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Development of 500MPa grade environmental friendly automobile structural steel

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Abstract. The microstructure, properties and oxide scale of the 500MPa grade environmental-friendly automobile structural steel were investigated by utilizing optical microscope (OM), transmission electron microscopy (TEM) and X-ray diffraction (XRD). The results show that the “black ash” was consisted of massive oxide scale and a small amount of powdered oxide scale, the phase proportion of FeO, Fe₃O₄, and Fe₂O₃ were 41.9%, 55.6% and 2.5%, especially. By reasonable controlling of the proportion and morphology of FeO and Fe₃O₄ in the structure of oxide layer can prevent oxide scale peeling. The process of “fast rolling at high temperature” combined with lower-temperature coiling could obtain the optimal oxide scale structure, which is thin and consisted of lamellar Fe₃O₄/Fe eutectoid transformation product, and with a small amount of FeO distributed uniformly and island-like in Fe₃O₄. The mechanical properties and the surface quality can meet the user's requirements when the finish rolling temperature and coiling temperature were 900 °C and 570 °C, the thickness of the oxide scale was less than 7.5μm, the proportion of FeO, Fe₃O₄ and Fe₂O₃ were 7.0%, 86.2% and 6.8%, especially.

1. Introduction

With the development of steel production technology, China's steel industry has reached the international standards in terms of equipment condition, dimensional accuracy and performance of steel. However, China has not systematically studied the surface quality of hot-rolled steel for a long time. The surface quality of hot-rolled strip is frequently problematic, such as the iron oxide scales is not easy to remove, iron oxide scale is easy to fall off, exist red rust on the surface and pickling defect and so on, which seriously hindered the improvement of the quality of steel products. Both the surface quality and mechanical properties have been important and long-term issues in industrial companies, especially for automobile companies. Therefore, many steel mills have made greater efforts to improve the surface quality of the strip to meet customers' demands [1-4].

Because the strips are exposed to not only high temperature but also a humid atmosphere, surface oxidation of the steel is unavoidable. Since the oxide layer is removed with a high-pressure water spray before the strip enters the finishing rolling, controlling the oxide growth in the finishing rolling mill and laminar cooling process is very important. It is well known that the oxide scales on steel composed of three layers: an outermost layer of hematite (Fe₂O₃), an intermediate layer of magnetite (Fe₃O₄) and a thick inner layer of wustite (FeO) [5-6]. Fe₃O₂ is rufous and desquamating easily; Fe₃O₄



is dark blue and desquamating difficultly; FeO is loose and porous, it is easy to fall off. The oxide scales usually used pickling before stamping, with the requirement of environmental protection and energy saving, more and more automobile companies are stamping directly without pickling, which will result in environment pollution, mold damage, and cause scratch defects of the structure parts. Thus, it is important to develop a new steel product with an excellent adhesion between oxide layer and matrix to meet the requirement of no scale off stamping.

The objective of this research is to development a 500MPa grade automobile structure steel with an excellent surface quality in the mill line. To study the effect of hot rolling process parameters on the growth behavior of the oxide layer.

2. Material and experimental procedures

2.1. Composition

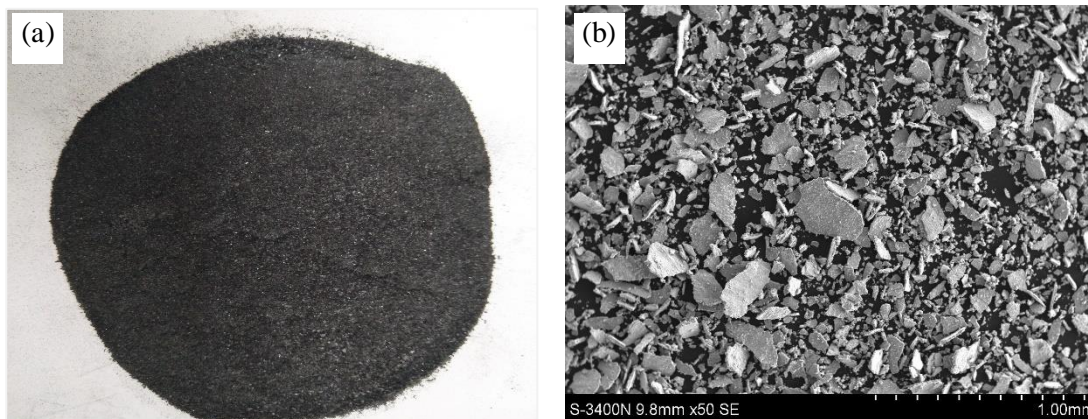
In order to obtain excellent fatigue properties and cold formability, it is compatible with the addition of Nb and Ti microalloys in the low carbon manganese steel. The chemical composition of the tested steel is shown in Table 1.

