are:

$$YS = (2305.105 - 2.5604\ T).\ t^{(3.4589 - 0.551 \ln T)} \quad (1)$$

$$TS = (1528.94 - 1.3410\ T).\ t^{(1.994 - 0.322 \ln T)} \quad (2)$$

$$CVN = (0.03544\ T - 24.3566).t^{2} + (-0.52484\ T + 357.7386).t + (1.0706\ T - 493.13) \quad (3)$$

In eqns. (1) to (3), T is the tempering temperature in °C and t is the time of tempering in hours. YS and TS are the yield strength and tensile strength respectively in MPa, CVN is the –50 °C Charpy impact value in Joules. The above equations are valid within the experimental region (tempering temperature = 600-700 °C and tempering time = 0.33-12 hour) and can be effectively used to determine the properties at a particular combination of tempering time and temperature.

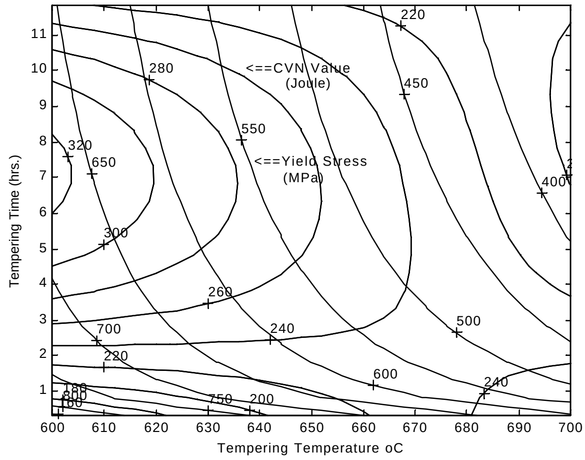

Fig. 10 - Contour plot of YS and –50 °C CVN value (Region: 600 °C - 700 °C and 0.33 - 12 hours).

Fig.10 shows the iso-property contours of yield strength and Charpy impact value. The nomogram gives an idea of the properties for a particular combination of heat-treatment variables.

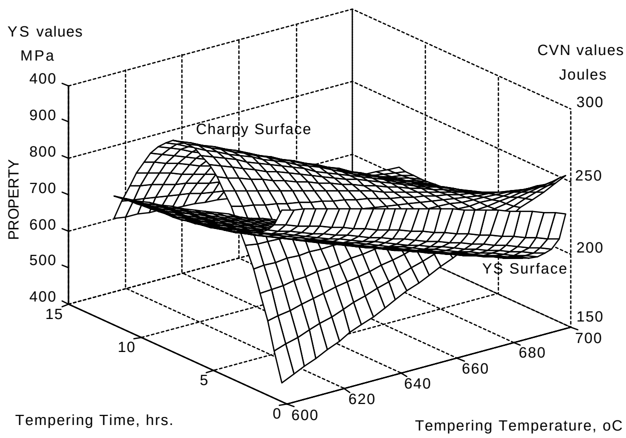

Fig. 11 - Yield strength response surface superimposed on Charpy response surface (Region: 600 °C - 700 °C and 0.33 - 12 hours).

To visualize the nature of variation of yield strength and Charpy value with tempering time and temperature, the response surfaces are shown in a 3-dimensional space with Charpy surface superimposed on the YS surface (Fig.11). It can be observed from this figure that the optimum combination of properties lies in the region of tempering time varying between 0.3-0.8 hours and tempering temperature varying between 600-700 °C.

Table 4. 2^2 Design Matrix showing heat-treatment variables and responses (Region 600 °C - 700 °C and 0.33 - 2 hours)

HEAT TREATMENT VARIABLES				RESPONSES	
Tempering Temperature		Tempering Time		YS (MPa)	–50 °C CVN value (J)
Coded value X_1	Decoded value x_1 (°C)	Coded value X_2	Decoded value x_2 (hour)		
–1	600	–1	0.33	843.7	150
–1	600	+1	2.00	708.3	224
+1	700	–1	0.33	556.2	247
+1	700	+1	2.00	483.6	234

It was therefore decided to carry out further analysis in this region of tempering time and temperature using 2^2 factorial design of experiments. Table 4 shows the design matrix for YS and –50 °C CVN value prepared over a tempering temperature range of 600-700 °C (base level temperature = 650 °C) and tempering time 0.33-2 hours (base level = 1.165 hour). The regression equations formed using this design matrix are

$$YS = 647.95 - 128.05\, X_1 - 52\, X_2 + 15.7\, X_1 . X_2 \qquad (4)$$

$$CVN = 213.75 + 26.75\, X_1 + 15.25\, X_2 - 21.75\, X_1 . X_2 \qquad (5)$$

where $X_1 = (x_1 - 650) / 50$ and $X_2 = (x_2 - 1.165) / 0.835$ (6)

x_1, x_2 are natural values of temperature and time of tempering in °C and hours respectively, YS is the yield strength (MPa) and CVN is the Charpy V-notch value at –50 °C (Joule). X_1 and X_2 are in coded form and can be decoded by using the relations given in equation (6).

