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Effect of tempering temperature on microstructure and mechanical properties of low alloy high strength steel

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Abstract

The effect of tempering temperature on the microstructure and mechanical properties of a low alloy steel has been investigated using optical microscopy, hardness testing and Charpy V-notch impact testing at -20°C, -40°C and -60°C. At first, the laboratory based study revealed that tempering the specimens at 650°C resulted in lowest hardness with highest impact energy. Based on this, the heat-treated industrial steel showed sufficiently high toughness after Charpy V-notch impact testing at -40°C. Fractography after impact testing showed predominately ductile dimple rupture in the sample tempered at 650°C. The presence of decomposed martesitic laths in prior austenite was noticed in the specimens tempered at 650°C and 680°C.

1. Introduction

Quenched and tempered low alloy steels are widely used when high mechanical strength is a main requirement for the specific application. Tempering will suppress brittle fracture and ensure a desired combination of strength and ductility of martensitic steel [1]. The solid solution of quenched martensite containing supersaturated carbon effectively increases the hardness and strength of low alloy and carbon steels. However, the martensite structure is rarely used in a non-tempered condition because the high internal stresses associated with the transformation cause the material to be lacking in ductility [2-3]. In view of this, the ductility/toughness of the quenched martensitic steels is adjusted as per the requirement through tempering at different temperature [4].

The change of microstructure during tempering is generally understood to be composed of three distinct and overlapping stages, i.e. (a) the formation of the transition carbide, (b) the transformation of retained austenite to ferrite and cementite, and (c) the replacement of transition carbide and martensite by cementite and ferrite [5]. Depending upon the required final properties and microstructure, tempering is carried out in the range of 170-700°C. Mechanical properties of quenched steel directly depend on the degree of quenched steel hardening. Fracture toughness and fatigue limit depend on microstructural constituents, and distribution of the usual intermetallic particles and non-metallic inclusions [6]. However, apart from the effect of tempering temperature, the strength of the martensitic structure is dominated by the carbon content and the (Ms–Mf) temperature range [7]. The hardenability in steels is influenced by the chemical composition i.e. amounts of carbon as well as the presence of alloying elements, grain size of austenite, temperature and time for austenitization and the structure of steel before quenching [8].

Silicon as a substitutional solute in ferrite has remarkably effect in generating dislocation during quasi-static tensile deformation and thereby increases dislocation density and hence, increases strength of steel [9,10]. Minimum impact transition temperature has been reported when Si varies in the range of 0.40-0.60% depending on carbon and manganese content [11,12]. It decreases carbon solubility in ferrite and fine carbide formed during air cooling. The softer ferrite resists fracture better. On the other hand, the increase in impact transition temperature beyond 0.60% has been attributed to substantial solid solution strengthening effects, which overshadow carbide precipitation effects [11,12]. Addition of Ni up to 9% in martesitic steels has been mainly carried out to increase cleavage fracture resistance and thus, to lower ductile to brittle transition temperature [13]. However, higher Ni also influences temper embrittlement in martensitic steel [14]. The Ni content in commercial high strength martensitic steel varies from 1.4% to 2.0% [15].

The main objective of this paper was to study the effects of tempering temperature on hardness and Charpy impact energy of a quenched and tempered steel for a commercial production. Though the effect

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of different elements are known from the literatures, the exact required composition is not available for achieving a particular mechanical properties like hardness and Charpy impact energy. In view of this, a particular composition with moderate Ni was designed so that the steel will be economical without compromising the required mechanical properties. After hardness study through laboratory scale heat treatment, the plant trial was carried out.

2. Experimental procedures

The chemical composition of the quenched and tempered grade low alloy steel studied in this investigation is given in Table 1. The initial slab size was 2300 mm (L) x 900 mm (W) x 250 mm (T). The slabs were hot rolled at 1150°C into 150 mm thick plates after soaking at 1280°C for nearly 4 hours.

