The Effect of Delay Time after Hot Rolling on the Grain Size of Ferrite

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It is necessary that the yield ratio (YR) of fire-resistant steel for construction is below 0.8. However, during the industrial production of fire-resistant steel, the rapid cooling rate of the laminar cooling configuration makes the ferrite grain become fine and increases the yield ratio (YR>0.8). In order to reduce the YR, by using Gleeble 1500 thermomechanical simulator the experiment of the effect of delay time after hot deformation on the grain size of ferrite was carried out. The results indicate that the hot deformation can increase the system free energy and the “ledges” generate at the grain boundary of deformed austenite, which leads to the increase of the ferrite nucleation rate and the refinement of ferrite grain. However, with the increase of delayed time, that the release of deformation energy and the reduction of “ledges” would decrease ferrite nucleation rate and increase the grain size of ferrite. Based on the experimental results, the technology to prolong the delay time after hot rolling is adopted in the industry production. The tested results indicate that the technology is effective to reduce the YR of fire-resistant steel and the YR decreases to 0.79 from initial 0.84.

KEY WORDS: hot-strip; delayed time; relaxation; yield ratio; ledge.

1. Introduction

The steel for high-rise building must have the capability to resist strong wind, earthquake and fire, so it would possess the low yield ratio (YR<0.8: yield strength/tensile strength). In the meantime, it would also have the fire-resistant capability in event of fire. Therefore, the alloying elements niobium and molybdenum must be added into the steel. However, because niobium and molybdenum can strongly restrain the recovery and recrystallization of austenite and the hot-strip is immediately water cooled to the coiling temperature after hot rolling, the grain size of ferrite easily becomes fine. The research results have indicated that the grain refinement is stronger to increase the yield strength than to increase the tensile strength, so the grain refinement of ferrite can lead to the increase of yield ratio. In order to solve the problem of high yield ratio (YR>0.8) of 490 MPa class fire-resistant hot-strip, the technology to prolong the delay time after hot rolling is adopted. The technology can obtain the appropriate grain size of ferrite and decrease the YR. The results of industry production indicate that the technology is effective to reduce the YR of fire-resistant steel and the YR decreases to 0.79 from initial 0.84. Moreover, the experiment of the effect of delay time after hot rolling on the grain size of ferrite was carried out on Gleeble 1 500 thermomechanical simulator and the related mechanism was discussed.

2. Experimental Procedures

2.1. Hot-rolling in Laboratory

The steel was prepared by melting in a vacuum induction furnace for this research. The chemical composition of steel is shown in Table 1. Table 2 shows the manufacturing conditions. The rolling test was carried out in a F300 mm rolling mill in a laboratory. The original thickness of forged billets was 100 mm and the final thickness of plates was 6mm after rolling. Having been hot rolled, the plates was water cooled to 580°C at 15°C/s, held at this temperature for 1 h, and then cooled to the room temperature at 0.1°C/s.

2.2. The First Industry Trial-production

The steel was hot rolled in hot strip mill, of which the chemical composition, the finish-rolling temperature and the coiling temperature are nearly the same as those in the laboratory and yet the cooling rate is different, as shown in Table 3. The cooling mode in industry production is the laminar cooling configuration, as shown in Fig. 1. At first, the hot-strip after hot rolling enters the main cooling zone, of which cooling capacity is so strong that it merely takes 8 s for the hot-strip of 6 mm thickness to be water cooled to 640°C from 880°C. The cooling rate is up to 30°C/s and twice as rate as the laboratory. Then the hot-strip would pass a section of air cooling zone, in which the temperature of hot-strip reduces little. Finally, the hot-strip enters the refined cooling area, in which the temperature of hot-strip is water cooled to coiling temperature 580°C. The cooling
rate of refined cooling zone is also 30°C/s.

