Effects of Normalizing Processes on Microstructure and Impact Toughness in Ti-bearing Weld Metal of Multilayer MAG Welded HSLA Steel

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The influences of different normalizing heat treatments on microstructure, non-metallic inclusions and impact toughness in MAG Ti-bearing weld metal of HSLA steel has been studied. It has been shown that for the Ti-bearing weld metal the impact toughness after normalizing treatment decreases significantly against prolonging holding time and increasing normalizing temperature. The Mn-depleted zone forms around the Ti-bearing phase (MnTiO$_3$) precipitated on Mn–Si oxide. Proeutectoid ferrite preferentially nucleates at the Mn-depleted zone and the interface between austenite and proeutectoid ferrite becomes the nucleation sites for pearlite thereafter. Mn-depleted zone formation increases the pearlite nucleation sites, and makes the pearlite fine. The dissolve of Ti-bearing precipitate causes disappear of Mn-depleted zone at strong normalizing processes (longer normalizing time and higher normalizing temperature), and the number of ferrite nucleation sites decreases, then the pearlite become coarser, which causes the deterioration of impact toughness.

KEY WORDS: normalizing process; weld metal; impact toughness; Ti-bearing precipitate.

1. Introduction

Ti has a great affinity for carbon atom, nitrogen atom and oxygen atom, therefore, Ti carbides, Ti nitrides, Ti carbon-nitrides and Ti oxides can precipitate easily in steel. These precipitates have great influence on the mechanical properties and microstructure of steels. The calculation of solubility products has shown that the consequence of these phases is Ti oxides, Ti nitrides and Ti carbides. However, the final precipitates depend on the processing the material undergone and the other microalloy elements, such as Nb and V. There will be Ti-bearing oxides in a weld metal by manual metal arc welding (MMAW), submerged arc welding (SAW), metal active gas welding (MAGW) and in Mn–Si–Ti deoxidized steel. Simulation results show that few Ti nitrides precipitate in the Heat-Affected Zone (HAZ). The Ti-bearing complex carbonitrides can pin and refine the grain, and the Ti-bearing oxides have an effect on the microstructure, especially for acicular ferrite formation, during the transformation. However, the behavior and effect of Ti-bearing oxides in weld metal on the microstructure and property after heat treatment is seldom reported.

In the present study, the effect of normalizing heat treatment on the behavior of Ti-bearing oxides and weld metal microstructure and impact toughness has been investigated and discussed.

2. Experimental Procedures

Two kinds of low carbon steel welding wires with a diameter of 1.0 mm were used in the experiment to weld a 12 mm thick S355J2W (base metal) steel plate by the metal active gas (MAG) welding. The geometry of the joint was Y groove with the 60° groove angle. The joint was welded by 4 welding layers and the shielding gas was 82%Ar+18%CO$_2$. The schematic diagrams of the test plate and groove type were shown in Fig. 1. Table 1 gave the chemical compositions of the welding wires and the base metal. The chemical compositions of the weld metals were analyzed and shown in Table 2. Since there was CO$_2$ in the shielding gas, nearly 60% Ti was burnt in the welding process, and the Ti content in the weld metal was lower than that in the welding wire. Therefore, the correspondent weld metals welded by welding wire 1 and 2 were defined as the weld metal without Ti and Ti-bearing weld metal, respectively. The mechanical properties of the steel plate were shown in Table 3.

The heat treatment parameters for the welded joint were shown in Table 4. Specimens for the impact property test of the weld metal were extracted from these joints transversely to the welding direction using an electrical spark wire cutting machine and were machined to a dimension of 55 mm×10 mm×10 mm. The V notch was machined in the center of the weld metal on the cross section of the joints. The impact tests were examined on the swing impacting test

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The electrolytic etch was 10% HClO₄ + alcohol. Then were prepared by using twin-jet machine Struers Tenupol-5 “wet-dry” paper to P1500 grade. The thin foil specimens were prepared from small 3 mm diameter cylinders cut from the weld metal. The cylinders were then sliced to a thickness of 0.5 mm, using an electrical spark wire cutting machine at –20°C. The thickness was reduced to 90 μm by grinding, using μm and weld metal without Ti addition. When the normalizing, the columnar grain zone disappeared and the primary ferrite (PF) that includes both grain boundary and intragranular polygonal ferrite, ferrite with second phase (FS) that contains all grain boundaries nucleated lath-like morphology of ferrite and intragranular acicular ferrite (AF). The microstructure in FGZ is polygonal ferrite. The AF can increase the weld metal impact property. It is also shown that with small addition of Ti the size of the PF in the CGZ is much finer and the acicular ferrite is well-developed, as shown in Fig. 3(b). The size of polygonal ferrite in the FGZ for Ti-bearing weld metal is a little smaller, as shown in Fig. 3(d). Therefore, the impact property for the Ti-bearing weld metal in as weld condition is better than that of weld metal without Ti.