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

C	Si	Mn	P≤	S≤	Nb	Ti
0.05~0.08	0.10~0.15	1.4~1.6	0.010	0.008	0.03~0.06	0.01~0.03

2.2. “Black ash” defect

Automobile structural products with conventional process have a large amount of surface oxide scale falling off during the stamping process, which called for “black ash” defect. The macro and micro images and the Electronic Data Systems (EDS) analysis of “black ash” were shown in Figure 1 and table 2. It can be seen from the results that the “black ash” consists of two forms of oxide scale, which mainly composed of massive oxide scale and with a small amount of powdered oxide scale. The thickness of the peeled iron oxide is about 9μm. The Fe element in the oxide scale accounts for 60~66%, and the O element accounts for 32~38%.



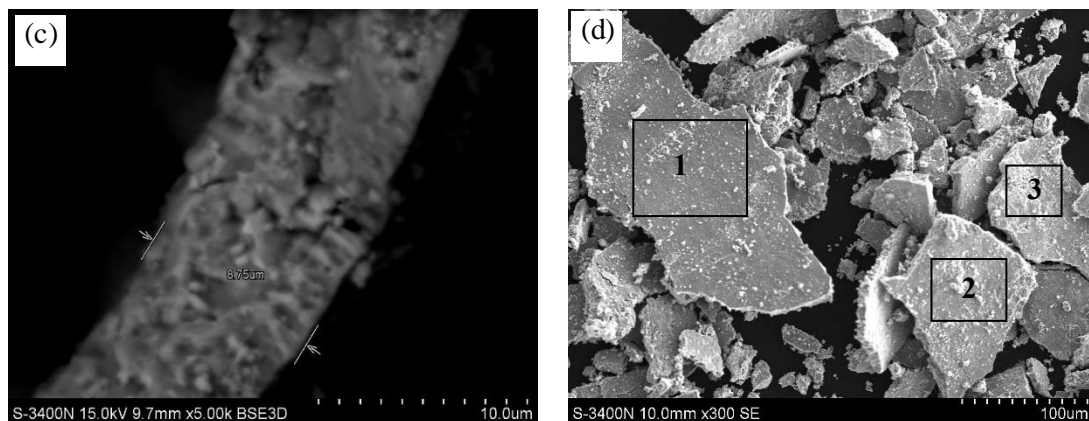


Figure 1. The Macro and micro images of the “black ash” defect (a) Macro image, (b) Micro image, (c) Thickness of oxide scale, (d) Location of EDS analysis.

Table 2. The EDS analysis of the “black ash”.

Spectrum	O	Al	Si	Mn	Fe	Total
1	33.52			0.58	65.90	100.00
2	32.94	0.20	0.29	0.82	65.75	100.00
3	37.22	0.27	0.24	0.88	61.39	100.00

The phase of “black ash” was studied by X-ray diffraction analysis, the result is shown in table 3. It can be seen that the phase of the “black ash” is composed of Feo, Fe_3O_4 , and Fe_2O_3 . The Fe_2O_3 account for 2.5%. FeO and Fe_3O_4 account for 41.9% and 55.6%, especially.

Table 3. The phase analysis result of “black ash”.

Phase Content/%	FeO	Fe_3O_4	Fe_2O_3
	41.9	55.6	2.5

2.3. Industrial trial production

The 500MPa grade automobile structure steel with anti-stripping oxide scale structure was test in the commercial mill line. The final product thickness chose 8mm, the soaking temperature was 1200 °C, the rough rolling temperature(RT2) was 1050 °C and 1000 °C, the pressure of descaling water was more than 18MPa, the finish rolling temperature(FDT) were 840 °C and 900 °C, the coiling temperature(CT) was 600 °C and 570 °C. The hot rolling process is shown in table 4.