The significance of the coefficients of X_1, X_2 and $X_1.X_2$ in equations (4) and (5) have been given elsewhere [10]. The equations (4) and (5) are simpler in nature compared to equations (1) and (2). This is because the response surfaces represented by equations (3) and (4) are almost plane in nature (Figs. 12 and 13) since the range of variations of tempering time has now been narrowed down to 0.33-2 hour.

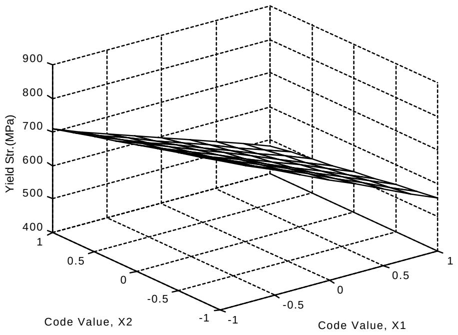

Fig. 12 - Charpy response surface (Region: 600 °C - 700 °C and 0.33 - 2 hours).

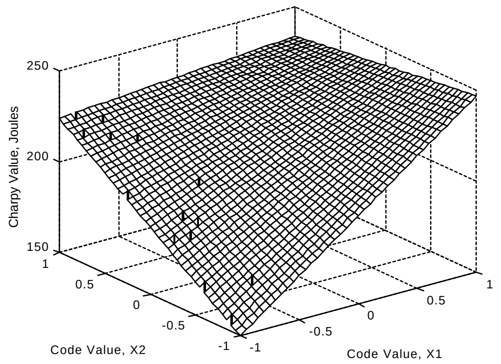

Fig. 13 - Yield strength response surface (Region: 600 °C - 700 °C and 0.33 - 2 hours).

Equations (4) and (5) have been used for finding the optimum combination of properties. This has been done using Grid Search technique. A suitable computer program drawn for this purpose utilized equations (4) and (5) to search for the condition where the Charpy value is a maximum with a constraint of YS ≥ 630 MPa. The program calculated Charpy value and YS at different coded values of tempering temperature and time (i.e. $X_1$and X_2). It was seen that the optimum condition occurred at $X_1 = 0.35$ and $X_2 = –0.55$. Decoding the above values using equation (6), it was found that the optimum combination (i.e. YS = 634.75 MPa, CVN = 218.91 joule) occurred at a tempering temperature of 670 °C (corresponding to $X_1 = 0.35$) and tempering time of 0.5 hour (corresponding to $X_1 = –0.55$).

4. Conclusions

On the basis of the above discussions, the conclusions are summarized as

i) The steel (Cu-bearing HSLA-80) responds well to tempering and significantly alters the mechanical properties between 600-700 °C tempering temperature.

ii) Maximum strength is obtained at a tempering temperature of 450 °C. However, the low-temperature toughness property is very poor at this tempering temperature. This is because of the precipitation of fine Cu-precipitates along with dislocations and coherency strains without recovery.

iii) The strength decreases with increase in temperature beyond tempering temperature of 450 °C till 600 °C. After this the strength again increases and the toughness also improves. This is explained by the formation of new austenite at higher temperatures and subsequent transformation to martensite/bainite on quenching.

iv) The mechanical properties (yield strength, tensile strength, low-temperature toughness) are quantified over a wide range of tempering time varying between 0.33-12 hours and tempering temperature varying from 600-700 °C. To help perceive the variations of responses with time and temperature of tempering, the response surfaces of YS and Charpy value are shown in a 3-dimensional space. The superimposed plot of response surfaces indicates the feasible region where the optimum combination of properties occurs.

v) A nomogram (contour plot) is constructed to allow the user to select the heat-treatment parameters for obtaining a particular combination of properties.

vi) Statistical design of experiments is applied in a narrower range of heat-treatment variables (tempering temperature = 600-700 °C, tempering time 0.33-2 hours). The regression equations obtained from the 2^2 design matrix are used for the purpose of optimization.

vii) Optimization has been done using Grid Search technique. The optimum values are found to be YS = 634.75 MPa and –50 °C CVN value = 218.91 joule occurring at a tempering temperature of 670 °C and tempering time of 0.5 hour

viii) By proper selection of heat-treatment parameters high strength together with high low-temperature toughness property can be achieved in copper precipitation strengthened HSLA-80 steel.

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