Cause analysis of surface micro-cracks on 12Mn pipeline steel

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Abstract. In order to find out the causes of defects in 12Mn pipeline steel produced by a Chinese steel pipe plant, the characteristics of the defects are analyzed and discussed by means of metallographic microscope, scanning electron microscope, ASPEX scanning electron microscope and transmission electron microscope and so on. The physical and chemical tests results show that there are several typical micro-cracks after slight grinding the pipe outer skin in which a few large particles of brittle alumina reside, which possess the macro-characteristics of continuously curving and extending along the radial direction as well as many clear and smooth inner edges and few secondary branches, so that the cracks are not experienced a long and high-temperature oxidation process. The industrial tests indicate that most of the defects have been proved occurred in the quenching and tempering process of the steel pipe production. The main cause of steel pipe cracking is the stress concentration arose from some large brittle inclusions in the quenching process. When the critical strength of the steel pipe is exceeded by quenching stress, a micro-crack will generate and propagate along the grain boundary. These defects of steel pipes could be eliminated by means of taking steelmaking process optimization measures.

1. Introduction

12Mn pipeline steel is a kind of high-quality tempered pearlite seamless pipeline steel developed by a steelmaking company in China, which has already widely applied in natural gas, oil and other long-distance pipeline projects for the characteristics of high strength, toughness, good weldability and low cost. With the purpose of meeting requirements of microstructure and properties, 12Mn hot-rolled pipe is usually needed to be quenched and tempered [1-3]. However, a large number of steel pipes are found a typical defect which is subcutaneous micro-crack on the pipe outer surface after an ultrasonic flaw detection. They can be delivered normally after surface grinding at the expense of increasing the cost of labor and reducing the efficiency of production.

There has been much literature about the surface defects of steel pipes. The main causes are improper quenching process [4-5], unreasonable piercing process [6] and defects of billet itself, such as surface cracks [7-8], surface scratches [9] and large nonmetallic inclusions [10].To find out the cause concerning defects of 12Mn steel pipe, the characteristics and formation mechanism of defects on 12Mn steel pipe were analyzed through a series of tests, for example, metallography, scanning electron microscope, transmission electron microscope and so on. Furthermore, industrial tests were carried out to verify the analysis results. On this basis, the improved process measures are put forward to eliminate the defects on the outer surface of the finished steel pipe and to improve the quality of product.

2. Experiment scheme

2.1 Experimental material

The experimental material was the hot rolled seamless pipe produced by a steelmaking company in China. The main chemical composition is shown in Table 1. The dimension of finished pipe was Φ 168mm × 10.97mm × 12m. The production process was round billet → inspection → annular heating furnace → piercing process→ hot rolling → magnetic flux leakage testing → quenching and tempering → ultrasonic testing → finishing → warehousing.

Table 1. Main chemical composition (wt. %) of test steel

C	Si	Mn	P	S	N	Alt
0.09 ~ 0.14	0.25 ~ 0.40	1.15 ~ 1.35	≤0.018	≤0.008	≤0.008	0.02 ~ 0.045

2.2 Physical testing and chemical analysis

The defects bearing samples which were cut from the steel pipe with flaw detection alarm were machined into a size of 10 mm × 10 mm × 15 mm by wire cutting and then rubbed crack surfaces with sandpaper before polishing. In order to obtain the microstructures near cracks as well as morphology and composition of inclusions and so on, optical microscope, several detection methods such as ASPEX scanning electron microscope and energy dispersive spectrometer were used to analyze the

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processed samples etched by 4% nitric acid alcohol solution at room temperature for about 2s ~ 5s. After that, several thin layers samples with thickness of about 0.6 mm were cut off from the defects bearing samples before rubbing and polishing to 30 µm by fine grit sandpapers, and then examined by transmission electron microscope.

2.3 Industrial tests

In order to determine the process of cracks causing, the steel pipes were subject to ultrasonic flaw detection immediately after hot rolling, and then quenched by water to room temperature where holding at 850°C for 50 min and tempered by air cooling where holding at 550 ℃ for 75 min before ultrasonic flaw detection once again, as shown in Figure 1. At the same time, it was necessary to carry out the quality tracking and statistic works.

