Grain Refinement in Steels and the Application Trials in China

Han DONG, Xingjun SUN, Weijun HUI, Shulan ZHANG, Jie SHI and Maoqiu WANG

Central Iron and Steel Research Institute, National Engineering Research Center of Advanced Steel Technology, Beijing 100081, China.

(Received on February 5, 2008; accepted on May 21, 2008)

In order to control microstructure and performances of steel products, the investigation has been taken to the ways to refine grains in steels and the mechanisms, the availability in industrial production of ultrafine grain steel products, and the applications of ultrafine grain steel products. It is shown that strength of steels can be increased without detriment to ductility when ferrite grains are refined to less than 4 \( \mu \text{m} \) in low carbon steels. Based on the study of deformation induced transformation, a new rolling technology has been developed to refine ferrite grains in plain low carbon steel rebar and strip. Plain low carbon steel rebars of 400 MPa grade, with ferrite grains in the range of 4–8 \( \mu \text{m} \), have been developed as the candidate to replace microalloyed steel rebars. High strength plain low carbon steel strips in the yield strength range from 345 to 420 MPa have been used for components conventionally made of microalloyed steels. Ultrafine grain microalloyed steel strip has also been developed. A new on-line cementite spheroidization softening technology has been studied for the wire rod production of medium carbon bolt steel. It is obvious that deformation could also induce pearlite transformation in eutectoid steel.

KEY WORDS: high strength steel; grain refinement; deformation; transformation.

1. Introduction

Grain refinement is regarded as the only method to improve both the strength and toughness of steels simultaneously. Since 1960’s, numerous works have been devoted to ferrite grain refinement in HSLA steels. The TMCP technology has been well established as an effective method for grain refinement, and widely used in industrial production of HSLA steels. However, the minimum grain size of ferrite achieved by conventional TMCP is limited to 10–20 \( \mu \text{m} \) for plain C–Mn steels, and 4–5 \( \mu \text{m} \) for microalloyed steels. Since 1990’s, some ultrafine grained approaches for bulk metals, such as severely plastic deformation, ultimate use of TMCP, heavy deformation and annealing and so on, have been developed and intensively studied in the laboratory. By using these methods, the ferrite grain has been refined to an order of micron or submicron, with both the strength and toughness of steels being improved significantly. However, there still exists a big gap to be overcome between these laboratory trials and industrial scale production of ultrafine grained steels at present.

It has been well known for a long time that the applied stress or strain could induce diffusionless transformation to occur in steels, e.g., from austenite to martensite in TRIP steel, austenitic stainless steel and duplex steel. In recent years, people have been studying the austenite to ferrite transformation induced by deformation in order to obtain finer ferrite grains. There are indeed several terminology names to describe this kind of transformation, dynamic transformation, strain induced (enhanced) ferrite transformation, etc. Due to the transformation being associated with all deformation variables, strain, strain rate, temperature and deformation manner, the authors named the transformation as “Deformation Induced Ferrite Transformation” (abbreviated as DIFT). DIFT is one of the transformations associated with accumulative deformation energy change, which is proved to be an effective method for grain refinement in both plain low carbon steel and microalloyed steel. DIFT has been applied to refine ferrite grains to 3 \( \mu \text{m} \) in plain low carbon steels and 1 \( \mu \text{m} \) in microalloyed steels, with yield strength to be increased to the level of 400 MPa and 800 MPa respectively. The work of the authors shows that the transformation is induced through the increase of both austenite free energy and nucleation site density caused by deformation applied. It has been proved that proeutectoid ferrite can be induced from austenite in low carbon steel. Deformation induced transformation (DIT) could also happen in medium carbon hypoeutectoid steel and eutectoid steel. The DIT rolling technology seems to be one of the effective ways to refine grains into an order of microns in industrial scale.

The authors will review the developments of steel research activities on fine grained plain low carbon steel, grain refinement in microalloyed steel, softening medium carbon steel and fine grained eutectoid steel based on the understanding of phenomenon of DIT.

