1. Introduction

Compact strip production (CSP) is an advanced manufacturing technology in the present iron and steel industry, which is widely adopted for hot strips production from a continuous cast slab in order to save energy and improve the productivity of steels.1–4) Compared to the low carbon steel produced by conventional hot-rolling process, CSP is characterized by high strength and fine grain size. This process allows the production of HSLA grades. Their room temperature states inherited by phase transformation from high temperature determine their mechanical behavior. Therefore, the study of the microstructure evolution of low carbon steel by CSP process is of great interest to improve the steel properties.

It is well known that the nonmetallic inclusion, such as oxides, sulphides or nitrides, formed during the solidification of steel, may change the metallurgical properties of steel. Their morphologies, sizes and distributions in steel are dominant factors, which affect some of qualities, such as weld ability, mechanical performance, or tensile strength in fatigue conditions.5) Thus, the control of their size, microstructure and distribution of precipitates is a means to increase steel properties.

The purpose of present work is to investigate the microstructure evolution and precipitation of low carbon steel produced by compact strip production (CSP) was investigated by means of scan electron microscope (SEM), transmission electron microscope (TEM) analysis and small angle X-ray scattering technique. It was found that the remarkable grain refinement can be attained by a large number of oxide and sulfide dispersive precipitates, and combining with the rapid solidification during continuous casting as well, which have dominant effect on strengthening. Consequently, the precipitation strengthening based on CSP process is also discussed in this paper.

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Table 1. Chemical composition of experimental steel (wt%).

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Al</th>
<th>Al</th>
<th>Ca</th>
<th>[O]</th>
<th>[N]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.043</td>
<td>0.021</td>
<td>0.17</td>
<td>0.010</td>
<td>0.004</td>
<td>0.021</td>
<td>0.019</td>
<td>0.0022</td>
<td>0.0036</td>
<td>0.0053</td>
</tr>
</tbody>
</table>

2. Experimental

The steel is cast into slab with a continuous casting machine, and rolled in the hot strip mill in Baotou Iron and Steel Co. Ltd. The casting speed was 0.11 m/s, the soaking temperature is 1 150°C, finishing rolling temperature is 900°C, the drum supporting force controlled the thickness of the molten sheet to 65 mm, and the strip was water cooled on the transportation roller tables and coiled at about 600°C after a laminar cooling system. The strain rate of deformation is high in the last three stands, especially in the last stand up to 120 s⁻¹.

Specimens were cut from casting slab and ultra-thin hot coil. Table 1 shows the chemical compositions of the test steel. It can be seen that the steel is high purity since the content of carbon, silicon, phosphorus and sulfur is very little. Microstructures of the low carbon CSP steel were investigated using electrolytic dissolution techniques, optical microscope, scanning electron microscope (SEM) of FEI-Quanta 400, and transmission electron microscope (TEM) of JEM JEOL 2010 operating at 200 kV coupling an energy dispersion spectrometer (EDS). The bulk specimens for OM and SEM observation were chemically etched with 4% Nital etchant. Specimens for TEM observation were cut...
from the bulk with a thickness of 0.2 mm and mechanically ground to 50 \( \mu \text{m} \), thin foil samples of \( \varnothing 3 \text{ mm} \) were finally prepared by jet-polishing method using the solution of 10% perchloric acid and 90% acetic acid. Carbon extraction replicas were also prepared through the standard procedures.

Precipitates were obtained after removing ferric oxide layer of CSP specimen and low temperature electrolysis, then separated and determined using chemistry dissolving method. Size distribution of particles was measured by means of small angle X-ray scattering goniometer according to GB/T13221-91 (ISO/TS 13762-2001).

3. Results

Sections of the samples perpendicular to the casting direction were etched and showed solidified structures. The feature of the final microstructures was mainly of polygonal ferrites and some pearlites seen in optical micrographs. **Figure 1** (a) indicates the equiaxed crystal zones formed in the surface, the diameter of the grains is about 20 to 30 \( \mu \text{m} \), while the columnar dendrite could be observed in the center region of casting slab from Figs. 1(b) and 1(c), since there is no constitutional under cooling for the formation of equiaxed ferritic grain.\(^7\) Figure 1(d) show the fine and uniformity crystal microstructure transformed from casting dendrite by hot rolling process.

It is also observed that a lot of inclusions exist in the specimen of molten slab and hot rolled sheet as well in **Fig. 1.** According to the statistics data, the inclusions in continuous cast slab were larger than that of rolled sheet, and the size of circular inclusions in rolled sheet was decrease to 1.0 \( \mu \text{m} \) from \( \varnothing 10 \mu \text{m} \) in cast slab.

