Fractographic Analysis of Mechanical Properties of Microalloyed Steel

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Abstract: Extensive efforts made over the past few decades have enhanced the rising performance of High-Strength Low-Alloy steels. Use of thermomechanical processing was considered for this research. However, the desired mechanical properties are obtained by formulating alloys. Further, to enhance mechanical properties, impact energy, the subsequent quenching and tempering are used. The metallurgical transformation caused by deformation followed by cooling and/or heat treatment has added influences on steels’ mechanical properties. The rational decrease in impact energy value is complex.

Keywords: Metallurgy, Microstructure, Microalloyed steels, Mechanical Properties, Quenching and Aging, SEM

1. Introduction

High-Strength Low-Alloy (HSLA) steels are a special class of low alloy steels that develop better mechanical properties and higher atmospheric corrosion resistance through their chemical composition. HSLA steels are generally produced with a focus for obtaining mechanical properties rather than restriction of chemical compositions. Due to dramatic increase in the consumption of oil and gas for transmission and their production in the recent decades resulted in the use of pipelines [1-4]. Besides, various mining companies adopted the pipelines to transport iron ore to decrease transportation cost over large distances. To fulfil this requirement, the pipes used need to be of larger diameters and also withstand high pressures. HSLA steels’ development avoids requiring excessive wall thickness and therefore has transformed the pipeline projects. This development significantly contributes to the reduction of project cost [5-9].

Over the last five decades, steels' strength, toughness and weldability have been enhanced by new processing routes and alloy design [10-12]. During the conditioning at a low temperature such as during cooling, particles are formed in ferrite. Some particles are formed in austenite and α-γ interface during transformation. An optimum combination of mechanical properties was achieved with the dislocation-precipitate interaction. The studies on the cold forming and ageing behaviour of line pipe HSLA steels have yielded conflicting results due to difficulties faced owing to high dislocation density existing in the solution treated matrix [12-14]. The effect of both precipitates and dislocations on X65 HSLA steel’s ageing behaviour has been studied in the present investigation. Charpy V-notch (CVN) impact energy has been used for the measurement of mechanical properties. Light Microscopy and SEM have been used for microstructural characterization. The article's main highlights are:

a. Cold and hot working by rolling causes strengthening in the given steel via strain hardening.
b. TMT treatments results in the variation of mechanical properties.
c. The treatment involving 35% deformation results in optimum impact energy values in the PA condition.

2. Materials and Methods

2.1 Alloy Composition
Sheets of commercial HSLA X-65-line pipe steel [composition (wt.%): C-0.0616 S-0.0046 P-0.0046 Mn-0.001 Si-0.04 Cu-0.045 Cr-0.09 Ni-0.24 Nb-0.234 Ti-0.23 V-0.34 N-0.67] in the form of a sheet with dimension 300 mm × 300 mm × 24 mm was procured.

2.2 Treatments and Mechanical Properties
The steel was subjected to various thermal and mechanical treatments. In its "As Received" (AR) condition, the X65 line pipe steel was given the following treatments at various time ranges. Firstly, AR material was austenitised for 180 min at 1000°C followed by oil quenching (OQ). The OQ treated material for various time ranges of 5 - 1500 min was aged at 700°C followed by water quenching. Following oil quenching, the OQ material, was for various time ranges from 5 - 1500 min, aged at 700°C and quenched in water. The material was then tempered at 700°C for 120 min and again quenched in water. Then it was cold-rolled to reduce its thickness by 35%, 50% and 65%. The steel was then finally aged at 400°C from 5 - 3000 min followed by water quenching. For each treatment 3 samples were used for comparison.

As per ASTM E23 specification, the specimens for Standard Charpy V-notch were prepared. These specimens were subjected to Charpy V-Notch Impact tests for evaluating its mechanical properties. Impact test results on AR, oil quenched (OQ), quench tempered and aged (QTA), cold worked (CW), peak aged (PA) and over-aged (OA) specimens were obtained at room temperature.

3. Results and Discussions

3.1. Impact Energy
To determine the impact energy Charpy-V test was conducted. Impact energy was measured as the energy absorbed up to the fracture under impact loading.

Impact energy is the energy absorbed upto the fracture under impact loading.