3. Results and Discussion

3.1. Impact Property and Microstructure of the Weld Metal in as Weld Condition

The weld metal impact energy at –20°C in as weld condition are 119 J and 143 J for weld metals without Ti and with Ti bearing, separately. 13 Since multi-pass welding (4 layers) is applied in the experiment as shown in Fig. 1, the weld metal microstructure composed of columnar grain zone (CGZ) of the last weld layer and fine grain zone (FGZ) for the first three layers, as shown in Fig. 2. The microstructures of the weld metal are shown in Fig. 3. The microstructure in CGZ is subdivided into three constituents, i.e., primary ferrite (PF) that includes both grain boundary and intragranular polygonal ferrite, ferrite with second phase (FS) that contains all grain boundaries nucleated lath-like morphology of ferrite and intragranular acicular ferrite (AF). The microstructure in FGZ is polygonal ferrite. The AF can increase the weld metal impact property. It is also shown that with small addition of Ti the size of the PF in the CGZ is much finer and the acicular ferrite is well-developed, as shown in Fig. 3(b). The size of polygonal ferrite in the FGZ for Ti-bearing weld metal is a little smaller, as shown in Fig. 3(d). Therefore, the impact property for the Ti-bearing weld metal in as weld condition is better than that of weld metal without Ti.

3.2. The Effect of Normalizing Processes on the Toughness of the Weld Metal

The impact energy of the Ti-bearing weld metal decreases against increasing temperature, as shown in Fig. 4. After normalizing, the columnar grain zone disappeared and the impact property improved both for the Ti-bearing weld metal and weld metal without Ti addition. When the normalizing temperature is 1100°C, the impact energy of the Ti-bearing weld metal and the weld metal without Ti is nearly the same. It also can be shown that the normalizing temperature has significant effect on the Ti-bearing weld metal. The impact property of the weld metal against prolonging holding time at 900°C is shown in Fig. 5. Results showed that the impact property of the Ti-bearing weld metal decreases significantly against prolonging holding time

### Table 1. Chemical compositions of base metal and welding wires (wt.%)

<table>
<thead>
<tr>
<th>Material</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>S</th>
<th>P</th>
<th>Cu</th>
<th>Nb</th>
<th>Cr</th>
<th>Ni</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base metal</td>
<td>0.067</td>
<td>1.32</td>
<td>0.002</td>
<td>0.019</td>
<td>0.41</td>
<td>0.21</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Welding wire 1</td>
<td>0.040</td>
<td>0.41</td>
<td>1.09</td>
<td>0.0034</td>
<td>0.010</td>
<td>0.48</td>
<td>0.01</td>
<td>0.87</td>
<td>0.021</td>
<td></td>
</tr>
<tr>
<td>Welding wire 2</td>
<td>0.048</td>
<td>0.40</td>
<td>1.09</td>
<td>0.0043</td>
<td>0.011</td>
<td>0.45</td>
<td>0.01</td>
<td>0.85</td>
<td>0.074</td>
<td></td>
</tr>
</tbody>
</table>

### Table 2. Chemical compositions of weld metal (wt.%).

<table>
<thead>
<tr>
<th>Material</th>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Ni</th>
<th>Cr</th>
<th>Ti</th>
<th>Cu</th>
<th>N</th>
</tr>
</thead>
<tbody>
<tr>
<td>Without Ti</td>
<td>0.045</td>
<td>0.39</td>
<td>1.11</td>
<td>0.010</td>
<td>0.007</td>
<td>0.84</td>
<td>&lt;0.05</td>
<td>&lt;0.01</td>
<td>0.41</td>
<td>0.0031</td>
</tr>
<tr>
<td>Ti-bearing</td>
<td>0.051</td>
<td>0.36</td>
<td>1.07</td>
<td>0.009</td>
<td>0.008</td>
<td>0.83</td>
<td>&lt;0.05</td>
<td>0.028</td>
<td>0.41</td>
<td>0.0034</td>
</tr>
</tbody>
</table>

### Table 3. Mechanical properties of the base metal.

<table>
<thead>
<tr>
<th>Yield strength/MPa</th>
<th>Tensile strength/MPa</th>
<th>Elongation/%</th>
<th>–20°C impact energy/J</th>
</tr>
</thead>
<tbody>
<tr>
<td>370</td>
<td>505</td>
<td>30.0</td>
<td>175</td>
</tr>
</tbody>
</table>

