Microstructure and Properties of Quenched-and-Aged Plates Produced from a Copper-Bearing HSLA Steel

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For the first time in India, quenched-and-tempered (Q&T) plates of a copper-bearing high-strength lowalloy (HSLA) steel have been commercially developed for naval structural applications. A 50 ton production heat was made through electric arc furnace (EAF)-vacuum arc degassing (VAD) route and continuously cast into 170 mm thick slabs. These slabs were conditioned, reheated in walking-beam furnace and hot rolled in plate mill into plates of 10 to 16 mm thickness. The as-rolled plates were hardened through oil quenching and subsequently tempered (aged) at 630 °C to achieve the combination of highstrength and good low-temperature impact toughness.

The microstructures of heat treated plates showed fine acicular ferrite with grain sizes ranging between ASTM No. 9 and 10. From the standpoint of tensile properties, Q&T plates of all thicknesses exhibited significantly higher yield strengths than the minimum stipulated value of 552 MPa for HY-80/HSLA-80 steels. The elongation (22.20 to 26.00%) and reduction in area (62.12 to 67.62%) values achieved also exceeded the respective minimum requirements of 20 and 50% stipulated for such steels. The trend in variation of Charpy V-notch (CVN) impact energies at room temperature, -18, and -62 °C not only showed significantly higher values than that stipulated for HY-80 and HSLA-100 steels at -18 °C, but also indicated that the CVN impact energies achieved (105.15 to 144.25 J) at -62 °C were higher than the estimated value of 90 J for HSLA-80/HSLA-100 steels at this temperature.

Keywords copper-bearing plate steel, HSLA-80, HY-80, precipitation hardening acicular ferrite

1. Introduction

Copper-bearing, low-carbon plate steels, which acquire high strength as a consequence of precipitation hardening, have attracted considerable interest for various applications. Generically speaking, these series of steels evolved from IN-787, which was developed by the International Nickel Company in the late 1960s (Ref 1). For structural applications, the American Society of Testing Materials (ASTM) designated steels with a nominal chemistry of 1 wt% Cu, 0.8 wt% Ni, 0.7 wt% Cr, 0.2 wt% Mo, and a small quantity of niobium as A 710, whereas that for pressure vessel applications was designated as A 736 (Ref 2). Over the years, these types of steels have been used in numerous applications such as mining and dredging equipment, offshore platforms, valves for arctic pipelines, and so forth. In the past few years, the U.S. Navy considered replacement of the HY-80 steel by HSLA-80 because of the latter's superior properties: high yield strength (80 ksi, 552 MPa, minimum), excellent low-temperature toughness, and good weldability.

Metallurgically speaking, HSLA-80 is a precipitation-hardening ferritic steel containing about 1.2 wt% Cu. The high

strength achieved in this steel is attributed to the precipitation of ε -copper particles during aging (Ref 3). Copper, which is the primary element responsible for precipitation strengthening in the steel, has been found (Ref 4) to have a maximum solubility of 3% at the austenitizing temperature and limited solubility at room temperature. When the steel is quenched rapidly from the austenitizing temperature, a considerable amount of copper is retained in the ferrite and upon aging at appropriate temperature, is precipitated as ε -copper. These fine copper precipitates, being coherent with the matrix, impede dislocation movement, and this results in increased yield strength. Niobium also plays an important role in achieving high strength in this steel. Owing to its limited solubility at the austenitization temperature, niobium forms fine niobium carbonitrides that prevent austenite grain coarsening. Thus in these high-strength low-alloy (HSLA) steels, copper acts as the primary strengthening element, while niobium has a secondary strengthening role. Initially, copper-bearing, precipitation-aged steels were developed for use in the hot-rolled and aged condition. Subsequently, with the formulation of ASTM specifications for such types of steels, three classes of steels-class 1 for rolledand-aged plates, class 2 for normalized-and-aged plates, and class 3 for quenched-and-aged plates—were developed.