**Figure 2** shows SEM observation and EDS (energy dispersive spectrum) analysis of circular, bar and cementite inclusion in experiment steel, which can be identified as oxide, carbide and sulfide+oxide in cast slab and rolled sheet by shape combining with the EDS analysis. Figure 2(a) indicates the circular inclusions consisting of manganese and aluminate oxide in continuous cast slab, whereas some of the elongated inclusions by the rolling operation were of MnS based complex in **Fig. 2(b).** Figure 2(c) show fine-scale square carbide particles distributed in hot rolled sheet, which identify as \( \text{M}_2\text{C} \), AIN and MnS. Mass percentage and size distribution of \( \text{M}_2\text{C} \) particle in rolled sheet are measured by 3014 X-ray diffracto-spectrometer/kratky small angle scattering goniometer. The test result of SAXS is given in **Table 2,** where \( \phi(D) \) is the size distribution frequency of the particle, which represent the average mass percent of \( \text{M}_2\text{C} \) particles every 1 nm’ interval. From the table we can see the particles of 10–18 nm in diameter account for 0.5%, therefore the \( \text{M}_2\text{C} \) particle would have great effect on precipitate strengthening.

It is obviously that the size of inclusion in hot rolled sheet is smaller than that of molten slab, which indicate that the complex inclusions formed during continuous casting, and get fining after hot rolled course. In addition, **Fig. 2(c)** indicates the microstructure consisted of a network of cementite outlining the grain boundaries. In the case of rolled sheet, the spheres are ferro-mangano-aluminate; the carbide particles were generally located along ferrite grain boundaries or at triple junctions in the microstructure. Carbide in steels exist as fine cementite since the content of pearlite is so little, which have great effect on strength of steel. The precipitate including carbide, \( \text{Al}_2\text{O}_3 \), AIN and MnS can be manipulated by heat treatments to form fine particles.

**Figure 3** shows the TEM observation of the low carbon steel sample rolled and coiled at 600°C. As expected, precipitates can be seen randomly dispersed within ferrite matrix, the average size of particles is 10 to 40 nm in the Fig.
in addition, a low internal dislocation density was revealed, which resulted from the great reduction of single rolling pass during continuous rolling, these internal dislocation and deformation band provide nucleation site, and result in the high nucleation rate of ferrite crystal. Moreover, the dislocation/precipitation interaction in ferrite matrix would be one of the dominant reasons of strengthening. It was observed that the precipitates are mostly spherical in appearance, and the TEM microstructure of extraction replica also reveal the existence of precipitates clearly in Fig. 3(b). Large precipitates have been identified as carbide particles through electron dispersive analysis as shown in Fig. 3(c), but the size distribution of other shapes could not be estimated because the number was so small, which indicates that they are present in the austenite prior to precipitate from the austenite during cooling.

Selective area electron diffraction study was carried out with TEM to clarify the orientation relationship between the tiny precipitates and the α-Fe matrix. Figure 3(d) shows the diffraction pattern of α-Fe matrix when beam electron

<table>
<thead>
<tr>
<th>Size interval (nm)</th>
<th>RDX (%/nm)</th>
<th>Mass Fraction %</th>
<th>Cumulative %</th>
</tr>
</thead>
<tbody>
<tr>
<td>5–10</td>
<td>0.20</td>
<td>1.0</td>
<td>1.0</td>
</tr>
<tr>
<td>10–18</td>
<td>0.50</td>
<td>4.0</td>
<td>5.0</td>
</tr>
<tr>
<td>18–36</td>
<td>0.35</td>
<td>6.3</td>
<td>11.3</td>
</tr>
<tr>
<td>36–60</td>
<td>0.12</td>
<td>2.8</td>
<td>14.1</td>
</tr>
<tr>
<td>60–96</td>
<td>0.25</td>
<td>9.1</td>
<td>23.2</td>
</tr>
<tr>
<td>96–140</td>
<td>0.25</td>
<td>10.9</td>
<td>34.2</td>
</tr>
<tr>
<td>140–200</td>
<td>0.28</td>
<td>16.7</td>
<td>50.9</td>
</tr>
<tr>
<td>200–300</td>
<td>0.23</td>
<td>22.8</td>
<td>73.6</td>
</tr>
<tr>
<td>300–430</td>
<td>0.09</td>
<td>11.8</td>
<td>85.4</td>
</tr>
<tr>
<td>430–620</td>
<td>0.08</td>
<td>14.6</td>
<td>100.0</td>
</tr>
</tbody>
</table>
beam parallel to [100] direction. A lot of diffraction spots are seen in addition to the matrix spots. It is difficult to determine the relationship between the particles and the matrix from this pattern. If we assume that the particles have a cube relationship with the matrix, the spots that do not belong to the matrix correspond very well to those of ferrite phase with the zone axis being parallel to [001]. Thus, the assumption that the tiny have cube–cube relationship with the matrix is reasonable. The dark field image in Fig. 3(e) is taken using the super lattice spots shown in the inset diffraction pattern of Fig. 3(d) and reveals that the precipitates (bright) are very small and coherent with the matrix; it would show a very effective pinning in ferrite matrix.10) The coherent orientation relationship between the tiny precipitates and the \(\alpha\)-Fe suggests that these tiny particles may precipitate from the \(\alpha\)-Fe instead of the \(\gamma\)-Fe. Furthermore, the coherent orientation relationship may influence the interfacial energy between the particle and the matrix as well as the nucleation process of the particles.10)