### Table 4. Normalizing processes of the joints.

<table>
<thead>
<tr>
<th>Number</th>
<th>Heat treatment processes</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Normalizing at 900°C for 1 h</td>
</tr>
<tr>
<td>2</td>
<td>Normalizing at 1000°C for 1 h</td>
</tr>
<tr>
<td>3</td>
<td>Normalizing at 1100°C for 1 h</td>
</tr>
<tr>
<td>4</td>
<td>Normalizing at 1200°C for 1 h</td>
</tr>
<tr>
<td>5</td>
<td>Normalizing at 900°C for 0.5 h</td>
</tr>
<tr>
<td>6</td>
<td>Normalizing at 900°C for 2 h</td>
</tr>
<tr>
<td>7</td>
<td>Normalizing at 900°C for 5 h</td>
</tr>
</tbody>
</table>
while the impact property of the weld metal without Ti does not change obviously, as shown in Fig. 5. When the holding time is 5 h, the impact energy of the two different weld metals becomes closer. The Ti-bearing weld metal is much more sensitive to the normalizing treatment.

3.3. The Effect of Normalizing Processes on Microstructure

When the normalizing temperature is 900°C and 1000°C, the microstructure is polygonal ferrite and pearlite and when the normalizing temperature is 1100°C, the pearlite (pearlite and degenerated-pearlite (DP)) become coarser, as shown in Fig. 6. The size of pearlite increases against increasing temperature, as shown in Figs. 6(b) and 6(h). The SEM micrographs of weld metal after different normalizing treatments are shown in Fig. 7. The pearlite area at higher normalizing temperature has become coarser, as shown in Figs. 7(b) and 7(d).

After different normalizing holding time, microstructure of weld metal without Ti and Ti-bearing weld metal has no obvious difference, as shown in Fig. 8. With prolonging holding time, the pearlite becomes coarser, as shown in Figs. 8(b) and 8(d). The statistical result of pearlite size after normalizing is shown in Figs. 9(a) and 9(b). The number of pearlite in Ti-bearing weld metal less than 15 μm significantly decreases and that of the pearlite in the size 15–35 μm increases as the normalizing temperature increasing from 900°C to 1200°C. When normalized at 900°C, the number of fine pearlite in the Ti-bearing weld metal is more than that in the weld metal without Ti, as shown in Fig. 9(a). When normalized at 1200°C, the fine pearlite in the Ti-bearing weld metal is much less than that in the weld metal without Ti, as shown in Fig. 9(b). Impact toughness of weld metal with fine pearlite will be better than that of weld metal with coarse pearlite.

3.4. Effect of Normalizing Heat Treatment on the Ti-Bearing Precipitates

The inclusions in weld metal have an important effect on the mechanical properties and it is necessary to investigate
the Ti-bearing precipitates after normalizing treatment. The morphology of the Ti-bearing precipitates has changed both by prolonging the holding time and increasing the normalizing temperature, as shown in Fig. 10. Figures 10(a) and 10(b) shows the effect of holding time on the MnTiO precipitate on the Mn–Si oxides at 900°C, and Figs. 10(c) and 10(d) show the effect of normalizing temperature on the MnTiO precipitate on the Mn–Si oxides. The White particles on black substrate are MnTiO precipitates and the black substrate is the Mn–Si oxides. Figures 10(e) and 10(f) are the EDS analysis to the white particle and black substrate.

The white particle is the MnTiO precipitate as shown in Fig. 10(e), and the black substrate is the Mn–Si oxide as shown in Fig. 10(f). The Ti-bearing precipitates on the Mn–Si oxides disappears after normalizing heat treatment both by prolonging holding time, as shown in Figs. 10(a) and 10(b), and by increasing normalizing temperature, as shown in Figs. 10(c) and 10(d). It means that some elements in the Ti-bearing precipitates diffuse. The selected area diffraction...
patterns (SADPs) of the inclusions shows that the matrix of the inclusions is amorphous, as shown in Figs. 11(a) and 11(b), and the Ti-bearing precipitates are still MnTiO₃, as shown in Fig. 12. Composition analysis by using line scan analysis of TECNAI F30 was carried out and shows that there exists Mn-depleted zone between the inclusions and matrix at 900°C/1 h normalizing, as shown in Fig. 13. Precipitation picture in Fig. 13 is same as that in Fig. 10(c), normalized after 900°C/1 h, and the EDS analysis for MnTiO₃ precipitation is shown in Fig. 10(e). The Mn-depleted zone at higher normalizing temperature (1200°C/1 h) disappears, as shown in Fig. 14. Including picture in Fig. 14 is same as that in Fig. 10(d), normalized after 1200°C/1 h, EDS analysis is shown in Fig. 10(f).