Table 4. The hot rolling process of the tested steel.

Process	Soaking temperature/ °C	Rough rolling temperature/ °C	Finish rolling temperature/ °C	Coiling temperature/ °C	F7 Strip speed/m/s
1	1200	1050	840	600	≥4
2	1200	1000	900	570	≥6

3. Material and experimental procedures

3.1. Properties

The yield strength, tensile strength, and elongation data obtained from tensile tests of the tested steel under different process are listed in Table 5. When the finish rolling temperature and coiling

temperature were 840 °C and 600 °C, the yield strength, tensile strength, elongation and yield ratio were 565MPa, 615 MPa, 26.5% and 0.918, respectively. When the finish rolling temperature and coiling temperature were 900 °C and 570 °C, both the strength and the elongation were decreased, the yield strength and tensile strength reduced to 540MPa and 598MPa, respectively, the elongation and yield ratio reduced to 25.5% and 0.903, respectively. Based on the analysis above, it can be seen that the reduction of the finish rolling temperature was beneficial to the improvement of the comprehensive mechanical properties, which under the traditional process with low finish rolling temperature and high coiling temperature are optimal.

The cold forming performance of the tested steel and the adhesion of the oxide scale were evaluated by the 180° cold bending test, the width of the cold bending sample was 35mm. The results showed that the tested steels under different process have a good cold forming performance. It has a little oxide scale desquamates from cold bending samples when the finish rolling temperature and coiling temperature were 840 °C and 600 °C, while no oxide scale desquamates when the finish rolling temperature and coiling temperature were 900 °C and 570 °C.

Table 5. Mechanical properties, formability, adhesion performance of the tested steel.

Process	Yield strength	Tensile strength	Elongation	180° Cold bending	
	/ MPa	/MPa	/ %	Formability	Adhesion
1	565	615	26.5	OK	Little
2	540	598	24.5	OK	No
Standard	≥500	550~570	≥22.0	OK	No

3.2. Microstructure

Figure 2 shows the optical microstructure (OM) images of the tested steel with different finish rolling temperatures and coiling temperatures. The microstructure was composed of ferrite (bright phase). When the finish rolling temperature and coiling temperature were 840 °C and 600 °C, the ferrite was polygonal ferrite, the grain boundary was smooth and straight, as shown in Figure 3a. When the finish rolling temperature and coiling temperature were 900 °C and 570 °C, the microstructure was also composed of ferrite, while the uniformity was worse than conventional rolling process, and it has banded tissue. It can be seen that the low finish rolling temperature was favorable for the refinement and uniformity of ferrite grain.

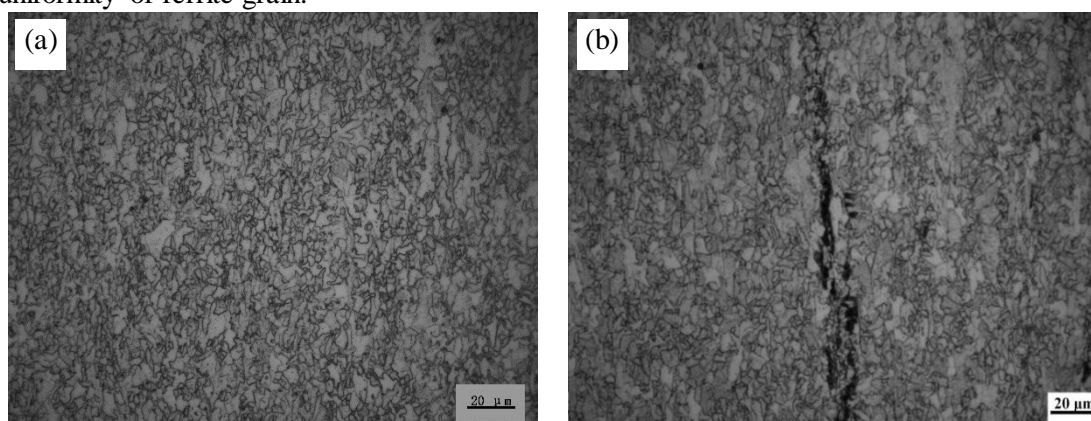


Figure 2. OM images of the tested steel with different finish rolling temperature and coiling temperature (a) 840 °C, 600 °C; (b) 900 °C, 570 °C