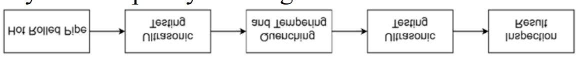

Figure 1. Flowchart of industrial tests

3. Experimental results and discussion

3.1 Macro-characteristics of cracks

Generally, most of the defects on steel surface are not easy to be found by naked eyes after ultrasonic flaw detection. It is observed that there are several typical micro-cracks after slight grinding the pipe outer skin, as shown in Fig. 2 (a). These micro-cracks are mainly linearly distributed under the outer skin of the steel pipes, most of which are in the shape of slender single line, with the length of about 5 ~ 30cm as well as the width of about 30 ~ 100 µm and the depth of generally less than 1mm. Fig. 2 (b) shows the morphology of the polished cross section of a crack bearing sample, which possesses macro-characteristics of continuously curving and extending along the radial direction as well as many clear and smooth inner edges and few secondary branches.

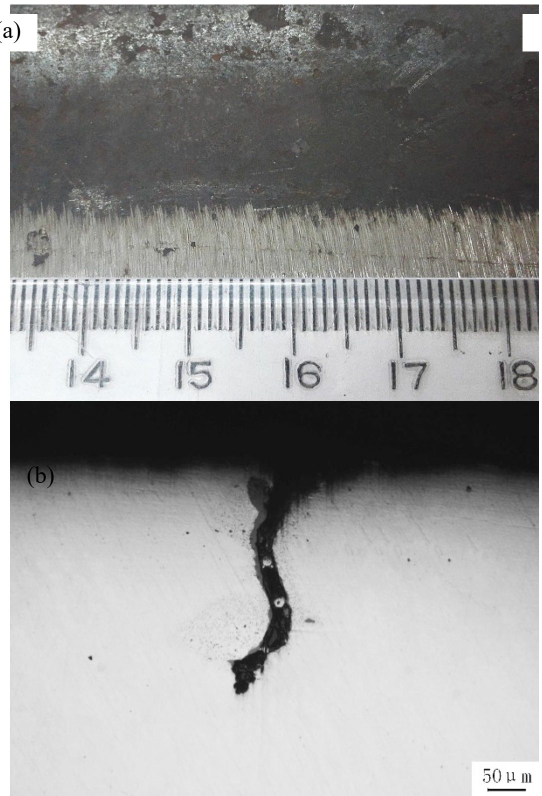

Figure 2. Morphology of typical surface micro cracks (a) after grinding; (b) crack depth

3.2 Micro-characteristics of cracks

3.2.1 Metallographic structure

The defects bearing samples were etched with 4% nitric acid alcohol solution after being polished, the microstructures near a crack are shown in Fig. 3. The ferrite grains are slightly coarse near the crack end region, while relatively uniform near the crack tip region, as shown in Fig. 3 (a), which indicates that the region where a crack is located can bear a strong thermal stress during quenching. There is no obvious decarburization phenomenon on both sides of the crack, which shows that the crack is not experienced a long and high-temperature oxidation process. The extension line of the crack tip could be clearly visible in the area where the crack extends along the radial direction of the steel pipe, as shown in Fig. 3 (b), and the matrixes around the crack tip are considered as tempered pearlites which distribute evenly.

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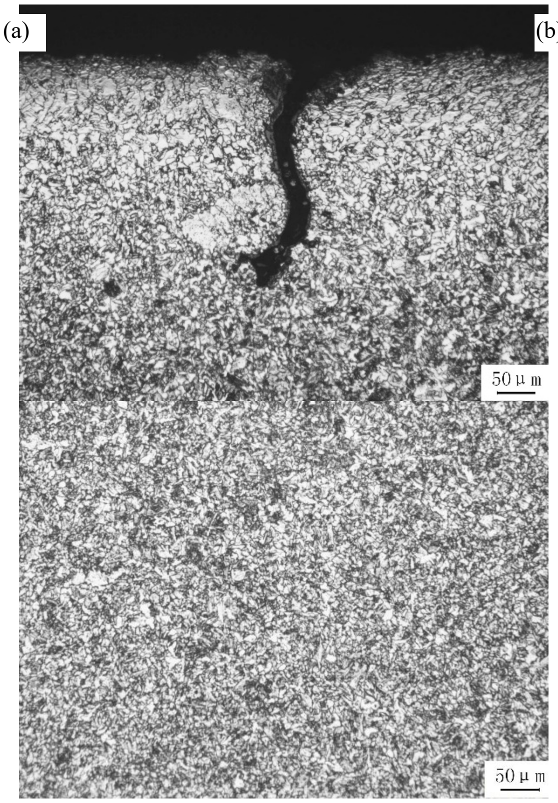


