Production of gauge 1.25Cr-0.5Mo and carbide distribution in heat treatment structures

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Abstract: In order to research into the structural features of heat treated gauge 1 1/4Cr-1/2Mo steel, relevant constituents are designed and smelted, and tests inclusive of heating, rolling, heat treating and PWHT are conducted. The tested steel plate resists the relevant processes, PWHT test and step cooling. Handled in different temperatures, the steel plate demonstrates regularity in its carbide distribution formed in corresponding heat treated structures.

1. Introduction
Cr-Mo steel applied in petrochemical industry and heat-engine plants permanently confronts great heat, high pressure and hydro attack. Composed of relatively high amount of alloy, Cr-Mo steel can produce bainite structure in air cooling conditions. As a heat-resisting metal, high temperature-chemical stability and mechanical stability are its most basic features. Gauge steel plates are characterized by weightiness and thickness, which may well lead to uneven temperature in cross section or a lack of synchronization of structural shift. Based on pressure and temperature, current hydrotreater adds to the tempering embrittlement of 2 1/4Cr-1Mo steel as amount of Cr and Mo increase, and in long term brittleness and material malleability decrease inextricably surfaces. Research shows that tempering embrittlement is fundamentally when alloy steel was being tempered or subsequently being cooled the gathering of inimical elements like P, Sn, Sb, As in austenite matrix which undermines the cohesive force of matrix in grain boundary. When tension or shock applies, grain boundary is subject to breakage and causes Intergranular fracture [1-7]. In consequence, the study of gauge and high-alloy1 1/4Cr-1/2Mo steel’s heat treatment process and uniformity plays a vital role in material’s long-term performance.

2. Experiment method
(1) The smelting of molten steel
The hydrotreater was designed in line with the chemical composition of 1 1/4Cr-0.5Mo steel plate. Our major concern touches upon the following points: Firstly, the impurity elements including P, Sb, Sn, As. Secondly, the content of Mn is around 0.5%, considering Mn’s contribution to overall intensity.

| Specimen | C% | Si% | Mn% | P%≤ | S%≤ | Alt% | Cr% | Mo% | Sb | Sn | As | J | X |
|----------|----|-----|-----|-----|-----|------|-----|-----|----|----|----|---|---|
| 1        | 0.14 | 0.55 | 0.49 | 0.007 | 0.002 | 0.034 | 1.21 | 0.498 | - | - | 0.00 | 6 | 71.8 | 7.4 |
| 2        | 0.12 | 0.56 | 0.45 | 0.007 | 0.002 | 0.034 | 1.18 | 0.486 | 0.00 | 0.00 | 0.00 | 9 | 87.9 | 9.5 |

Molten iron desulphurization technique and converter double slag technique were employed to get...
rid of S and P. Deoxidation alloying was conducted out of converter. S was further cleared through LF refining process and H through RH process. Heavy plate casting followed.

(2) Heating method

Temperature control within the heating furnace kept the tapping temperature of the steel plate. The steel plate here is 400mm thick. The heating duration is illustrated in the following table.

| Cold billet thickness/mm | Heating duration/min |
|-------------------------|----------------------|
| 250                     | 250–300              |
| 300                     | 300–450              |
| 400                     | 400–500              |

(3) Rolling of steel plates

For hydroformed 1 1/4Cr-1/2Mo steel plates thinner than 90mm, normally hot rolling is preferred. Rolling passes were not separated and in rough rolling and pass reduction was heightened. In finish rolling multiple methods were available in rolling shape control, leaving no holding time between passes. The finish cooling temperature was around 650℃. For hydroformed 1 1/4Cr-1/2Mo steel plates thicker than 90mm, rolling passes were not separated and in rough rolling, and its finish cooling temperature was around 670℃.

(4) Heat treatment process

Hydroformed 1 1/4Cr-1/2Mo steel plates thicker than 90mm were normalized in our effort to retrieve bainite structure, with normalization temperature fixed at 930℃. Corresponding holding time was 60±1min after plate core are heated to expected temperature. Tempering temperature was initially settled at 700℃.

3. Results and discussion

3.1 composition design

(1) Impact of Mn

Like Si, Mn can drive tempering embrittlement. That said, Mn is different from Si as it exerts bigger influence on steel’s hardenability. When hardenability is required and ferrite is undesirable, the amount of Mn should remain low but not too low.

(2) Impact of Sn, Sb, P and As

Sn, Sb, P and As are all impurity elements that cause marked embrittlement, therefore should be kept in low levels in smelting. Besides, the brittleness sensitivity coefficient X (X = (10P+5Sb+4Sn+As)*10^{-2}≤15ppm) according as both national and American standards is supposed to be minimum, and this echoes with the need to be technically curbed.