2. Fine Grained Plain Low Carbon Steel

Based on the study of DIFT and recrystallization of austenite, a new DIFT rolling technology has been developed to produce plain low carbon steel products with fine grains, Fig. 1. The largest difference between DIFT rolling and conventional TMCP is that the former makes full use...
of DIFT on the basis of suitably controlling austenite state. First, the austenite is deformed at a relative higher Zener–Holoman parameter in order to refine the austenite grain size by dynamic or static recrystallization more efficiently. The fine grained austenite obtained is favorable for DIFT and ensures the homogeneity of the final microstructure. Second, the fine grained austenite is deformed in the non-recrystallization temperature region, which is not only the necessary prerequisite of DIFT but also helpful for the ferrite grain refinement during cooling. It was thought before that the recrystallization of plain low carbon steel was very easy to occur and therefore there usually not existed a so-called non-recrystallization region in practice. However, our previous study showed that under the condition of relatively lower deformation temperature and shorter interpass period, it was possible that the deformed austenite cannot be recrystallized or only partially recrystallized, which creates the condition for DIFT. Third, fine grained ferrite is induced by deformation during rolling at a relative lower temperature. Finally, the retained austenite that has been divided into small colonies by DIF is transformed into fine ferrite during cooling.

For a low carbon hypoeutectoid steel, 0.17%C–0.27%Si–0.71%Mn, the specimen was soaked at 1150°C for 5 min and then cooled at 20°C/s to 810°C to suppress austenite transformation \((A_{3s}=835°C, \ A_{3f}=714°C)\). At 810°C, the specimen was compressed 50–60% at 0.1/s and then water cooled to the ambient temperature. It is obvious that fine and equiaxed ferrite grains can be induced from austenite through deformation, Fig. 2(a). The ferrite nucleated at grain boundaries and deformation bands respectively. The retained austenite was divided into small colonies which would transform into ferrite during successive cooling. The size of ferrite grains induced is in the micron scale and the ferrite grains transformed from retained austenite are smaller than that of grains transformed conventionally. So, the final ferrite grains can be refined into the micron scale.

One of the main results of DIFT is the formation of much finer ferrite grains after transformation, which could be used to refine both austenite and ferrite grains in steel. Because of micron order of ferrite grains can be obtained through DIFT in plain low carbon steel and microalloyed low carbon steel, it has been identified as an effective method in potential industrial scale for microstructure control. This is the basic idea for the grain refinement in plain low carbon steel to increase its yield strength from 200 MPa grade to 400 MPa grade. This is what we call DIFT rolling technology.10–12) We have made our effort to introduce this idea to the steel people in China in recent years. The new TMCP based on DIFT rolling has been used by some steel plants to produce fine grained PLC steel strip and rebar. The ferrite grain size can be refined from \(\sim 20 \mu m\) to 3–8 \(\mu m\) and accordingly the yield strength is raised from \(\sim 200 \text{ MPa}\) to the level of 350–400 MPa, Fig. 3. Comparing with the GB standards (GB means Chinese National Standard) of plain low carbon steels, yield strength of plain low carbon steel rebars and strips is improved through grain refinement remarkably. Tens of thousand tons of plain low carbon steel strips and steel rebars have been produced to meet the increasing demands for the higher strength and lower cost steel products. Typical microstructures of fine grained rebar and strip are shown in Figs. 2(b) and 2(c).
3. Grain Refinement in Microalloyed Steel