Figure 3. Metallographic structure of surface crack of quenched sample
(a) crack body; (b) crack tip

3.2.2 Morphology and composition of inclusions

Fig. 4 and Tab. 2 describe the morphology and chemical compositions of inclusions in the crack. The inclusions which are in shape of oval and distributed in the middle and tip of the crack are mainly identified as single-phase alumina with the size of 20 ~ 50 μm. The chemical compositions of the inclusions are almost the same, and there has been found a small amount of titanium element in the crack. Generally, alumina inclusions whose size are less than 100 μm are mostly deoxidized products. In addition, they are difficult to float up and to remove by liquid slag in molten steel, and eventually remain in solidified billet. Alumina inclusions, whose sizes are hardly changed during hot deformation, are characterized by brittleness. If the large size of brittle inclusions gathers in steel pipe, stress concentration point can originate from hot deformation or heat treatment process, which is the most important reason for steel pipe cracking.

In the quenching stage of steel pipe production, the surface layer of steel pipe bears the strong dual effects of thermal stress and transformation stress, which are easy to form a large stress concentration around the large-size alumina inclusions. Once the instantaneous stress exceeds the fracture strength of the steel matrix, micro-cracks will form and extend along the low strength grain boundary.

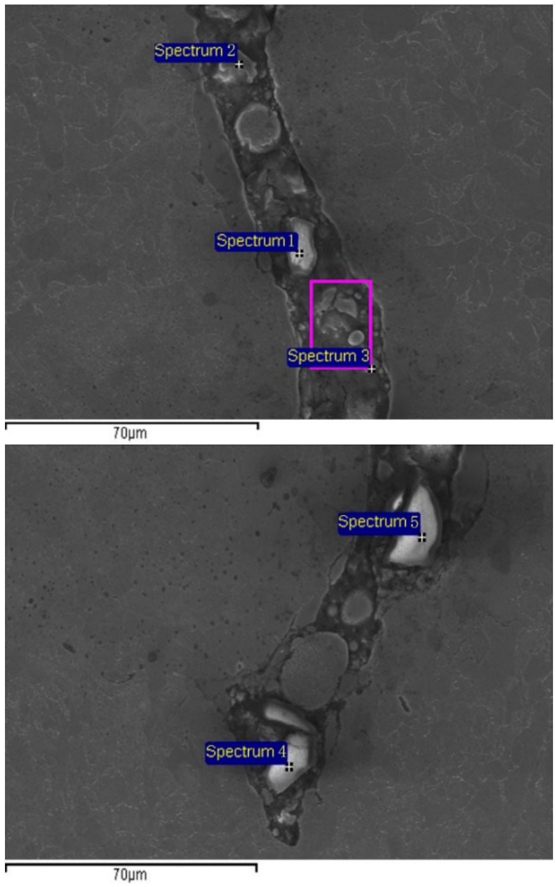


Figure 4. SEM morphology of the inner surface crack of the sample

Table 2. Chemical compositions of inclusions (wt. %)

Spectrum	C	O	Al	Fe
1	/	52.52	46.16	0.88
2	0.54	21.09	/	76.72
3	3.99	27.01	/	64.37
4	0.43	52.37	46.24	0.68
5	0.33	51.64	45.87	0.86

3.2.3 The type and distribution of inclusions.

ASPEX scanning electron microscope was used to detect inclusions on the across section with an area of 24.8 mm2 near the crack, and the size of inclusions is characterized by average diameter. The results show that a total of 1249 inclusions are detected. Fig. 5 (a) shows the main types of inclusions analyzed, in which oxide inclusions account for the highest proportion, followed by sulfide and alumina. Fig. 5 (b) shows the size distributions of the main types of inclusions, it can be seen that most of the inclusions are within 10 μm in diameter, especially that within 2 ~ 4 μm, but there are still a small number of large-size inclusions over 20 μm.

ASPEX can record the coordinate position of inclusions in the detection area, where Y represents the radial direction of steel pipe and X represents the tangent direction of steel pipe circumference. It can be concluded that the distribution of inclusions on cross section of the sample is not uniform, as shown in Fig. 6 and Fig. 7. Many inclusions gather together in an area of the cross section in which there are a few large inclusions scattered, whose size are larger than 30 μm, which contributes to favorable condition for stress concentration during steel pipe quenching.