- A marginal change in impact energy was observed after solution treatment at 1000°C.
  - After tempering at 700°C and subsequent ageing at 400°C there was a significant improvement in the steel's impact toughness.
  - However, ageing after tempering, marginally decreased the values of impact energy.

- It was observed that the impact energy initially got enhanced with the increase in the degree of deformation i.e., 50% thickness reduction by cold working. However, further increase in the degree of the deformation, deteriorated the impact energy.
  - The optimum value of 178J was with 50% thickness reduction by cold working.
  - The minimum value of 47J was with 65% thickness reduction by cold working.

- The impact energy decreased with increase in the degree of deformation in PA (peak-aged) condition.
  - 35% thickness reduction by cold working resulted in optimum impact energy value of 224J.
  - 65% thickness reduction by cold working, exhibited the minimum impact energy value of 50J in PA condition.

3.2. Characterization
The steel reveals in As-Received (AR) condition shows ferrite-martensite microstructure. The existence of flow lines reveals that the steel might have been given cold/hot rolling treatment shown in Figure 1(A). The solution treatment at 900°C results as shown in Figure 1(B) in predominantly ferrite together with martensitic needles which subsequently transforms to acicular ferrite at 1000°C. It is also observed that austenitization at 1000°C causes the formation of few carbide particles within and along the “grain boundaries” (Figure 1(C)). However, austenitization at 1100°C results in further growth of ferrite grains.
and carbide particles as shown in Figure 1(D). It is also seen that the higher austenitization temperatures results in coarse grain boundaries.

In this work microstructural transformations of X65 HSLA steels after tempering at 500°C, 600°C and 700°C respectively have been examined and it has been observed that tempering at 700°C leads to tempering of martensite together with larger ferrite grains as shown in Figure 2(a). Further, the results are examined after the deformation in cold working at 35%, 50% and 65% and it has been observed with optical micrographs after 35%, 50% and 65% deformation that ferrite grains are strained primarily in the direction of rolling, predominantly ferritic microstructure together with coarse precipitate particles along the grain boundaries and severe deformation to 65% results in further fragmentation of grain boundaries respectively as shown in Figure 2 (b-d).

Figure 1 (A-D): Optical micrographs showing microstructures of X65 HSLA steel

Ageing, in general, is accompanied with simultaneous diffusion and nucleation and growth processes. In this work, ageing results in the formation of fine precipitate particles after 5 min. Subsequent ageing causes the grain coarsening, which is accompanied with the segregation of precipitate particles along the “grain boundaries” and coarsening of precipitate particles. Further, ageing lead to grain refinement and polygonization of ferrite up to 1500 min. However, prolonged ageing at 3000 min again results in the coarsening of grain and pre-existed precipitate particles as well.
The optical micrographs in various stages of ageing at 400°C after 35% deformations. In this work ageing after cold working initially causes the recrystallization after 5 min. However, ageing up to 15 min results in the grain coarsening with sharp grain boundaries and then up to 30 min results in the formation of coaxial grain. The precipitation occurs either with the formation of new particles or coarsening of pre-existing particles at 300, 600, 1500 min respectively (Figures 3(i)).

The optical microscopic examination after 50% cold work reveal that ageing initially causes the recrystallization and segregation of coarse carbide particles along the grain boundaries. A few coarse precipitate particles are also seen within the grains after 5 min and 15 min. Subsequent ageing to 30 min results in a uniform distribution of fine precipitate particles. Progressive ageing to 300 min again results in grain growth and coarsening of precipitate particles within ferrite matrix. However, ageing to 600 min involves the grain refinement and formation of fine precipitate particles.

Figure 2 (a-d): Optical micrographs showing microstructures of X-65 HSLA steel

Figure 3(ii) shows the optical micrographs after 1500 and 3000 min. It is observed that prolonged ageing again follow the coarsening and growth of grains and precipitate particles.

The substantial optical microscopic examinations were directed in various stages of ageing after 65% cold work. Ageing in initial stages results in the recrystallisation and precipitation. Ageing also results in uniform distribution of fine precipitate particles up to 60 min. However, subsequent ageing causes the
coarsening of precipitate particles after 600 min. Prolonged ageing to 3000 min is accompanied by the retention of cold rolling texture together with existence of high density of fine precipitate particles (Figure 3 (iii & iv)).