As shown in Fig. 4(a), a small cluster of particles were observed at junctions of a curved grain boundary. The recrystallization nuclei in these alloys would form primarily adjacent to nanometer-sized particles that are produced during casting and the movement of the boundary need overcoming the pinning force of particle, which will result in a local increase in the driving force due to changes in boundary curvature and thermal activation.11) In conclusion, small particles can restrict subsequent grain boundary movement and promote a fine-grained microstructure through Zener drag. Smith12) attributes to Zener the analysis of the pinning force exerted by particles on grain boundary, and Azmir Har et al. also simulated particle pinning force acting on the grain boundary.13) The geometry of such an interaction is shown in Fig. 4(b).

Coherent particles exert twice the pinning force as incoherent particles. Recrystallization studies have confirmed that when the precipitates are coherent they strongly pin the recovered dislocation substructure and restrict the recrystallization process14); when they are larger they effectively inhibit grain boundary migration and slow grain growth.
4. Discussion

Microstructure and precipitation evolution in CSP low carbon steel were analyzed by means of SEM and TEM, the precipitates include fine MnS and FeS particle with size about 40 to 200 nm at crystal boundary, in addition the small particles of oxide and carbide.

The square and bar inclusions were composed of MnS with a small amount of oxides, which were elongated by the rolling operation. On the contrary, circular inclusions in continuous cast slab and rolled sheet were composed of SiO$_2$, Al$_2$O$_3$ and a small amount of other complex oxides. Zener pinning refers to the inhibiting effect of precipitates particles on grain growth in polycrystalline materials was observed in this work, and the degree of contact between grain boundaries and second phase particles was introduced to predict the grain size limit in the presence of pinning effect of second phase particles in 2-D Monte Carlo simulations.\textsuperscript{15}

It is obvious that there are distinct differences between CSP and conventional processes in ingot casting, reheat schedule, rolling process, delivery speed on the table and so on.\textsuperscript{4} The process for the production of hot strip from a continuous cast thin slab allows the production of HSLA grades. Their room temperature states inherited by phase transformation from high temperature austenitic states, which can be strongly influenced by the chosen hot rolling routes, determine their mechanical behavior. In conventional cast processes, fine precipitates after cooling will dissolve again during the reheating course before hot-rolling; while the continuous casting slab of CSP process is direct-rolling without any reheating process, thus almost all the residual deoxidize sediment and sulfide dispersive precipitates were unequilibrium, which contribute to grain refining and precipitation strengthening as well, therefore hot rolling production characterized by high strength (>330 MPa) and fine grain size (4.5–8 $\mu$m) after $\gamma$ to $\alpha$ phase transformation. During the whole CSP rolling process, large number of nanometer fine carbide, oxide, sulfide and nitride precipitates retard the movement of austenite boundary, which is useful for producing ellipsoidal-shaped grains with good drawing properties. In addition, refined original austenite grain combined with controlling hot worked austenite process can also be commercially used to produce ultra-fine ferrite grain microstructure.\textsuperscript{16} The accelerating super cooling leads to an increase of the ferrite nucleation rate, and finally to fine ferrite grain sizes.\textsuperscript{17}

By understanding the role of particles on the recrystallization process in the steels, thermo mechanical processing can be designed to tailor particle size and manipulate the final grain structure. The cold-rolled material with coherent particles was very resistant to complete recrystallization even at temperatures near the melting point, indicating that nucleation was limited by particles pinning the recovered dislocation substructure that results in the form of ellipsoidal-shaped grains with good drawing properties. This is in agreement with practical production of Baotou Iron and Steel Co. Ltd.

The refinement of sulfide is accompanied by F1 to F5 hot-rolling process. Dispersive precipitates with size about 5 to 20 nm were formed at the dislocation line and grain boundaries. Therefore, it is considered that the ultra-fine particles formed by deoxidize sediment and sulphides during continuous cast process induced precipitation at direct-rolling process prohibit grain-growth. Consequently, the generation of this homogeneous fine-grained structure resulted in the elongation and strength achieved by optimizing the balance between grain nucleation and growth.

5. Conclusions

In this work the microstructure evolution and precipitation of extra low carbon steels by compact strip production process are discussed. Precipitation of the fine particles were unequilibrium, the remarkable grain refinement can be attained by residual deoxidize sediment and sulfide dispersive precipitates during the hot rolling process, and this dynamic interaction of the precipitates with the recovery of dislocations seems primarily responsible for the recrystallization of the steel, and consequently to the precipitation strengthening.

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