3.1. Laboratory Trial on 700 MPa Grade Ultrafine Grained Steel

The dissolved niobium can retard DIFT but the precipitation of niobium will promote DIFT if recrystallization of austenite does not occur. The role of niobium in DIFT should be taken into consideration in the development of DIFT rolling procedure of niobium-containing steels. We have developed a new TMCP for the production of ultrafine grained Nb-microalloyed steel in laboratory. A steel slab with thickness of 30 mm was firstly soaked at 1200°C for 30 min, followed by five pass rolling to a final thickness of 3 mm, then cooled at 20°C/s to 550°C and finally cooled slowly in a furnace to room temperature to simulate coiling process of the strip. The role of the first pass rolling at 1000°C is to refine austenite grains by recrystallization considering that fine austenite grains will be beneficial to DIFT. The role of the second pass rolling at 950°C is to promote the precipitation of (Nb, V)(C, N), which can help to accelerate DIFT kinetics. The final three pass rolling at 820°C is DIFT rolling as confirmed by the microstructural observation on water quenched samples. The final microstructure obtained in the experimental steel with chemical composition of 0.094%C–0.47%Si–1.38%Mn–0.1%V–0.04%Nb–0.02%Al–0.018%N is shown in Fig. 4(a). It can be seen that ultrafine ferrite grains of 1.5 μm in mean size is obtained. The grains are much smaller than those produced by conventional TMCP (≥5 μm). The yield strength of the experimental steel can be over 700 MPa.

3.2. Industrial Production of High Strength CuPCrNi Weathering Resistance Steel

There exists a steady demand of high strength for weathering resistance steel. Based on SPA-H steel, chemical compositions and/or processing variables were modified in order to obtain finer grains through DIFT rolling: one was to modify tandem rolling processing variables for the steels with chemical compositions of conventional SPA-H steel; another one was to apply DIFT rolling to steels microalloyed with niobium. Five heats were melted in convert furnace, of which two heats were steels microalloyed with 0.03% niobium.

For conventional SPA-H steel rolled at low temperature, the mean yield strength ($R_{p0.2}$) and ultimate tensile strength ($R_m$) of the strips are 430 MPa and 540 MPa respectively, comparing with $R_{p0.2} = 400$ MPa and $R_m = 510$ MPa of commercial SPA-H steel strips, Table 1. As DIFT rolling was applied to microalloyed steel, the mean yield strength and ultimate tensile strength of strips were increased to 510 MPa and 605 MPa respectively. The strength increment

![Fig. 3. The improved yield strength of plain low carbon steel rebars and strips through grain refinement, comparing with the GB (Chinese National) standards of plain low carbon steels which are specified as QXXX.](image)
is mainly due to grain refinement from commercial SPA-H of 10 µm to microalloyed steel of 4 µm with good uniformity across the section, Figs. 4(b) and 4(c).

4. DIT and Spheroidization in Medium Carbon Steel

It has been demanded to shorten or even eliminate offline carbide spheroidization treatment for medium carbon
steel. Little work has been done on the on-line softening of medium carbon steels, which have ferrite plus spheroidized cementite structure instead of ferrite and pearlite structure being obtained in as-rolled state by controlled rolling and cooling.\textsuperscript{13,14} The purpose of our study is to investigate the effect of DIT rolling on spheroidization of medium carbon steel.

Figure 5 shows the microstructures of the commercial medium carbon 0.36%C–0.62%Mn steel ($A_{\text{e}3}=812^\circ\text{C}$, $A_{\text{e}1}=727^\circ\text{C}$, $A_{\text{r}1}=645^\circ\text{C}$ and $A_{\text{r}3}=550^\circ\text{C}$ at cooling rate of 5°C/s) subject to deformation at different temperatures. For the specimen deformed at 950°C, which is much higher than $A_{\text{e}3}$, there was no ferrite transformed. When the specimen was deformed at 800°C, there was some equiaxed ferrite formed mainly at prior austenite grain boundaries, which suggests that deformation induced ferrite was formed although the deformation temperature of 800°C is higher than $A_{\text{e}3}$. When deformation temperature was decreased to 700°C, which is also higher than $A_{\text{e}3}$, fine equiaxed ferrite plus pancake-like martensite microstructure was obtained in the quenched specimen. The volume fraction of equiaxed ferrite increased and the grain size of ferrite decreased with the decrease of deformation temperature and the increase of deformation strain. Ultrafine ferrite grains of about 2 μm in size could be obtained in specimens subject to larger strain at lower deformation temperature. There are few spheroidized cementite particles within austenite (now martensite) directly after deformation for the water cooled specimens. However, most of cementite particles along the boundaries of deformation induced ferrite grains are rod-like or sphere-like ones. It may suggest that deformation induced cementite transformation (DICT) could accompany with DIFT.