To understand the mechanical properties and microstructure of quenched and tempered steel, hot rolled plates were cut into specimens 40 mm (T) x 60 mm (W) x 70 mm (L). These specimens were heat treated at 920°C for soaking time 2 minutes/mm (80 minutes) in laboratory muffle furnace. The shorter time at higher temperature was considered to avoid over oxidation of samples. This temperature is above Ac_3 temperature. For low alloy steels containing carbon between 0.08 and 1.4 wt.%, Ac_3 temperature is given by the following Hougardy's empirical equation [16].

$$Ac_3 = 902\text{-}225C\text{-}11Mn\text{+}19Si\text{-}5Cr\text{+}13Mo\text{-}20Ni\text{+}55V$$

Based on the above calculation, the temperatures had been selected in austenitic regime, i.e, 920°C. This was followed by quenching in water. After water quenching, samples were tempered at different tempering temperatures such as 500, 560, 600, 650, and 680°C for 4 min/mm.

Charpy impact tests were conducted on regular size Charpy V-notch specimens as per ASTM E 23 (10 mm × 10 mm × 55 mm) on pendulum-type impact testing machine using 300 J hammer at cryogenic temperatures such as -20, -40 and -60°C. Instron Wolpert Rockwell Hardness Tester Series 600MRD^{TM} was employed for present study. The specimens for the light optical microscopy were prepared by successive grinding in different grades of SiC paper followed by cloth polishing using alumina suspension.

The polished specimens were etched with Nital solution (2% Nitric acid solution & 98% Ethyl Alcohol) for a few seconds, followed by rinsing with water. The etched specimens were examined by using inverted metallographic microscope of Olympus make. Fractographic examination of the fractured Charpy specimens was carried out with the help of EVO MA 10 Carl Zeiss scanning electron microscope (SEM).

3. Results and discussion

3.1 Hardness and Charpy impact energy

After tempering of as quenched martensitic steel, the variation of toughness of the quenched and tempered grade steel with tempering temperature is shown in Figure 1. It indicates that the toughness increases as the tempering temperature increases. By increasing tempering temperatures from 500 to 680°C, the impact energy also increases except at 600°C. Initially the impact energy remains nearly constant on increasing temperature from 500 to 560°C while it substantially drops at 600°C. This may be attributed to temper embrittleness of P due to segregation of P to grain boundaries, which leads to decrease in grain boundary cohesion and hence decrease in Charpy impact energy. Phosphorus is considered as most dangerous surface-active impurity for materials used in nuclear reactor vessels. Presence of other elements determines the kinetics and thermodynamics of the grain-boundary segregation of phosphorus. For example, increasing Ni content from 1% to 2% in 0.02% P containing steel increases temper embrittlement temperature from 540°C to 580°C [14]. Therefore, as the present steel contains 1.62% Ni along with other elements, it may be influencing highest temper embrittlement of P at 600°C. Further increase in tempering temperature to 650ºC and 680ºC leads to increase in impact energy. This is because of extensive recovery and reduction of boundary regions along with substantial reduction in dislocation density in tempered structures [17]. However, the decrease in impact energy at 680°C for -20°C and -40°C is not clearly understood from this study.

Table 1. Chemical composition of quenched and tempered grade steel plate (wt.%).

