

## THE MICROSTRUCTURE OF A PIPELINE STEEL SUBJECTED TO CONTROLLED ROLLING AND ACCELERATED COOLING

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In this paper, the microstructure and mechanical properties of a clean pipeline steels subjected to controlled rolling and accelerated cooling have been studied by means of optical microscopy, transmission electron microscopy (TEM) and X ray diffraction (XRD). The results show a mixed microstructure of acicular ferrite and polygonal ferrite, as well as the martensite thin film at grain boundaries, can be obtained for pipeline steels from heavy hot deformation in the non-recrystallization temperature range and followed by accelerated cooling. The mechanical testing showed there is a good combination of the property with the mixed microstructures of acicular ferrite, polygonal ferrite, granular ferrite and grain boundary thin film.

### Introduction

In recent years, some research work has been focussed on improving the purity and obtaining fine microstructures for steels aimed at a better combination of high strength and high toughness [1], for instance, pipeline steels. Pipeline steels may suffer damages during their service, such as hydrogen induced cracking, stress corrosion cracking, etc., when they are subjected to a wet corrosive environment containing hydrogen sulfide [2, 3]. Therefore, achieving higher toughness that enhances the crack propagation resistance is crucial to the development of high strength pipeline steels. There are generally two ways for improvement of mechanical properties of pipeline steels. One way is related to modification of the chemical composition of steels and TMCP (thermomechanical controlled processing) that strongly affects the microstructure of steels is another way.

It is believed that damages such as hydrogen induced cracking and stress corrosion cracking are related to the chemical segregation of impurities such as S, P, O and N, as well as the precipitation of carbides [2-5], especially those at grain boundaries that may result in a low temperature intergranular fracture of steels [6]. For this reason, decrease of the those impurities content is accordingly expected to improve the service properties of pipeline. TMCP is an effective way to improve the mechanical properties for pipeline steels, in which the role of composition and processing parameters (such as soaking temperature, rolling temperature, finishing temperature, cooling rate, etc.) have been widely studied in the past two decades [6-11].

In the present work, the microstructure of a pipeline steel with much lower content of S, P, O and N, after an optimized TMCP, has been deeply studied. It was found that acicular ferrite is dominant in microstructure and a new phenomenon was observed that there are some thin film phases at grain boundaries of acicular ferrite and granular ferrite. The TEM image showed that the thin films are totally different in morphology from the conventional M/A islands formed after acicular ferrite or bainite during a continuous cooling process [10, 11]. The mechanical testing showed there is a good combination of the property with the mixed microstructures of acicular ferrite, polygonal ferrite, granular ferrite and grain boundary thin film.

### 2. Experimental procedure

The experimental steel was melted in a 25kg vacuum induction melting furnace and the chemical composition is shown in Table 1. The ingot was forged into three samples with a size of 150×80×60mm that could be used in the following hot rolling experiment.

The hot rolling experiment was conducted in a laboratory rolling mill in which both rolling and cooling processes can be well controlled. The rolling samples were heated at 1200°C for 60min and then hot rolled to a thickness of 8mm by five passes at a temperature range of 1150°C to 800°C. The finish temperature was at about 900°C (A), 850°C (B), and 820°C (C), respectively. The hot rolled sample was subsequently cooled down to about 400°C at a cooling rate about 30°C /s and then kept at that temperature for 180min.

Table 1

Chemical composition of experimental steel

Element	C	Si	Mn	P	S	Nb	V	N	O
Amount(%)	0.025	0.24	1.56	0.0020	0.0006	0.039	0.019	0.0062	0.0043

The microstructure of samples was examined by using optical microscopy, transmission electron microscopy (TEM) and X ray diffraction (XRD). Thin foils for TEM observation were prepared by a twin jet polisher in an electrolyte containing glacial acetic acid and 10% perchloric acid, and performed in a Philips JEM-200 TEM at 100kV. The XRD samples were prepared by mechanical polishing followed by electropolishing, and carried out in a D/MAX-rB X-ray diffractometer with Cu  $K_{\alpha}$  radiation at 40kV and 100mA.

### 3. Results and Discussions

A typical microstructure of hot rolled plates is given by Fig.1, which shows a mixed microstructure with acicular ferrite, polygonal

ferrite, granular ferrite and a few of thin dark field at grain boundaries that could not identified using optical microscopy (Fig.1a). With lower the finishing rolling temperature, the amount of polygonal ferrite and the dark fields decrease (Fig.1b). Fig.1c shows the magnification of fig.1b. It can be found that the thickness of grain boundaries is inhomogeneous, and some are thicker than the normal grain boundaries, which indicates that there exist some fine phases at grain boundaries. In addition, some faint acicular ferrite was found inside the polygonal ferrite. The mechanical property test showed that the experimental steel has both high strength and toughness (shown in Table 2), and both the strength and toughness increase with decreasing of finishing rolling temperature.