Spheroidized cementite could be obtained after controlled cooling of deformed specimen. The volume fraction of spheroidized cementite increased with decreasing deformation temperature, especially when the deformation temperature is lower than 750°C. When the steel was deformed at 700°C, a fairly spheroidized microstructure was obtained though there was still a little volume fraction of lamellar cementite existing. Though the underlying mechanism of strain-induced cementite spheroidization is still under debate, the cementite particles evenly distributed along ferrite grain boundaries might be related with the direct decomposition of very small retained austenite islands existing between ferrites during deformation and subsequent cooling. These results suggest that only very small carbon-rich retained austenite prefers to transform to ferrite plus cementite instead of pearlite during deformation and cooling.

5. DIT and Grain Refinement in Eutectoid Steel

Whether the pearlite can be induced has not been clarified. Thus it is necessary to study the pearlite transformation under deformation further and expand the application of DIT in high carbon steel. A eutectoid carbon steel (0.75%C–0.26%Si–0.66%Mn–0.002%S–0.00073%P, $A_{\text{r}1}=725^\circ\text{C}$, $A_{\text{r}3}=550^\circ\text{C}$ at a rate of 20°C/s) was selected to study the deformation induced pearlite transformation (DIP).

One specimen was austenitized at 1 150°C for 60 s, cooled to 700°C at a rate of 20°C/s, then quenched in water. For the deformation experiments, after cooled to 700°C, the specimens were compressed at different deformation variables, cooled in the water. The thermal dilation test was performed on the Thermecmastor-Z simulator, in which the specimens were soaked at 1 000°C for 180 s, cooled to 700°C and compressed by 0, 30, 60% respectively, held at 700°C for 1 200 s, and then cooled in the water. It can be seen that there is no pearlite, which proves that the pearlite did not form before the specimen was cooled to 700°C. Fig. 6(a). However, under plastic deformation at 700°C, there is pearlite transformed from austenite, Fig. 6(b). It can be seen that the interlamellar spacing of the induced pearlite was very small, which was about 0.1–0.2 μm. Fig. 7. And, the colony of the pearlite was also small in size. For the smaller colony and the finer interlamellar spacing, the induced pearlite can enhance the property of the high carbon steel.

The dilation experiment results show that during isothermal holding the relative dilation change of the specimen deformed to 60% was smaller than the one deformed to 30%, i.e., the amount of the pearlite formed during isothermal holding for the specimen deformed to 60% was smaller, Fig. 8. The difference must be caused by the occurrence of the pearlite transformation before deformation or during deformation. According to optical microstructure observation, Fig. 6(a), it is impossible for the austenite transformed to pearlite before deformation. So the experiment proves that the pearlite was induced during deformation. The experiment also shows that the fraction of the induced pearlite increased with the reduction increase.

In terms of the relation between the relative dilation $\Delta D/D_0$ and phase fraction, neglecting the pro-eutectoid ferrite, at 700°C, the fraction of the induced pearlite ($f_\text{p}$) can be calculated by the relative dilation change, $\Delta D/D_0=0.66f_\text{p}$.
(where \(\Delta D\) is the measured diameter change of specimen, \(D_0\) is the initial diameter of specimen). Considering the transformation plasticity, the fraction of the induced pearlite for the specimen deformed to 60\% at 700°C is calculated to be about 8.69\%, which is consistent with the OM result.

From Fig. 8 it can be also seen that the relative dilation of the undeformed specimen almost did not increase until 800 s during isothermal holding. However, the deformed specimen expanded quickly at the beginning of the holding. This means the incubation period of the deformed specimen was shorter, which is related with the accumulated energy stored in the specimen. The accumulated energy induced the pearlite transformation. The larger the accumulated energy, the earlier the initiation of the transformation was.