(3) Impact of Cr and Mo

Cr gives a evident lift of steel’s high temperature performance by improving steel’s anti-oxidation, which prevents graphitization, and corrosion resistance (hydro attack). Properly heated, Cr-Mo steel can acquire tensile strength and comprehensive mechanical properties. But as Cr increases, susceptibility to tempering embrittlement will soar; especially when Cr accounts for 2.0~3.0%, susceptibility to embrittlement is higher.

Mo is the pivotal source of steel’s high temperature strength. With its strong tendency to form carbide, Mo can prevent pearlite transformation and make steel astoundingly subject to bainite, thereby promotes the comprehensive mechanic performance of Cr-Mo steel. In the meantime, Mo can make steel tempering more stable, eliminate or reduce tempering embrittlement of Cr-containing steel.

3.2 Rolling process

95mm thick hydroformed 1.25Cr-0.5Mo steel mainly adopted direct converter steelmaking, at the
same time combining rough rolling of double passes width spread and longitudinal rolling. The pass reduction and pass reduction rate is illustrated in figure 1. From figure 1 we can note that rough rolling of steel plate remained steady, with 4-5 pass reduction rates above 35mm each, while the maximum pass reduction rate nearing 25%. The innermost part of the thick plate was sufficiently deformed, thereby adding to the mechanic homogeneity in the thickness of steel plate, and thus the great level of stability in the rolling process.

![95mm-sized plate’s pass reduction](image1)

**Figure 1** distribution of pass reduction and corresponding rate of 95mm thick steel

60mm thick hydroreformed 1.25Cr-0.5Mo steel mainly adopted forming pass plus 6-9 pass width spread plus longitudinal rolling, whereas 2 steel plates adopted forming pass and full cross-rolling technique, whose impact on performance pending further research. As shown in the following Figure 2, the overall pass reduction stood fewer than 30mm, and the low level was due to the a lengthier width of the steel plate. That means pass reduction concentrated at the width spread passes, and after longitudinal rolling the rolling force and torque were relatively high, thus a low level of pass reduction. The width of the steel is a major reason for a meager single-pass reduction and reduction rate in rough rolling.

![60mm-sized plate’s pass reduction](image2)

**Figure 2** distribution of pass reduction and pass reduction rate of 60mm thick steel

### 3.3 Heat treatment process

For 60-95mm thick hydroreformed steel plate, the normalizing temperature was $930\pm 10^\circ C$, holding time afterwards was 95min. Chilled water flow rate following normalization was shown in Figure 3.

After weak cooling water treatment, plate shape of 95mm thick plate remained steady. The self-tempering temperature ranged between 160°C and 180°C. Temperature difference of the whole plate was less than 20°C, reaching the foreseen goal.

| Specimen | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | Roll speed m/min |
|----------|---|---|---|---|---|---|---|---|---|----|------------------|
| 60mm     | 120| 485| 120| 200| 580| 200| 90 | 325| 90 | 81 | 14               |

![Table 3 Amount of water used on 60-95mm sized hydroreformed steel plate](image3)

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3.4 tempering performance and carbide distribution

95 mm thick hydroreformed 1.25Cr-0.5Mo steel plate has gone through post-tempering performance test, as shown in Figure 4. Holding post-tempering mechanic parameters held as a reference, we can see that all mechanic performance fits into requirements of the protocol, and residual amount of intensity created room for the improvement of mechanic performance for Max.PWHT and Min.PWHT.

| specimen | Rel /MPa | Rm /MPa | A /% | Z /% | Shock temperature | Impact energy/J | cold bending d=3a |
|----------|----------|----------|------|------|-------------------|----------------|-----------------|
| 1        | 443      | 589      | 27.5 | 72.2 | -20℃              | 169 195        | 241 up to par   |
| 2        | 436      | 584      | 25.5 | 65.1 | -20℃              | 245 239        | 261 up to par   |
| 3        | 440      | 580      | 27.0 | 72.1 | -20℃              | 177 162        | 220 up to par   |
| 4        | 448      | 597      | 26.5 | 62.1 | -20℃              | 177 172        | 180 up to par   |
| 5        | 448      | 592      | 23.5 | 63.5 | -20℃              | 179 220        | 229 up to par   |
| 6        | 435      | 590      | 25.5 | 62.7 | -20℃              | 198 214        | 213 up to par   |
| 7        | 438      | 586      | 23.5 | 65.0 | -20℃              | 105 188        | 114 up to par   |
| 8        | 444      | 590      | 26.5 | 66.2 | -20℃              | 205 249        | 186 up to par   |

Hydroreformed 1.25Cr-0.5Mo steel plates turned predominantly lath bainite after being tempered in 700℃, and the carbide mostly took on beam current distribution in the form of bainite. Part of the carbide was rod-shaped about 1μm long, part sphere-shaped with a diameter between 200nm and 500nm, and some other smaller sphere-shaped carbide distributed therein.