2.3. Thermomechanical Simulator Experiment on the Delay Time after Hot Deformation

Thermomechanical simulator experiment was performed on Gleeble 1500 thermomechanical simulator. The samples were heated to 1200°C for 3 min, cooled to 880°C at 5°C/s, deformed with the reduction of 0.4 and the strain rate 1/s. After deformation the samples were held 0, 10, 20 and 30 s respectively, cooled to 580°C at 30°C/s, and then cooled to room temperature at 0.1°C/s.

2.4. The Second Industry Trial-production

In the second industry trial-production, the composition, reheating temperature, finish-rolling temperature and cooling temperature of steel are the same as those in the first industry trial-production. The difference from the first industry trial-production is that the hot-strip was not immediately water cooled when it entered the main cooling zone, but the water does not spray until the hot-strip has run 15 s on the runout table, as shown in Table 4.

3. Results

3.1. Mechanical Properties and the Microstructures Under the Laboratory Conditions

The JIS G3136 standard for 490 MPa class low-yield fire-resistant steel is shown in Table 5. The tensile specimens were got from the as-rolled plate in the transverse direction. The mechanical properties are shown in Table 6. It can be seen that the mechanical properties under such the chemical composition and technology can satisfy the mechanical properties requirement of 490 MPa class fire-resistant steel. The microstructures of steel are ferrite and pearlite, as shown in Fig. 2(a).

3.2. Mechanical Properties and the Microstructures of Steel in the First Industry Trial-production

The tensile specimens were got from the as-rolled hot-strip in the transverse direction. The mechanical properties are shown in Table 7. It can be seen by comparison that the strength of steel in the industry trial-production is higher than that under the laboratory conditions, and the YR becomes high. The microstructures of steel are also ferrite.

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| C    | Si    | Mn    | Cr    | Mo    | Nb    | P    | S    |
|------|-------|-------|-------|-------|-------|------|------|
| 0.08 | 0.25  | 0.50  | 0.30  | 0.50  | 0.013 | 0.013| 0.010|

Table 1. Chemical composition of the steel tested in a laboratory (mass%).

| Thickness | Reheating  | Finishing-rolling | Cooling rate | Coiling |
|-----------|------------|-------------------|--------------|---------|
| (mm)      | temperature(°C) | temperature(°C) | (°C/s)       | temperature(°C) |
| 6         | 1200       | 880               | 15           | 580     |

Table 2. Technological parameters in a laboratory.

| Thickness | Reheating  | Finishing-rolling | Cooling rate | Coiling |
|-----------|------------|-------------------|--------------|---------|
| (mm)      | temperature(°C) | temperature(°C) | (°C/s)       | temperature(°C) |
| 6         | 1200       | 880               | 30           | 580     |

Table 3. Technological parameters in the first industry trial-production.

Fig. 1. Schematic diagram of laminar cooling configuration.
Table 4. Technological parameters in the second industry trial-production.

| Thickness (mm) | Reheating temperature (°C) | Coiling temperature (°C) | Cooling rate (°C/s) | Delay time (s) |
|----------------|---------------------------|--------------------------|---------------------|---------------|
| 6              | 1200                      | 880                      | 580                 | 30            |

*Delay time: Air-cooling time from finishing-rolling to accelerated-cooling.

Table 5. Specifications of 490 MPa low-yield fire-resistant steel.

| Applicable thickness Range, (mm) | σ_y (MPa) | σ_u (MPa) | Elongation (%) | YR (%) | σ_max (MPa) |
|----------------------------------|-----------|-----------|----------------|--------|-------------|
| 6–12                             | ≥325      | 490–610   | ≥17            | ≤80    | ≥217        |

Table 6. Mechanical properties of the steel tested in a laboratory.

| Thickness (mm) | Yield strength, σ_y (MPa) | Tensile strength, σ_u (MPa) | Elongation (%) | YR (%) | Yield strength at 600°C, σ_max (MPa) |
|----------------|---------------------------|-----------------------------|----------------|--------|-------------------------------------|
| 6              | 390                       | 500                         | 25             | 0.78   | 265                                 |