Microstructural observation and dilatometric analysis identified that the existence of the deformation induced pearlite transformation (DIPT). DIPT is influenced by the diffusion of carbon atoms. For the induced pearlite, the colony is small and the interlamellar spacing is small, which has good prospect for improving the properties of the high carbon steel.

6. Summary

(1) New DIFT rolling technology has been developed to produce ultrafine grained steel with finer grain than that produced by conventional TMCP. DIFT in hypoeutectoid steel can be used to obtain fine ferrite grains of 4–8 \(\mu m\) for plain low carbon steel and of 1 \(\mu m\) for microalloyed steel. Strength of the steels can be increased remarkably due to the grain refinement.

(2) Base on the understanding of DIT, a new on-line cementite spheroidization softening technology has been developed for the wire rod production of medium carbon bolt steel. It is shown by the investigations that deformation could induce pearlite transformation in eutectoid steel. Fine pearlite colonies and small pearlite lamellar spacing can be obtained through DIPT in eutectoid steel.

(3) The diffusional transformations from austenite to ferrite, pearlite and cementite could be induced through deformation due to the accumulative deformation energy change to free energy. DIT is worthy to pay more attentions with because it is and will be an effective way to control microstructures expected than ever before, then lead to the development of new steel products.

REFERENCES

1) I. Tamura, C. Ouchi, T. Tanaka and H. Sekine: Thermomechanical Processing of High Strength Low Carbon Steels, Butterworth & Co. Ltd., London, (1988), 156.

2) M. Niikura, M. Fujioka, Y. Adachi, A. Matsukura, T. Yokota, Y. Shirata and Y. Hagiwara: *J Mater. Process. Technol.*, 117 (2001), 341.

3) Y. Iwashashi, Z. Horita, M. Nemoto and T. G. Langdon: *Acta Mater.*, 45 (1997), No. 11, 4733.
4) W. Y. Choo, J. S. Lee, C. S. Lee and J. K. Choi: *CAMP-ISIJ*, 13 (2000), 1144.
5) H. Yada, C. M. Li and H. Yamagata: *ISIJ Int.*, 40 (2000), No. 2, 200.
6) P. J. Hurley and P. D. Hodgson: *Mater. Sci. Technol.*, 17 (2001), 1360.
7) Z. Q. Sun, W. Y. Yang, P. Yang, J. J. Qi and W. W. Zheng: Workshop on New Generation Steel, CSM, Beijing, (2001), 35.
8) M. R. Hickson, R. K. Gibbs and P. D. Hodgson: *ISIJ Int.*, 39 (1999), No. 11, 1176.
9) K. Nagai: Proc. of the 4th Workshop on the Development of High Performance Structural Steels for 21st Century, Pohang, Korea, (2002), 17.
10) H. Dong, Q. Y. Liu, Z. M. Yang, W. J. Hui, X. J. Sun, T. Peng, L. Q. Song, Y. Weng, Y. Zhang and Q. L. Wang: Proc. of the 2nd Int. Conf. on Advanced Structural Steels, CSM, Beijing, China, (2004), 47.
11) H. Dong, X. J. Sun, W. J. Hui, S. L. Zhang, Q. Y. Liu and Y. Q. Weng: Proc. of the 3rd Int. Symp. on Ultrafine Grained Structures, CSM, Beijing, China, (2005), 55.
12) H. Dong and X. J. Sun: *Curr. Opin. Solid State Mater. Sci.*, 9 (2005), No. 6, 269.
13) L. Storojeva, R. Kaspar and D. Ponge: *Mater. Sci. Forum*, 426–432 (2003), 1169.
14) H. Hata, H. Yaguchi, M. Shimotsusa, et al.: *Kobelco Technol. Rev.*, 25 (2002), 25.