![Figure 3](image-url) post-tempering carbide distribution of hydroreformed 1.25Cr-0.5Mo steel

EDS analysis indicates that the majority of the rod-shaped carbide was alloy cementite and the sphere-shaped carbide was carbide of Cr, which was inferred to be Cr7C3, pending TEM phase arrangement differentiation. The still less sizable sphere-shaped carbide was too limited in size for EDS to yield a report of its composition, thereby pending TEM phase arrangement differentiation. On austenite matrix mainly distributed alloy cementite as shown in Figure 4.
Table 5 Composition of each point position

|    | C   | Si  | Cr  | Mn  | Fe  |
|----|-----|-----|-----|-----|-----|
| 1  | 7.38| 0.52| 2.30|     | 89.79|
| 2  | 6.31| 0.65| 1.58|     | 91.46|
| 3  | 6.66| 0.57| 1.58|     | 91.19|
| 4  | 11.35| 0.61| 4.75|     | 83.29|
| 5  | 3.98| 0.69| 1.54|     | 93.79|
| 6  | 3.82| 0.61| 1.56|     | 94.01|
| 7  | 13.45| 0.60| 7.01| 1.37| 77.57|
| 8  | 7.61|     | 2.62|     | 89.77|
| 9  | 9.72| 0.72| 3.61|     | 85.99|
| 10 | 4.46|     | 1.66|     | 93.88|
| 11 | 10.90| 0.82| 3.14|     | 85.14|

3.5 Post-PWHT carbide morphological distribution

3.5.1 Min.PWHT Following 700°C tempering of 1.25Cr-0.5Mo steel, the post-Min.PWHT carbide morphological distribution took on the form shown in Figure 5. Compared with conditions during tempering, the 1.25Cr-0.5Mo steel tempered in 700°C demonstrated such features after Min.PWHT procession: Alloy cementite disappeared in huge amount while Cr’s carbide rose in proportion; in some spots Mo’s carbide together with MnS precipitated, though in meager amount.

Figure 5 The carbide’s post-Min.PWHT morphological features for hydroreformed steel tempered in 700°C

3.5.2 Max.PWHT Following 730°C tempering of 1.25Cr-0.5Mo steel, the post-Min.PWHT carbide morphological distribution took on the form shown in Figure 6. Post-Min.PWHT conditions didn’t vary much from tempering conditions, i.e. Cr’s carbide remained the bulk.
Following 700°C tempering of 1.25Cr-0.5Mo steel, the post-step-cooling carbide morphological distribution took on the form shown in Figure 7. The result after step cooling resembles that after Min.PWHT, as sphere-shaped Cr’s carbide still took the majority. As far as EDS’s results are concerned, some spots yielded composite carbide of Cr and Mo, though in meager proportion. Stable carbide distribution both prior to and after the step cooling contributes to lower level of ∆vTr55, bolstering post-tempering anti-brittleness performance of steel plates that are to be hydroreformed.

Following 730°C tempering of 1.25Cr-0.5Mo steel, the post-step-cooling carbide morphological distribution resembles that following Min PWHT procession, as sphere-shaped Cr carbide dominated in amount. Though some spots yielded Mo’s carbide, it was low in proportion. Stable carbide distribution both prior to and after the step cooling contributes to lower level of ∆vTr55, bolstering post-tempering anti-brittleness performance of steel plates that are to be hydroreformed.

For tempered 1.25Cr-0.5Mo steel, the post-step-cooling carbide morphological distribution was characterized by higher proportion of pearlite. Within ferrite matrix, carbide’s majority was still Cr’s
carbide, whose formation needs to be observed and analyzed through TEM.

Figure 9 The carbide’s post-step-cooling morphological features for hydroreformed steel tempered in 760 °C

Following 700 °C and 730 °C tempering of 1.25Cr-0.5Mo steel, the post-step-cooling carbide morphological size, distribution and positions largely remained the same, with its bulk Cr’s carbide. Following 760 °C tempering of 1.25Cr-0.5Mo steel, the post-step-cooling carbide demonstrated higher proportions of pearlite, formation mechanism of which pending TEM analysis.

4. Conclusion
1) The smelting of steel plate met set standards of the test, and both coefficient J,X met the requirements.
2) After tempering, all mechanic performance all fitted before-set requirements. Meanwhile the residual amount of intensity was relatively high thereby created room for the improvement of mechanic performance for Max.PWHT and Min.PWHT.
3) Compared with tempering conditions, post Min.PWHT features disappearance of alloying cementite and increase of Cr’s carbide in proportion; in some spots Mo’s carbide together with MnS precipitated, though in meager amount.
4) Following 700 °C and 730 °C tempering of 1.25Cr-0.5Mo steel, the post-step-cooling carbide morphological size, distribution and positions largely remained the same, with its bulk Cr’s carbide; post 760 °C tempering the steel yielded greater amount of pearlite.

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