C	Mn	P	S	Si	Cr	Mo	Ni	B	Al
0.225	1.06	0.011	0.005	0.29	0.75	0.42	1.62	0.0025	0.04

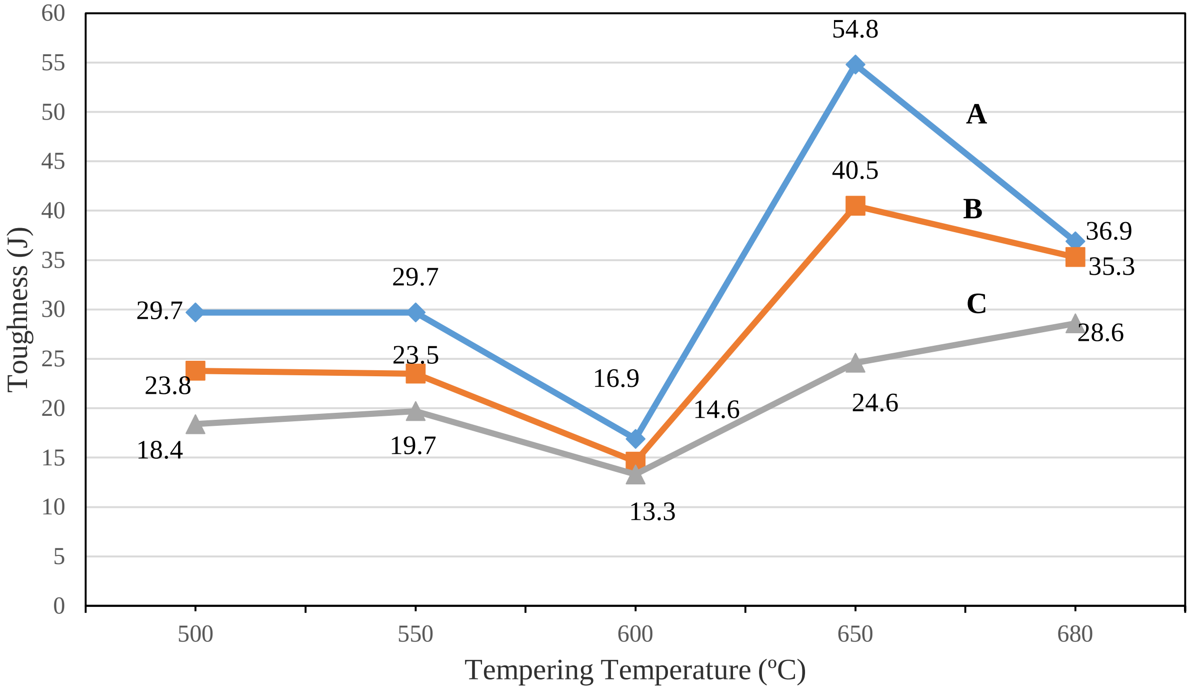

Figure 1. Relationship between toughness and tempering temperature of tested specimens (A= -20°C, B= -40°C and C= -60°C).

The embrittleness effect at 600°C has been further understood from the hardness measurement provided in Table 2. The table shows that hardness value has been significantly reduced at 650 and 680°C. This is because, the recovery is less at lower tempering temperature. The highest hardness at 600°C and 550°C is attributed to temper embrittleness effect of P. Further, the high hardness values along with low CVN energies at 550 and 600°C of this steel may be related to its temper embrittlement region. Further, the obtained hardness at 650°C was the requirement of the final steel plates to be produced commercially with impact energy >50 J. This may be considered as suitable temperature for obtaining best mechanical properties through industrial practice.

Therefore, based on the above finding of this laboratory study, industrial heat-treatment was carried out by austenizing the plates at 920°C and tempering at 650°C. The obtained results after Charpy impact test are shown in Table 3. It clearly shows that the impact energy of all of the specimens obtained after heat-treatment under industrial furnace is more than that obtained during laboratory condition. The one of the important factor for such increase in impact energy is austenite grain size, which depends on the austenization conditions like austenization temperature and soaking time. A high austenization temperature can alter the distribution of impurities in alloys steels. Also, at high temperature and more soaking time, austenite grain coarsening occurs. According to Hell-Petch equation [18], the impact energy depends on the prior austenite grain size. It improves with grain refinement. While the industrial heat-treatment with austenization was carried out at 920°C for 2 $\text{min}\cdot\text{mm}^{-1}$ for 300 mm thick slabs, the laboratory austenization in muffle furnace was carried out at 920°C for 2 $\text{min}\cdot\text{mm}^{-1}$but for 40 mm thick samples. As the sample size is different in these two conditions, the net soaking effect during laboratory study in the case of thinner sample (40 mm thick) is probably more, which resulted in higher austenite grain size and hence toughness decreased. Most importantly, the obtained hardness during laboratory tempering at 650°C is found to be similar to that obtained during industrial practice at same temperature as provided in Table 4. This precisely helped in lot of fuel saving, which was otherwise required to carry out the test directly in industrial furnace.

Table 2. Rockwell hardness of laboratory heat treated specimens after austenitized at 920°C for 2 min/mm and tempered at different temperatures for 4 min/mm.