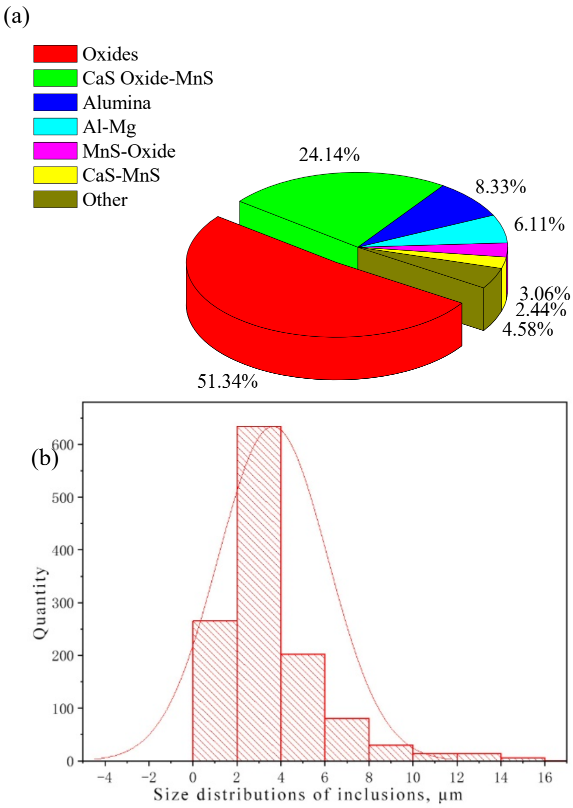


Figure 5. Main types and size distributions of inclusions (a) main types; (b) the histogram of size distributions

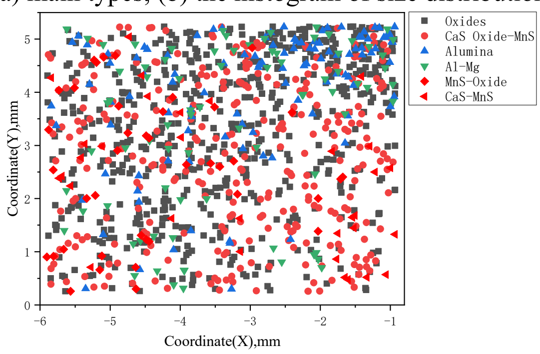


Figure 6. Location distributions of main types of inclusions

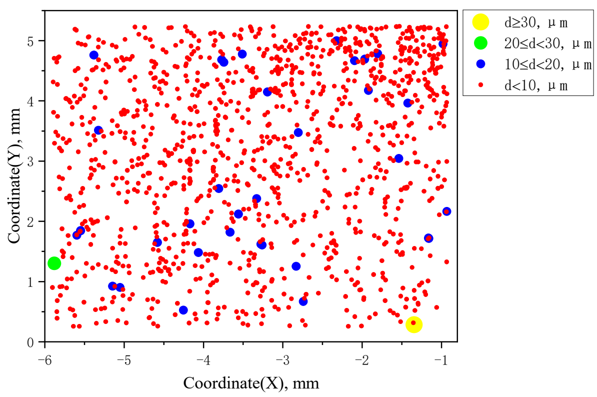
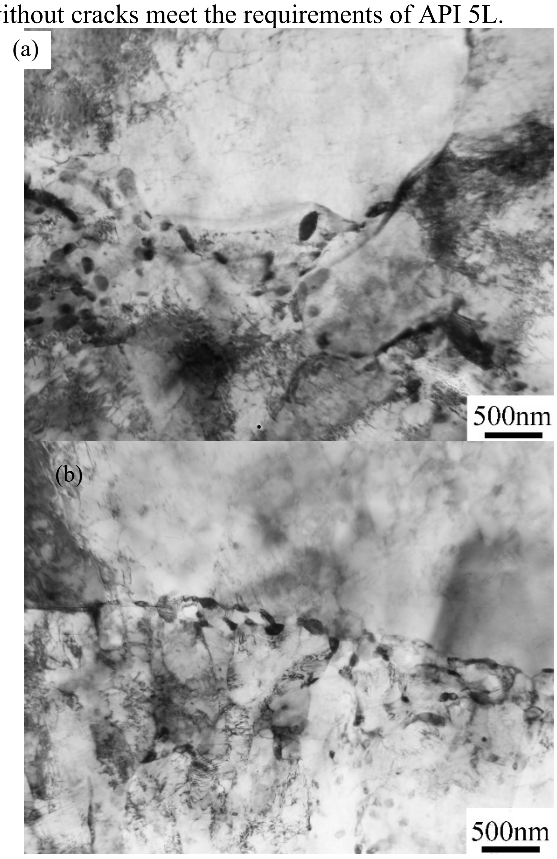