![Micrographs showing microstructural changes on ageing at 400°C](image)

**Figure 3 (i-iv):** Optical micrographs showing microstructural changes on ageing at 400°C

4. **Fractography**
Scanning Electron Microscopic examinations of the fractured surfaces of impact (Charpy-V) illustrated the features, which are closely correlated with impact energy of the steel under investigation. SEM study of impact fractured surfaces revealed the networks of fine/coarse dimples accompanied with precipitate nucleate voids. High impact energy values were associated with a network of uniformly distributed strained dimples. However, the low impact energy values were manifested by the network of coarse dimples and high density of coarse/fine voids.

4.1 **Impact Behavior**
It is manifested that the fracture is associated with the formation of coarse/fine dimple networks and coarse voids nucleated around the precipitate particles. Figure 4(A) represents the micrographs in AR condition. The high impact energy in AR condition results in fine network of coaxial elongated dimples. This reflects the ductile nature of failure in AR condition. The SEM micrographs in OQ condition represents that the fracture is accompanied with the growth of void (Figure 4(B)). A few coarse precipitate particles are also observed in OQ condition. Tempering results in the enhanced impact energy values in X65 HSLA. Accordingly, a fine
distribution of dimples is observed in QT condition (Figure 4(C)). The initial decrease in impact energy after 120 min of ageing is manifested by coarse dimples (Figure 4(D)).

Figure 4 (A-D): Scanning electron micrographs of impact fractured surface

Figure 5(a-d) is displaying the various results in different condition at 400°C for 3000 min. The relative improvements in impact energy after ageing are reflected by a network of fine dimples and fine void (Figures 5(a)) in over aged condition of ageing after quenching at 1000°C and tempering at 700°C.

Figure 5(b) reflects the effect of cold work and subsequent ageing on the impact behavior of X65 HSLA steels in over aged condition after 35pct cold work. It has been observed that various degrees of cold work cause the loss of impact energy. The loss of impact energy after various degrees of deformation is manifested by the existence of coarse dimple network with the presence of larger density of coarse and fine voids, which increases with increasing degree of deformation in peak aged condition after 50pct and in over aged conditions after 65pct (Figure 5(c & d)).

However, the increments in impact energy on ageing to peak condition after various degrees of deformations are manifested by the existence of network of elongated dimples (Figure 5(b)) as compared to that in CW condition. Moreover, the decrease in impact energy in PA conditions with increasing deformations is manifested by the cleavage or faceted network of dimples (Figure 5(c)).
However, the PA condition of TC65A treatments shows marginal increase in impact energy as compared to the corresponding value for CW condition. But this value is significantly lower than the corresponding impact energy value for 35% and 50% cold work. The SEM micrographs in PA condition (Figure 5(d)) of TC65A treatment exhibit networks of coarse dimples together with the large number of coarse precipitate voids. The existence of coarse voids at precipitate sites manifests the loss of impact energy after PA condition of TC65A treatment. Further decrease in impact energy on prolonged ageing (over ageing) in TC35A (Figure 5(b)) and TC50A (Figure 5(c)) treatments are manifested by the coarse network of dimples and relatively larger voids. However, over ageing condition of TC65A treatment results in marginal increase in the impact value. The marginal increase in over aged condition of TC65A treatment is reflected by relatively fine distribution of dimple network (Figure 5(d)).

5. Conclusions
The external forms of commercial steel products result from the hot/cold deformations, like rolling. However, the alloy design and thermal processing after deformations can achieve the desired mechanical properties. Furthermore, quenching and subsequent tempering also result in improved mechanical properties. The metallurgical transformation caused by deformations influences steels' mechanical properties followed by cooling or final heat treatment. Solution treatments involving the temperature range of 900°C -1100°C results in the hardening of microalloyed X65 HSLA steels. The hardening in this steel
is associated with microstructural transformation in the austenite range. The study reveals that solution treatments of microalloyed X65 HSLA steel results in the recrystallisation and coarse precipitates particles within the ferrite grain and along the grain boundaries.

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