In India, the HY-80/HSLA-80 grades of steels had not yet been commercially produced, and therefore efforts were directed at Steel Authority of India Limited (SAIL) to indigenously develop such types of steel plates through an electric arc furnace (EAF), vacuum arc degassing (VAD), and continuous-casting (CC) route. This article discusses the structure/property aspects of these commercially produced steel plates.

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2. Experimental

2.1 Steel Processing

A 50 ton EAF heat was made and then subjected to VAD treatment and ladle refining to improve steel cleanliness. The chemical composition of the steel heat is shown in Table 1. The molten steel was continuously cast into 900 mm wide by 170 mm thick slabs, which were then conditioned to remove any apparent surface defects. The conditioned slabs were heated up to 1280 to 1300 °C in a walking-beam furnace and soaked for $2^{1}/_{2}$ to 3 h. During soaking in the walking-beam furnace, oxygen content was restricted to below 2% for minimizing oxidation of slabs.

Following reheating, slabs were hot rolled in the plate mill into 10, 12, 14, and 16 mm thickness plates by following a conventional draft schedule. During hot rolling, a finishing temperature of 900 \pm 15 °C was maintained. Descaling with water and salt spray was used during rolling to facilitate removal of sticky scales. Proper flatness of the rolled plates was ensured by hot leveling.

The as-rolled plates were ultrasonically tested and then heat treated by quenching and tempering. During hardening, the austenitization temperature was maintained at 925 ± 10 °C, while soaking time was varied between 30 and 45 min, depending on plate thickness. After austenitization at the hardening temperature, the plates were quenched in oil and then tempered at 630 ± 10 °C. The tempering (aging) time was varied between 110 to 160 min depending on the plate thickness. Following tempering, the plates were cooled in still air to ambient temperature.

2.2 Evaluation of Plate Properties

The tensile properties of the heat treated (quenched-andtempered) steel plates were evaluated in accordance with ASTM specification A 370-95. For tensile testing, coupons were selected from 10, 12, 14, and 16 mm thick plates and machined into standard rectangular test specimens of 12.5 mm width. Tensile tests on 10 mm thick plate samples were carried out in a 10 ton capacity "INSTRON-1195" model (INSTRON Ltd., High Wycombe, Buckinghamshire, England) universal testing machine, while specimens of 12, 14, and 16 mm thick plates were tested in a 25 ton capacity "INSTRON-1273" (INSTRON Ltd., High Wycombe, Buckinghamshire, England) dynamic testing machine. During testing, a crosshead speed of 2 mm/min was employed, and a 50 mm gage length (GL) extensometer was used for determining the elongation percentage. For each plate thickness, at least two specimens were tested.

Charpy V-notch (CVN) impact properties were evaluated for full-size (10 by 10 by 55 mm), transverse-orientation specimens of 12, 14, and 16 mm thick heat treated steel plates in accordance with ASTM A 673/A 673 M 95. For each plate thickness, samples from at least two plates were tested, and for each plate, the average impact energy of three test specimens was reported. Impact tests were carried out at room temperature $28 \,^{\circ}$ C, -18 and $-62 \,^{\circ}$ C after immersing the specimens in a lowtemperature bath (maintained at the requisite temperature) for at least 15 min.

Optical microscopic examinations were carried out on etched and unetched specimens of heat treated steel plates to observe general microstructure and nonmetallic inclusion characteristics, respectively. The plate microstructures were observed at 500×, while grain-size measurements were made by linear-intercept method and also corroborated by ASTM comparison graticules at 100×. The distribution and nature of nonmetallic inclusions were observed in the longitudinal through thickness (LT) sections of the plate samples through optical microscopy at 100× and 500×, respectively. Further, inclusion assessment according to ASTM E 45-87 was carried out in a "Quantimet-600" (LEICA Imaging Systems Ltd., Cambridge, England) image-analysis system. Similarly, the fracture surfaces of impact specimens tested at -18 and -62 °C were examined in a "JSM-840A" (JEOL Ltd., Akishima, Tokyo, Japan) scanning electron microscope (SEM) to observe the general surface topography. Transmission electron microscopy (TEM) studies were also conducted on thin-foil specimens of heat treated plates in a 400 kV "JEM-4000EX" (JEOL Ltd., Akishima, Tokyo, Japan) TEM to observe precipitate characteristics.