Figure 7. Location distributions of main types of inclusions

3.2.4 *Morphology of precipitates*

Fig. 8 shows a transmission electron microscope photograph of the matrix near a crack, where Fig. 8(a) and Fig. 8(b) show the typical morphology and location of precipitates, most of which are in the shape of spherical with the size of about 100 ~ 300nm, and distributed along the grain boundary. The diffraction pattern of Fig. 8 (d) shows that these precipitates are granular cementite, and that no other types of precipitates are found.

Fig. 9(c) shows the morphologies of ferrite grain distributed near the crack, which have the characteristic of strip shape. This indicates that the recover behaviors of most ferrite grains only occur at 550 ℃ tempering temperature, whose dislocation cells and dislocation lines gradually disappear and dislocation density decreases as well as multilateralization occurs, so that sub-grains are formed. The mechanical mixtures of the recovered ferrites and granular cementite form tempered pearlite, which effectively eliminate the residual internal stress in the steel pipe and obtain good comprehensive mechanical properties. All the properties of tempered steel pipes without cracks meet the requirements of API 5L.



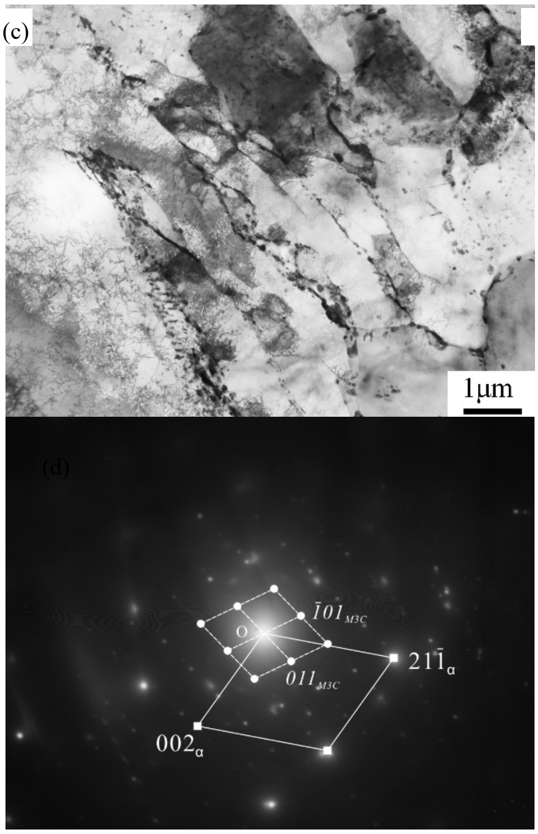


Figure 8. Precipitates near the crack
(a) and (b) the location of the precipitates; (c) grain shapes; (d) diffraction pattern

3.3 Ultrasonic Testing

In order to determine the source process of steel pipes cracking, there were 158 hot rolled pipes selected from the same casting scheme for two industrial tests, where 54 steel pipes were tested in the first time and 104 steel pipes were tested in the second time. Fig. 9 shows the flaw detection results of the industrial tests and indicates that there is 1 unqualified steel pipe occurred after hot rolling process in the first test while 0 unqualified steel pipe in the second test, and 2 unqualified steel pipes occurred after quenching and tempering process in the first test while 11 unqualified steel pipes in the second test.

To sum up, there is a total of 1 unqualified steel pipe occurred after hot rolling process with the defect rate of 0.63% and a total of 13 steel pipes occurred after quenching and tempering process with the defect rate of 8.23% in all 158 steel pipes tested. Therefore, it's almost certain that most of the defects have been proved occurred in the quenching and tempering process, and that almost all of them are considered as micro-cracks under the outer skin of the steel pipes.