Sample ID	Austenization temp. (°C)	Tempering temp. (°C)	Rockwell hardness, HRC	
			1	2
61	920	500	38	39
62	920	550	40	41
63	920	600	41	41
64	920	650	24	25

Table 3. Impact energy of industrially heat treated specimens after austenitized at 920°C and tempered at 650°C.

Specimen ID	Charpy temperature(^{0}C)	Impact energy(J)			
		1	2	3	Average (J)
R	-40	99.081	109.87	105.94	104.96
S	-40	112.45	107.98	120.97	113.8

Table 4. Rockwell hardness of industrially heat treated specimens after austenitized at 920°C and tempered at 650°C.

Sample ID	Average BHN	Rockwell hardness (HRc)
R	244	23.4
S	243.5	23.1

3.2 Microscopy

The light optical photo micrographs of tempered heat treated plates are shown in Figure 2. The microstructure consists of martensite laths and spherodized carbides of different orientation in the prior austenite grain boundaries as shown by arrow. However, the resolution of spheroidized carbide is not very clear in the light optical micrographs. This will be further examined using TEM in future study of this work. After water quenching, austenite has been completely transformed into marteniste. As shown in Figure 2 (a) to (d), the tempering leads to the decomposition of martensite laths. On increasing tempering temperature or during slower rate of tempering leads to the decomposition of martensitic lath into spheroidized carbide [17]. The effect of temperature has been demonstrated in Figure 2 (a) to (d). While the microstrusture in Figure 2 (a) consists of nearly all martensitic lath after tempering at 550°C, the carefully observation could reveal that the presence of decomposed laths of spheroidized carbide on increasing tempering temperature. The spheroidized carbide seems to be present in more amount in Figure 2 (c) and (d) as the specimens were tempered at higher temperature, i.e. 650°C and 680°C, respectively. This refinement in martensitic structure probably has resulted in the improvement in the toughness. Microstructure refinement is more effective in improving the resistance to the cleavage fracture than in increasing strength.

The fractured surfaces of specimens after Charpy impact tests are depicted in Figure 3. This was carried out to understand the mode of fracture initiation as a function of tempering temperature. Although some voids and dimples are observed in the fracture surface in Figure 3 (a), (b) and (c) for the specimens tempered at 500, 550 and 600°C, respectively, the presence of cleavage fracture can also be clearly noticed. On the other hand, the ‘dimple’ ruptures are clearly visible in Figure 3 (d), which was fractured after tempering at 650°C and that shows the tempered steel failed with a ductile fracture mode.

4. Conclusions

A high strength martensitic steel composition was designed for a commercial production purpose. The initial laboratory heat-treatment study revealed lowest CVN energy at 600°C, due to temper embrittlement of P at 600°C. Further, increase in tempering temperature to 650ºC and 680ºC lead to increase in impact energy and decrease in hardness value. Optical microscopy revealed that increasing tempering temperature leads to increase in the content of decomposed lath martensite. Fracture analysis indicated that fracture is predominately