3. Results and Discussions

3.1 Alloy Chemistry

The chemical composition (Table 1) of the production heat showed that the carbon content achieved was within the permissible range (as per MIL-S-24645A standard) for HSLA-80 steel (Ref 5), being slightly lower (0.054 wt%) than the maximum (0.06 wt%) stipulated value. A lower carbon content (0.054 wt%) is nonetheless preferable for achieving a lower carbon equivalent (CE) necessary for good weldability. The copper content achieved (1.01 wt%)-although within the permissible range (1.00 to 1.30 wt%) for HSLA-80 steel-is slightly lower than the maximum allowable level of 1.3 wt%. Copper is added in this steel primarily to achieve precipitation strengthening because copper has high solubility in the austenite phase and only limited solubility in ferrite. However, copper is associated with hot shortness, and therefore the production steel chemistry incorporated slightly higher (1.06 wt%) nickel than that stipulated for conventional HSLA-80 (0.70 to 1.00 wt%) to counteract this effect. It is well known

Table 1 Chemical composition of production HSLA he
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Element(a)	Composition, 9		
Carbon	0.054		
Silicon	0.33		
Manganese	0.79		
Sulfur	0.010		
Phosphorus	0.010		
Nickel	1.06		
Chromium	0.74		
Molybdenum	0.29		
Niobium	0.037		
Copper	1.01		

(a) Tramp elements: Arsenic, antimony, and tin in trace amounts, i.e., <20 ppm

that copper raises the impact-transition temperature (ITT), whereas nickel lowers it. The combined additions of copper and nickel in this steel therefore serve to nullify their individual effects on the CVN impact properties. It has been reported (Ref 2) that addition of about 0.9 wt% Ni and 0.04 wt% Nb has no significant effect on the aging behavior of copper-bearing steels, but increases strength by solid-solution strengthening and grain refinement. The concentrations of nickel and niobium achieved in this steel are similar to the values mentioned above. In copper-bearing steels, copper combined with other residual elements such as tin, arsenic, and antimony can lead to severe hot shortness under unfavorable reheating conditions. This is because tin and antimony decrease the solubility of copper in austenite, leading to the precipitation of a molten phase at the grain boundaries (Ref 6-8). In this steel, however, the tin, arsenic, and antimony contents are in trace amounts, and therefore their effect is not a factor.

The CE value for this steel when calculated in accordance with the formula (Ref 5):

$$CE = C + \frac{Mn + Si}{6} + \frac{Ni + Cu}{15} + \frac{Cr + Mo + V}{5}$$

is found to be 0.59%. This is conducive to good weldability as shown by the carbon (0.054 wt%)-CE (0.59%) location in zone 1 ("safe under all conditions") of Graville's diagram in Fig. 1 (Ref 9).

3.2 Microstructural Features

The nital-etched microstructures in all thicknesses of heat treated plates consisted of acicular ferrite. Typical optical micrographs showing acicular ferrite in 12, 14, and 16 mm thick plates are shown in Fig. 2(a), (b), and (c), respectively, at 500×. The average grain sizes of the plates, as determined by linear-intercept method were found to be very fine and corresponded to ASTM No. 9 for 16 mm thick plate and ASTM No. 10 for 10,



Fig. 1 Weldability of steel as a function of carbon content and carbon equivalent from Graville (Ref 9)

12, and 14 mm thick plates. Typical micrographs showing fine acicular ferrite (grain size, ASTM No. 10) in plates of 12 and 14 mm thickness are shown in Fig. 3(a) and (b), respectively, at 100×. Figure 4 shows the bright-field TEM photograph of a thin-foil specimen pertaining to a 14 mm thick Q&T HSLA steel plate at $30,000\times$. Fine dispersions of ε -copper precipitates