Table 7. Mechanical properties of the hot-strip in the first industry trial-production.

| Thickness (mm) | Yield strength, σ_y (MPa) | Tensile strength, σ_u (MPa) | Elongation (%) | YR (%) | Yield strength at 600°C, σ_max (MPa) |
|----------------|---------------------------|-----------------------------|----------------|--------|-------------------------------------|
| 6              | 460                       | 548                         | 17             | 0.84   | 312                                 |

Fig. 2. Microstructures under different cooling conditions. (a) In a laboratory (the grain size of ferrite: 13 μm); (b) in the first industry production (the grain size of ferrite: 8.5 μm); (c) in the second industry production (the grain size of ferrite: 10.5 μm).
and pearlite, but the ferrite grain becomes finer and the pearlite becomes finer and diffuser, as shown in Fig. 2(b).

3.3. Microstructures at Different Delayed Time after Hot Deformation

Figure 3 shows the microstructures of thermomechanical simulation samples, which were hold different time at 880°C after hot deformation, cooled to 580°C at 30°C/s, and then cooled to room temperature at 0.5°C/s. The delay time of Figs. 3(a), 3(b), 3(c) and 3(d) were 0 s, 10 s, 20 s, 30 s respectively. It can be seen that the grain size of ferrite increases with the increase of delay time.

3.4. Mechanical Properties and the Microstructures of Steel in the Second Industry Trial-production

The results indicate that prolonging the delay time after hot deformation is effective to reduce the YR under the conditions of not changing the chemical composition, the cooling rate and the other technology parameters. The mechanical properties of steel are shown in Table 8. The ferrite grain in the second industry trial-production obviously becomes coarse in comparison with that in the first industry trial-production. The microstructures of steel consist of ferrite and pearlite, as shown in Fig. 2(c).

4. Discussion

4.1. Effect of Cooling Rate on the Grain Size of Ferrite

It can be found by comparison from Figs. 2(a) and 2(b) that the ferrite grain obtained in the first industry trial-production is finer than that under the laboratory conditions, so both the strength of steel and YR increase. Although the chemical composition, the finishing-rolling temperature and the cooling temperature are nearly the same as those of the laboratory, the rapid cooling rate in the first industry production significantly increases the degree of supercooling and reduces the transformation temperature, which leads to the refinement of ferrite grain, the increase of the strength of steel and the increase of YR. In order to reduce the cooling rate, the sparse cooling style can be adopted. However, due to the restrictions of the production efficiency and lamellar cooling configuration, the practical cooling rate can’t be reduced. Therefore, the appropriate grain size of ferrite is the key to acquire the low YR under the condition of not changing the cooling rate.

4.2. Effect of Delay Time after Hot Rolling on the Grain Size of Ferrite

Figure 3 shows that the grain size of ferrite increases with the increase of delay time after hot rolling. Therefore, both the strength and YR of steel in the second industry trial production are lower than those of the first industry trial production, as shown in Tables 7 and 8. These changes indicate that the different delay time can affect the grain size of ferrite. It is concluded that there are two main influencing factors.

The first influencing factor is the change of deformed austenite. The deformation energy can increase the system free energy. Therefore, with the increase of delay time, the deformation energy would release by means of static recovery and recrystallization, which would decrease the system free energy, as shown in Fig. 4. Thus, the shorter the delay time after hot rolling is, the less the release of deformation energy is, which would lead to the increase of the transformation driving force of austenite to ferrite. On the contrary, the longer the delay time is, the more the release of deformation energy is, so the transformation driving force of austenite to ferrite would decrease. Obviously, the delay time can affect the transformation driving force of γ→α. In addition, it can be known from transformation thermodynamics that the critical nucleation radius is (In order to easily explain, it is considered that the nucleation of new phase is uniform.):

\[ r^* = \frac{2\sigma}{\Delta F^* + E} \] .............................(1)