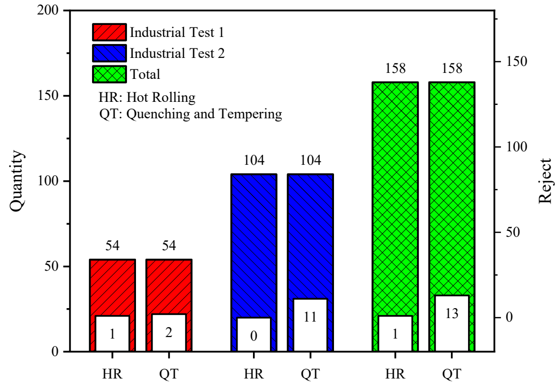


Figure 9. Industrial test results

3.4 Discussion

All the defects which are not easy to be directly observed by naked eyes and almost considered as tiny and slender micro-cracks under surface skin occur under the outer skin of the steel pipes. Microstructures of the cross section along the radial extension direction near the crack are distributed nonuniformly, where ferrite grains near the crack end area grow coarser than that of crack tip area and a few large alumina brittle inclusions reside in the crack. There is no significant difference between microstructures of both sides of the crack and that of the tip area. With few secondary branch cracks and no traces of high temperature oxidation, the inner surface edge of the crack is generally smooth and has no obvious decarburization phenomenon which is the typical characteristic of quenching crack [11-12], so that the micro-pores around the inclusions are not directly exposed to air during the process of deforming and rolling. The industrial flaw detection test results directly show that most cracks bearing steel pipes are found in the quenching and tempering stage of the production process, which proves that the micro-cracks are produced in the steel pipe quenching process. Due to the existence of brittle inclusions as well as the absence of oxidation and decarburization phenomenon in the cracks, the main cause of steel pipe cracking is the stress concentration arose from some large brittle inclusions in the quenching process. When the critical strength of the steel pipe is exceeded by quenching stress, a micro-crack will generate and propagate along the grain boundary.

In order that increasing cleanliness of steel billets should be effective it must be planned systematically. The total oxygen content in steel can be more stringent controls for decreasing large inclusions on optimizing the steelmaking process, such as adjusting soft blowing time, improving protective casting, avoiding slag entrapment, using a new type tundish covering agent and so on. The subcutaneous micro-cracks of steel pipes have been eliminated since taking steelmaking process optimization measures, so that the product quality and productivity of steel pipes are significantly improved.

4. Conclusion

(1) There are several typical micro-cracks after slight grinding the pipe outer skin, which possess macro-characteristics of continuously curving and extending along the radial direction as well as many clear and smooth inner edges and few secondary branches and are mainly linearly distributed under the outer skin of the steel pipes, most of which are in the shape of slender single line, with the length of about 5 ~ 30cm as well as the width of about 30 ~ 100 μm and the depth of generally less than 1mm.
(2) The region where a crack is located can bear a strong thermal stress during quenching. The crack is not experienced a long and high-temperature oxidation process. The extension line of the crack tip could be clearly visible in the area where the crack extends along the radial direction of the steel pipe, and the matrixes around the crack tip are considered as tempered pearlites which distribute evenly.
(3) The inclusions which are in shape of oval and distributed in the middle and tip of the crack are mainly identified as single-phase alumina with the size of 20 ~ 50 μm. Many inclusions gather together in an area of the cross section in which there are a few large inclusions scattered, whose size are larger than 30 μm, which contributes to favorable condition for stress concentration during steel pipe quenching. Most of precipitates are in the shape of spherical with the size of about 100 ~ 300nm, and distributed along the grain boundary. The diffraction pattern shows that these precipitates are granular cementite, and that no other types of precipitates are found.
(4) There is a total of 1 unqualified steel pipe occurred after hot rolling process with the defect rate of 0.63% and a total of 13 steel pipes occurred after quenching and tempering process with the defect rate of 8.23% in all 158 steel pipes tested. Therefore, it's almost certain that most of the defects have been proved occurred in the quenching and tempering process, and that almost all of them are considered as micro-cracks under the outer skin of the steel pipes.
(5) The main cause of steel pipe cracking is the stress concentration arose from some large brittle inclusions in the quenching process. When the critical strength of the steel pipe is exceeded by the quenching stress, a micro-crack will generate and propagate along the grain boundary. These defects of steel pipes could be eliminated by means of taking steelmaking process optimization measures

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