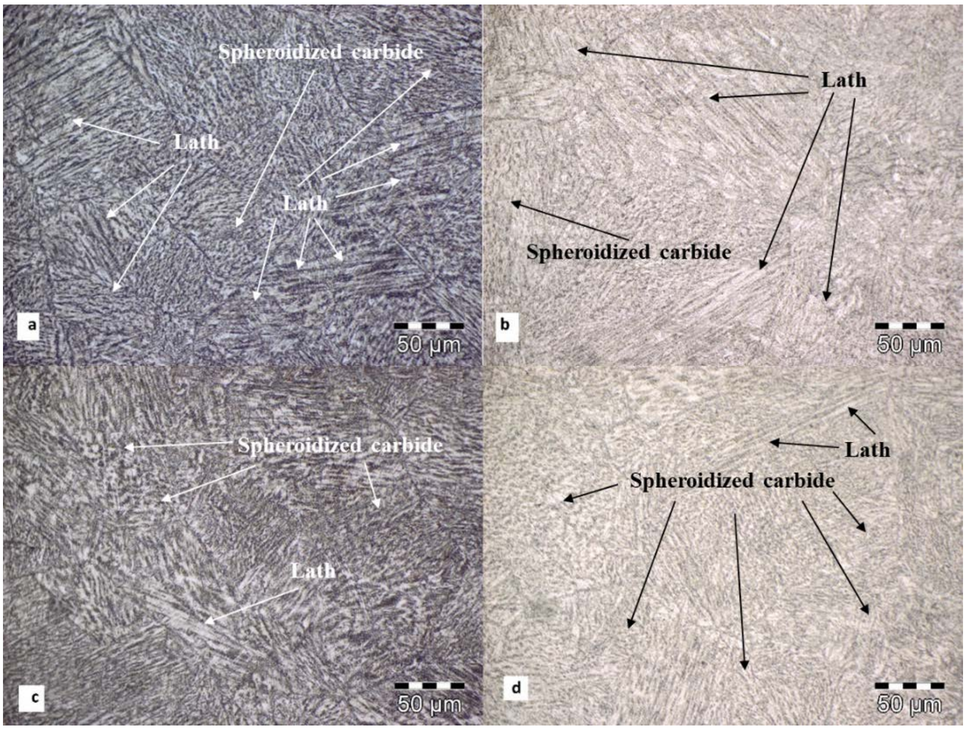

Figure 2. Optical micrographs of the specimens tempered at (a) 550°C, (b) 600°C, (c) 650°C, and (d) 680°C.

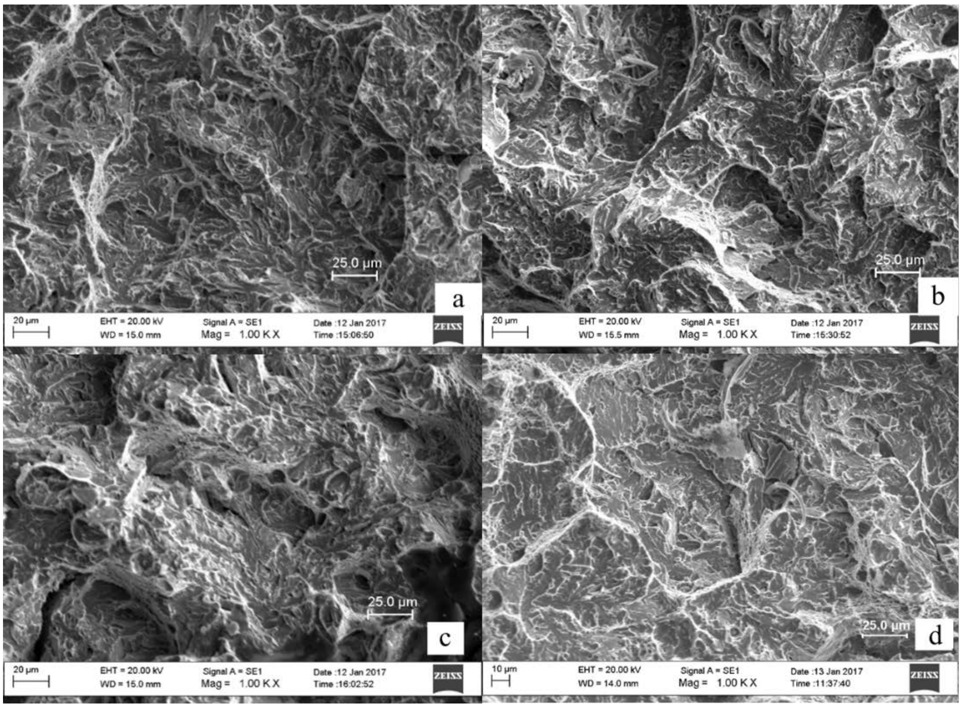

Figure 3. Fracture surface after impact testing of samples tempered at (a) 500°C, (b) 550°C, (c) 600°C, and (d) 650°C tested at -40°C.

ductile dimple rupture mode in the case of the sample tempered at 650°C. This temperature was selected for tempering of steel plates in industrial condition. Interestingly, further increase in toughness was obtained in the industrially produced steel tempered at 650°C.

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