where \( \sigma \) is the per unit area interface energy of new phase, \( \Delta F^* \) the change of volume free energy resulted from the occurrence of per unit volume new phase, and \( E \) the elastic distortion energy resulted from the formation of per unit volume new phase. In addition, \( \Delta F^* = -(1/V^m)\Delta^*G_m \) where \( V^m \) the mole volume, and \( \Delta^*G_m \) the driving force to form 1 mol new phase. The critical nucleation work of new phase is:

\[ \Delta F^* = \frac{4}{3} \pi r^* \sigma \] .............................(2)

where \( r^* \) is the critical nucleation radius, and \( \sigma \) the interface energy of unit area new phase. The nucleation rate of new phase is(10):

\[ \dot{N}_s = 10^{30} \exp \left( \frac{-DF^*}{RT} \right) \] .............................(3)

where \( R \) is the Boltzmann constant, and \( T \) the absolute temperature. Therefore, the free energy of the long delayed time deformed austenite would be higher than that of the delayed time short deformed austenite (\( \Delta^*G_m > \Delta^*G_{m*} \)). And because \( \Delta F^* = -(1/V^m)\Delta^*G_m \), based on Eq. (1), it can be acquired that the critical nucleation radius \( r^* < r^* \) and \( \Delta F^* < \Delta F^* \). Therefore, the ferrite nucleation rate of the
short delay time deformed austenite would be higher than that of the long delay time deformed austenite. Furthermore, after hot rolling the hot-strip is immediately water cooled and the delay time at elevated temperature is short, so there is no enough time for the ferrite grain to grow up. In addition, the coiling temperature is 580°C, at which the diffusion coefficient of carbon atoms is low, so the ferrite grain is difficult to grow up. After the ferrite grains nucleate, the growth of ferrite grain is controlled by the long-range diffusion of carbon atoms, and the growth rate of ferrite grain is defined as:

$$r^2 - r_0^2 = \frac{(C_\gamma - C_\alpha)}{(C_0 - C_\alpha)}Dt \quad \quad (4)$$

where $r_0$ is the nucleation radius of ferrite, $r$ the final grain radius of ferrite, $t$ the growth time, $C_\gamma$ the carbon concentration of parent phase, $C_\alpha$ the carbon concentration of $\gamma$ phase at $\gamma/\alpha$ interface, $C_\alpha$ the carbon consistency of $\alpha$ phase, and $D$ the diffusion coefficient of carbon atom. If the nucleation rate of ferrite is high, the nucleation sites of ferrite formed at the austenite grain boundary would quickly become saturated. Because the ferrite grains collide each other during the growth of ferrite grains and the interference among the growing ferrite grains is strong, the diffusion of carbon atoms and alloying elements between the new phase $\alpha$ and the parent phase is hindered, as shown in
Thus, the growth rate of ferrite is reduced. On the contrary, if the nucleation rate of ferrite is low, the nucleation sites of ferrite would no more be saturated and the impact among the growing ferrites would reduce. Therefore, there is enough space for ferrite grain to grow up, and so the ferrite grain becomes coarse. In addition, a lot of interfaces occur due to the formation and growth of ferrite grains. The interface energy is the driving force for ferrite grain to grow up. The formula on grain growth can be expressed as

\[ \bar{D}^2 - D_0^2 = \frac{2MeV}{\beta} t \]  

where \( \bar{D}_0 \) is the nucleation diameter of ferrite, \( \bar{D} \) the final grain diameter of ferrite, \( t \) the coarse time, \( \sigma \) the interface energy, \( \beta \) the constant which is related to grain shape, and \( M \) the migration rate of atom at grain boundary. \( M \) is defined by the Wilson–Frank formula:

\[ M = \frac{a v}{RT} \exp \left( -\frac{Q}{RT} \right) \]