Fig. 2 Optical micrographs showing acicular ferrite in HSLA steel plates. (a) 12 mm thickness. (b) 14 mm thickness. (c) 16 mm thickness. $500 \times$ (Art has been reduced to 83% of its original size for printing).

along with some NbCN particles are seen in a ferrite matrix. Energy-dispersive spectrometric (EDS) analysis (Fig. 5) of the larger particles corroborated the presence of niobium. The fine grain size achieved in the HSLA plates can be attributed to the control of finish-rolling temperature as well as to the grain-refining role of niobium in the steel chemistry.

Nonmetallic inclusions were examined in accordance with ASTM E 45-87 and ratings are shown in Table 2. The fields observed were found to be clean and virtually devoid of manganese sulfide (MnS) stringers. In a few places, however, tiny globular-oxide-type inclusions were observed. The virtual absence of MnS stringers is consistent with the low sulfur content (0.01 wt%) of this steel.

3.3 Tensile Properties

The tensile properties of heat treated steel plates of different thicknesses are shown in Table 3. As shown in this table, the yield strength (YS) achieved in test specimens of all plate thicknesses far exceeds the minimum stipulated requirement (Table 4) of 552 MPa for HY-80 and HSLA-80 grades of steels. The average values of YS obtained in test specimens of 10, 12,



Fig. 3 Optical micrographs showing fine-grained acicular ferrite in HSLA steel plates. (a) 12 mm thickness. (b) 14 mm thickness. $100\times$ (Art has been reduced to 83% of its original size for printing).

14, and 16 mm thick plates were found to be 641.56, 634.54, 637.30, and 608.68 MPa, respectively. The corresponding average ultimate tensile strength (UTS) values for 10, 12, 14, and 16 mm plate samples were found to be 710.24, 712.04, 712.74, and 699.82 MPa, respectively. From these data, it is evident that while the average values of YS and UTS are similar for 10, 12, and 14 mm thickness plates, these values—as expected—are marginally lower in case of the higher thickness 16 mm plates, presumably owing to slightly larger (ASTM No. 9) grain size. The YS/UTS ratios in tensile-tested specimens of 10 to 16 mm thick plates were found to be uniform and ranged between 0.87 and 0.91, which is expected for such high-strength steels.

Table 2Inclusion rating of commercially produced HSLAsteel plates as per ASTM E 45-87

Sulfide		Alumina		Silicate		Globular oxide	
Thin	Heavy	Thin	Heavy	Thin	Heavy	Thin	Heavy
						2	
Note:	A-Sulfide:	B-Alum	ina: C-Sili	cate: D-0	Globular o	xide	



Fig. 4 Bright-field TEM image of thin-foil specimen from 14 mm thick quenched-and-aged HSLA steel plate showing copper precipitates. 30,000×

The elongation values (Table 3) achieved in test specimens of 10, 12, 14, and 16 mm thickness plates ranged between 22.2 and 26.0% and were therefore higher than the minimum stipulated requirement (Table 4) of 20.0% elongation for



Fig. 5 EDS spectrum of niobium-rich particles in Q&T plates of the production HSLA steel



Fig. 6 Variation of CVN impact energy with temperature in transverse specimens of HSLA steel plates

HY-80/HSLA-80 grades of steels. The reduction-in-area (RA) values obtained in test specimens of 10, 12, 14, and 16 mm thickness plates ranged between 62.12 and 67.62% and were also higher than the minimum stipulated requirement (Table 4) of 50% for HY-80/HSLA-80 grades of steels.

3.4 Impact Properties

The CVN impact properties of full-size, transverse-orientation test specimens pertaining to 12, 14, and 16 mm thick heat treated plate samples, tested at 28 °C (room temperature), -18, and -62 °C, are shown in Table 5. At room temperature (28 °C), the CVN impact energies ranged between 194.86 and 196.49 J (average 195.67 J) for 12 mm thick plates, between 181.63 and 194.37 J (average 188 J) for 14 mm thick plates, and between 193.87 and 195.18 J (average 194.52 J) for 16 mm thick plates.