Table 2

Mechanical properties of hot rolled plates

No.	Tensile properties				V-notch impact energy (J)			
	$\sigma_s$ (MPa)	$\sigma_b$ (MPa)	$\delta$ (%)	$\psi$ (%)	28°C	-20°C	-50°C	-80°C
A	515	571	27.5	81.3	342	300	348	433
B	525	588	28.1	84.5	352	348	340	351
C	528	589	28.0	84.6	350	361	354	350

A: finish rolling teperature 900°C; B: finish rolling temperature 850°C; C: finish rolling temperature 850°C

Fig.2 shows the result of TEM analyses. It can be found that the structure is very complex and finer, and some fine ferrite grains are equiaxed (Fig.2a), in which the dislocation

density is very high. There is some needlelike ferrite in the structure (Fig.2b) and some tiny carbide was also observed in the ferrite matrix. A careful survey shows that there are some thin

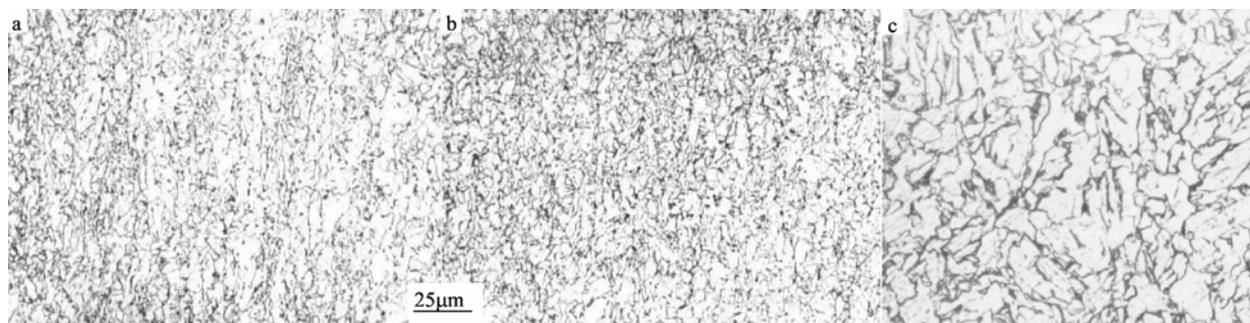


Fig.1. The optical microscopy of hot rolled plates finish rolling temperature 900°C; (b) finish rolling temperature 823°C; (c) magnification of b

films at grain boundaries either of needlelike ferrite or granular ferrite (Fig.2c). Thickness of the films was estimated to be in a range of 10~20nm. Microdiffraction was employed to analyze the structure of grain boundary thin films. Fig.3 shows a photo of the dark field with electron diffraction pattern. Analysis of the diffraction pattern indicates that the thin film is a body-centered cube structure. However morphology of the thin film is different from that of the ferrite matrix. If the current TMCP procedure is taken into account, the thin film found in microstructure should be a martensite film. In order to confirm whether the thin film is martensite or not, a sample that was fast cooled direct to room temperature after hot deformation was examined by using XRD and the result indicated that there is about 1% retained austenite in the sample. But for the sample cooled to 400°C, no retained austenite was found. Therefore, it is reasonable to consider the thin film as the transition product from thin retained austenite to martensite.

It is well known that the microstructure is much complicated after a continuous cooling from austenite temperature for low-carbon microalloy steels, which may contain polygonal ferrite, granular ferrite, acicular ferrite, bainite, pearlite and martensite, etc., under different cooling condition [12, 13]. Under most conditions, acicular ferrite is usually formed, that has good combination of strength and toughness. The acicular ferrite with low angle boundary makes them insensitive to etching.

Therefore fewer boundaries could be detected under optical microscopy. Formation mechanism of acicular ferrite is similar as that of bainite by a combination of diffusion and shear transformation modes, and growth of acicular ferrite generally involves a coherent or semi-coherent  $\gamma/\alpha$  interface and partitioning of carbon. This transformation mode always results in high dislocation density and high strength level for the steel [14].

From the result mentioned above, it can be found that most thin films exist at boundaries of acicular ferrite. This microstructure of acicular ferrite is different from that in references [12, 13], in which the M/A phases distribute as islands. Therefore the acicular ferrite transformation mechanism should be considered as an effect of the deformation at austenite non-recrystallization temperature.