where \( a \) is lattice constant, \( Q \) the atomic diffusion activation energy, \( v \) the vibration frequency of atoms near the interface, \( R \) the Boltzmann constant, and \( T \) the absolute temperature. Because the cooling temperature of hot-strip is 580°C, the diffusion activation energy of Fe atom is high under this temperature and it is difficult for the ferrite grain to grow up. Thus, under the conditions of relatively rapid cooling rate and low cooling temperature, the nucleation rate is the most important factor to determine the final grain size of ferrite. The greater the nucleation rate is, the finer the ferrite grain is. Therefore, the shorter the delay time at elevated temperature is, the finer the ferrite grain is. On the contrary, the longer the delay time is, the coarser the ferrite grain is. Just as shown in Figs. 3(a), 3(b), 3(c) and 3(d), the grain size of ferrite also increases with the increase of delay time.

Secondly, the grain boundary state is also the factor to influence the grain size of ferrite. Figs. 6(a), 6(b), 6(c) and 6(d) show the recrystallized microstructures of deformed austenite, of which the delay time at elevated temperature are 0 s, 10 s, 20 s, and 30 s respectively. It can be seen from Fig. 6 that the deformed austenite grains gradually change from the original prolonged state to the part static recrystallization state, and then to the full static recrystallization state. Despite the austenite grain shown in Fig. 6(a) is the coarsest and grain boundary area is the least, the ferrite grain after transformation (Fig. 3(a)) is the finest in comparison with Figs. 3(b), 3(c) and 3(d). Similarly, despite the austenite grain shown in Fig. 6(d) is the finest and grain boundary area is the biggest, the ferrite grain after transformation (Fig. 3(d)) is the coarsest in comparison with Figs. 3(a), 3(b) and 3(c). Just as discussed above, it is because of the change of delay time after hot deformation. On the other hand, it is because the deformation contributes to the change of the austenite grain boundary state. During the static recovery and recrystallization of deformed austenite, the atoms would rearrange with the increase of time, namely, the occurrence of relaxation. D.S. Ge reported that there were not only the point defect relaxation and the dislocation relaxation, but also the grain boundary relaxation. Furthermore, the research results have indicated that the nucleation site of ferrite is at the grain boundary of deformed austenite when deformation temperature is above 720°C, as shown in Fig. 5. Therefore, when the austenite is...
deformed, the dislocation would collide the grain boundary and interact with grain boundary, which would lead to the disorderly arrangement of atoms at grain boundary and the distortion of lattice, as shown in Fig. 7(a). Therefore, the grain boundary of austenite become rough and a lot of “ledges” generate, as shown in Fig. 7(b). It is known that the critical nucleation work $D_F^*$ of ferrite formed at “ledge” is only $\theta/\pi$ times as high as that at smooth grain boundary, where $\theta$ is the angle of “ledge”. Based on the Eq. (3), the smaller the critical nucleation work is, the bigger nucleation rate $J_s$ is and the finer the ferrite grain is. With the increase of delay time at elevated temperature, the grain boundary relaxation can take place. The atoms arranging disorderly begin to rearrange and the grain boundary of deformed austenite also become smooth, the “ledges” gradually reduce and the nucleation rate of ferrite decreases, so the ferrite grains also become coarse. On the whole, after hot deformation, the deformation energy and the “ledge” would increase the nucleation rate of ferrite, which would result in the refinement of ferrite grain. With the increase of delay time, the deformation energy would release and the “ledge” reduce, which would lead to the decrease of ferrite nucleation rate and the coarseness of ferrite grain.

5. Conclusion

(1) After hot deformation, the deformation energy and the “ledge” would increase the nucleation rate of ferrite, which results in the refinement of ferrite grain. But with the increase of delay time at elevated temperature, the deformation energy would release and the amount of “ledges” at grain boundary would reduce, which would lead to the decrease of ferrite nucleation rate and the coarseness of ferrite grain size.

(2) The results from the industry production indicate that the technology to prolong the delay time after hot rolling is an effective method to reduce the YR of fire-resistant steel.

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