At -18 °C, the impact energy values were found to range between 151.21 and 164.44 J (average 157.82 J) for 12 mm thick plates, between 136.51 and 140.14 J (average 138.32 J) for 14 mm thick plates, and between 144.65 and 158.37 J (average 151.51 J) for 16 mm thick plates. All these CVN energy values far exceed the minimum stipulated requirements (Table 4) of 81 J for HY-80 and 108 J for HSLA-100 steels at -18 °C.

For impact tests carried out at -62 °C, the impact energy values were found to range between 119.76 and 142.69 J (average 131.22 J) for 12 mm thick plates, between 105.15 and 144.25 J (average 124.70 J) for 14 mm thick plates, and between 109.56 and 138.61 J (average 124.08 J) for 16 mm thick plates.

The variation of CVN impact energies in transverse-orientation specimens of 12, 14, and 16 mm thick plates at room temperature (28 °C), -18, and -62 °C is shown (by solid symbols)

Table 3Tensile properties of commercially producedQ&T HSLA steel plates

Plate thickness, mm	Plate No.	YS, MPa	UTS, MPa	Elongation in 50 mm, %	RA %	YS/UTS
16	А	602.80	691.29	25.40	66.40	0.87
	В	614.56	708.35	22.80	62.12	0.87
14	С	653.83	717.99	23.00	67.62	0.91
	D	620.77	707.49	26.00	65.28	0.88
12	Е	616.13	705.80	25.00	64.80	0.87
	F	652.95	718.29	22.20	64.58	0.91
10	G	643.72	712.40	25.00	63.99	0.90
	Н	639.41	708.08	26.00	63.97	0.90

Table 4Specified yield strength and Charpy impacttoughness properties of HY-80, HSLA-80, HY-100, andHSLA-100 steels (minimum unless a range is shown)

Property	HY-80	HSLA-80	HY-100	HSLA-100
Yield strength, MPa	552-686	552-690	690-825	690-862
(ksi)	(80-99.5)	(80-100)	(100-120)	(100-125)
Elongation, %	20	20	18	18
Reduction of area, %	50	50	45	45
Charpy impact, J (ft ·]	lb)			
at –18 °C	81 (60)		81 (60)	108 (80)
at-84 °C	47 (35)	81 (60)	54 (40)	81 (60)
Source: Ref 5				

in Fig. 6. Figure 6 also depicts (by open symbols) the minimum stipulated CVN impact energy requirements (Table 4) for HY-80, HSLA-80, HY-100, and HSLA-100 grades of steels. It is evident from Table 4 as well as from Fig. 6 that while CVN impact energies are stipulated for HY-80, HY-100, and HSLA-100 steel grades at -18 and -84 °C, the requirement for HSLA-80 is specified at -84 °C only. However, because both the HSLA-80 and HSLA-100 grades of steels have the same CVN energy requirement (81 J) at -84 °C, it can be assumed that the HSLA-80 grade should at least possess similar (i.e., 108 J for HSLA-100 at -18 °C) or otherwise marginally higher energy value at -18 °C.