As the acicular ferrite transformation generally involves a coherent or semi-coherent  $\gamma/\alpha$  interface and partitioning of carbon, the effect of deformed austenite on transformation from austenite to ferrite and the morphology of final transformed products should be considered. On one hand the deformed austenite with high density substructure at no-crystallization temperature should impede the growth of coherent or semi-coherent  $\gamma/\alpha$  interface of acicular ferrite and accelerates the diffusion of carbon to  $\gamma/\alpha$  interface, this can result in a formation of carbon enriched austenite films generated at grain boundaries. But on the other hand it also increase the nucleation rates of both

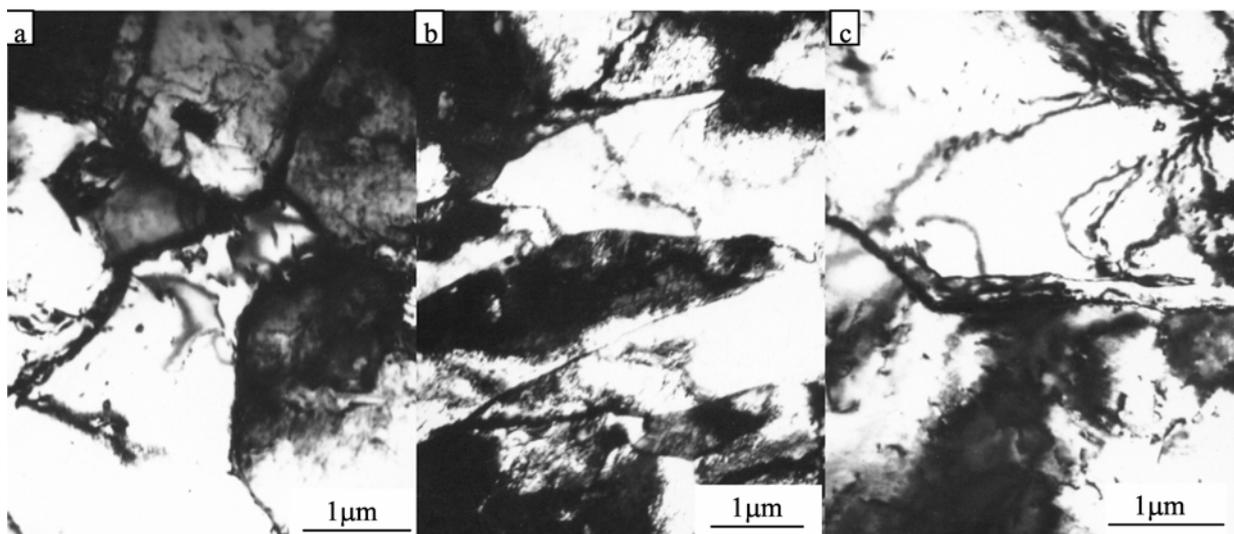


Fig. 2. TEM microstructure of hot rolled plates granular ferrite; (b) acicular ferrite; (c) grain boundary thin films

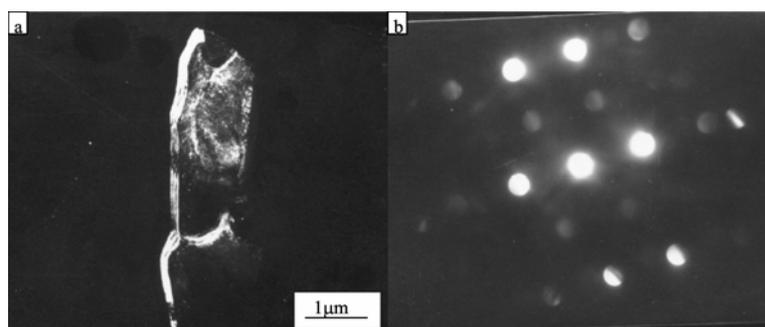


Fig. 3. Electron diffraction analysis of grain boundary thin film (a) dark field; (b) electron diffraction pattern

polygonal and acicular ferrite at the same times, especially, increase intragranular nucleation rates acicular ferrite. So the carbon riched austenite do not reach the content of transformation to ferrite or carbide. Hence, the carbon riched austenite transform to martensite during the isothermal process at 400°C. The martensite thin film should strengthen the grain boundaries and inhibit the crack generation and propagation, which greatly improves toughness of the steel.

#### 4. Conclusions

In summary, a mixed microstructure of acicular ferrite and polygonal ferrite, as well as the martensite thin film at grain boundaries, can be obtained for pipeline steels from heavy hot deformation in the non-recrystallization temperature range and followed by continuous cooling. The grain boundary martensite film is helpful to improve both strength and toughness of the steels.

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