4. Discussion

As is known to all, the heat treatment is an important method to adjust the mechanical property for material, and the basic principal is to change the dislocation through heat.
treatment. The strength property of the steel decreases and the toughness increases in common. Pearlite plays an important role on the cleavage behavior during the mechanical property test. It has been demonstrated that initial cracking in both smooth tensile and Charpy impact specimens depends largely on the result of shear cracking of pearlite in ferrite-pearlite steels. During the tensile test or the impact process, the localized slip bands in ferrite promotes cracking of the cementite plates, which is then followed by tearing of the adjacent ferrite laths. Cracks in adjoining cementite lamellae initially link up to form a fibrous microcrack. When the fibrous crack becomes large enough, it acts as a Griffith type crack in initiating unstable cleavage fracture. Therefore, the size and distribution of pearlite plays an important role on the impact property for high strength low alloy steel.

The normalizing heat treatment can change the morphology of the inclusions, as shown in Fig. 10, and the TEM experiment has shown that the Mn–Ti–O precipitate can dissolve both by prolonging the holding time and increasing the normalizing temperature, as shown in Fig. 11. H. S. Kim et al. calculated the effect of Ti on the solidus and liquidus temperatures of Mn–Ti–O for low carbon steels. As the Ti content increased from 0 to 120 ppm in low carbon steel, the oxide changed from Mn$_2$SiO$_4$ to MnTi$_2$O$_7$/Ti$_2$O$_3$ and the liquidus and solidus also increased with the Ti content. The solidus and liquidus of the MnTiO$_3$+Mn$_2$SiO$_4$ is between 1020°C to 1200°C. In our experimental here, though the Ti content is different, MnTiO$_3$ and Mn–Si oxide also found in the weld metal. As the Normalizing temperature and holding time is high and longer, MnTiO$_3$ precipitate is unstable and possible melt. C. Zhang and H. Goto et al. have reported that oxygen can diffuse into the matrix at higher temperatures of Mn–Ti–O for low carbon steel. It is probable that the Mn–Ti–O precipitate will melt and dissolve by Ti and O diffusion with increasing normalizing temperature and prolonging holding time, and then the Mn-depleted zone disappear. The detailed calculation and experiment research on the MnTiO$_3$ dissolving behavior need to be investigated further.

The Mn-depleted zone has an important effect on the transformation of austenite to ferrite. The Mn-depleted zone forms due to absorbing Mn by Ti$_2$O$_3$ or MnS precipitated on the Mn–Si oxides. In the present study, the Mn–depleted zone forms due to the MnTiO$_3$ precipitated on the Mn–Si oxides, as shown in Fig. 13, which agree well with the Ref. 19.

From the analysis and results above, it shows that the change of Ti-bearing precipitate has an important effect on the phase transformation of weld metal. During the air cooling for the Ti-bearing weld metal, the austenite grain boundary and the Mn-depleted zone around the Ti-bearing precipitate are the nucleation sites of proeutectoid ferrite and then the interface between austenite and proeutectoid ferrite become the nucleation site for pearlite thereafter. Mn-depleted zone formation increases the pearlite nucleation sites. Therefore, the pearlite size in the Ti-bearing weld metal under the same heat treatment is much smaller, comparing to the weld metal without Ti at relatively low normalizing temperature and short holding time. With the Ti-bearing precipitates dissolving with the too strong the normalizing processes (higher temperature and longer holding time), the Mn-depleted zone disappears, as shown in Fig. 14, and the nucleation sites of proeutectoid ferrite and pearlite thereafter decrease. Therefore, the pearlite will become much coarser. As the pearlite is fine and distributed uniformly in the matrix, the impact toughness of weld metal will increase. In the present study, the pearlite size become coarser while prolonging holding time and increasing normalizing temperature, and the impact toughness of Ti-bearing weld metal deteriorates, as shown in Figs. 4 and 5. For the Ti-bearing weld metal, the main factor affecting the impact toughness is the Ti-bearing precipitate, while for the weld metal without Ti, there is no change of the inclusions, and the impact toughness of weld metal has no obvious change, as shown in Figs. 4 and 5.

5. Conclusions

(1) With the prolonging holding time and increasing normalizing temperature, the impact toughness of Ti-bearing weld metal decreases significantly, and the impact toughness of weld metal without Ti does not change obviously.

(2) With prolonging holding time and increasing normalizing temperature, Ti-bearing precipitate will dissolve, and the Mn-depletion zone will disappear, which is the main factor for the deterioration of the impact toughness of Ti-bearing weld metal.

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