The CVN impact energy values achieved in transverse specimens of 12, 14, and 16 mm thick Q&T HSLA plates fall within the scatter band shown in Fig. 6. The scatter, quite understandably, is the least at room temperature (28 °C) and widens with lowering of test temperature. Nevertheless, even the lower boundary of the impact energy scatter band (Fig. 6) at -18 °C corresponds to a CVN value 136.51 J (Table 4) for 14 mm plate. This is significantly higher than the minimum stipulated CVN energy requirements for HY-80 (81 J), HY-100 (81 J), and HSLA-100 (108 J) at -18 °C. If the stipulated CVN energy values depicted for HSLA-100 steel at -18 °C (108 J) and -84 °C (81 J) in Fig. 6 are joined, then the estimated CVN impact energy value at -62 °C would presumably be 90 J. Because the CVN impact energy requirements for both the HSLA-100 and HSLA-80 grades of steels are the same at -84 °C, the CVN impact energy level of the HSLA-80 grade at -62 °C can be assumed to be similar (i.e., 90 J) to that of HSLA-100 steel. The lower boundary of the impact energy scatter band (Fig. 6) at -62 °C corresponds to a CVN value of 105.15 J (Table 4) for 14 mm plate. This is 15 J higher than the estimated 90 J value for HSLA-100/HSLA-80 steels at the same temperature. The CVN impact energy values of 12, 14, and 16 mm thick Q&T HSLA plates at room temperature, -18, and -62 °C (as seen from Table 4 as well as Fig. 6) are therefore higher than those of HSLA-80 and HSLA-100 grades at the aforesaid temperatures.

3.5 Fractography

The fracture surfaces of impact specimens tested at -18 and -62 °C showed existence of both ductile and brittle regions. Observations by SEM of impact-tested specimens showed presence of a very small, ductile region near the notch, followed by a fully brittle area. Figure 7(a) and (b) are typical SEM micrographs (1000×) showing the presence of ductile

Table 5Charpy V-notch impact properties of full-size,transverse orientation specimens of commercially producedQ&T HSLA steel plates at different temperatures

Plate thickness,	Plate	Average CVN impact energy, J			
mm	No.	28 °C	-18 °C	-62 °C	
16	А	193.87	158.37	138.61	
	В	195.18	144.65	109.56	
14	С	194.37	136.51	144.25	
	D	181.63	140.14	105.15	
12	Е	194.86	164.44	142.69	
	F	196.49	151.21	119.76	

dimples near the notch (Fig. 7a) and brittle cleavage facets away from the notch (Fig. 7b) in an impact-tested specimen (test temperature: -62 °C) of a 12 mm thick plate. Similar fracture topography showing coexistence of brittle and ductile regions were also observed in impact-tested specimens of 14 and 16 mm thick plates. Fractographic evidence of ductile features in impact specimens tested at -18 and -62 °C are therefore testimony to the high toughness values achieved at these temperatures.

4. Conclusions

The broad conclusions regarding structure/property aspects of the commercially produced copper-bearing HSLA steel plates are:

• Microstructurally, steel plates of all thicknesses (10, 12, 14, and 16 mm) exhibited fine acicular ferrite with grain sizes ranging between ASTM No. 9 and 10. The fine ferrite grain size obtained can be attributed to low finishing temperatures employed during hot rolling as well as due to the beneficial effect of niobium in impeding grain coarsening.





Fig. 7 SEM fractograph of Charpy impact specimen tested at -62 °C showing (a) ductile features near notch and (b) brittle cleavage region slightly away from notch. $1000 \times$ (Art has been reduced by 80% for printing).

- The steel is clean from the standpoint of nonmetallic inclusions, and the virtual absence of stringer-type MnS is an indication of the low sulfur content of the production steel.
- The average values of YS attained in plates of 10 to 16 mm thickness were substantially higher than the minimum stipulated value of 552 MPa for HY-80/HSLA-80 steels. Besides, the elongation (22.20 to 26.00%) and RA (62.12 to 67.62%) values achieved also exceeded the minimum stipulated requirements of 20 and 50%, respectively, for these steels.
- Charpy V-notch impact energy values obtained in full-size, transverse-orientation specimens of 12, 14, and 16 mm thick Q&T plates at -18 °C were significantly higher than the minimum stipulated values for HY-80, HY-100, and HSLA-100 grades of steels at this temperature. Also, the CVN impact energy band for the Q&T plates in the temperature range between room temperature and -62 °C also showed higher energy levels than that estimated (90 J) for HSLA-80/HSLA-100 grades of steels at -62 °C.

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