3.3. Oxide scale

Figure 3 shows the image and thickness of the tested steel analyzed by scanning electron microscope, the results showed that the morphology and thickness of oxide scale under different processes is

obviously different. When the finish rolling temperature and coiling temperature were 840 °C and 600 °C, the oxide layer was loose and porous, the thickness were about 9 μm . When the finish rolling temperature and coiling temperature changed to 900 °C and 570 °C, the oxide layer was closely, the thickness was reduced to less than 7.5 μm , and the eutectoid transformation of FeO was very sufficient.

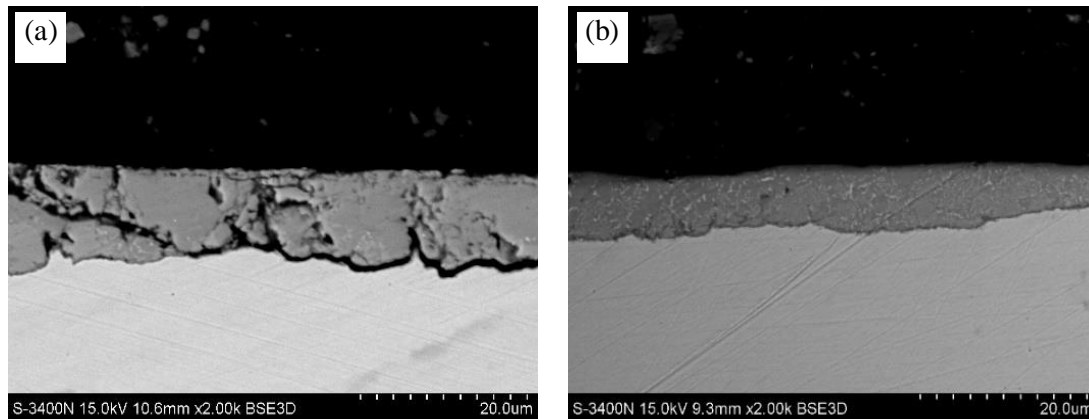


Figure 3. Morphology and thickness of oxide scale under different processes temperature (a) 840 °C, 600 °C; (b) 900 °C, 570 °C

The phase of the oxide scale of the tested steel under different process studied by X-ray diffraction analysis was shown in table 6. It can be seen that the phase of the oxide scale under different finish rolling temperature and coiling temperature were made up of FeO, Fe_3O_4 , and Fe_2O_3 , while their proportions were obviously different. When the finish rolling temperature and coiling temperature were 840 °C and 600 °C, the proportion of FeO, Fe_3O_4 and Fe_2O_3 were 45.8%, 53.4% and 0.9%, respectively. When the finish rolling temperature and coiling temperature were 900 °C and 570 °C, the proportion of Fe_3O_4 was more than 86%, while the FeO and Fe_2O_3 only account for 7.0% and 6.8%, especially.

Table 6. The phase the oxide scale of the tested steel under different process.

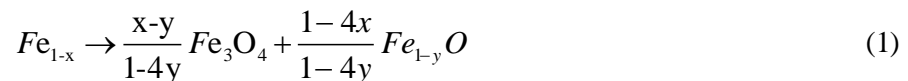
Process	Content/%		
	FeO	Fe_3O_4	Fe_2O_3
1	45.8	53.4	0.8
2	7.2	86.0	6.8

4. Material and experimental procedures

The optimal mechanical properties were obtained when the finish rolling temperature and coiling temperature were 840 °C and 600 °C, respectively, while the adhesion and thickness of oxide scale were not well, it was easy to fall off when forming. In order to improve the quality of oxide scale, the thickness of the oxide layer need to reduced, and the proportion of Fe_3O_4 need to increase.

According to the Fe-O phase diagram, FeO has a much higher oxygen content at higher temperatures (850 °C ~1000 °C) than at lower temperatures (350 °C ~550 °C). 570 °C is the eutectoid transformation of FeO. Researchers showed that Fe_3O_4 precipitates before the eutectoid reaction began, and no Fe precipitates was found before the eutectoid reaction began. The oxide layer containing mainly residual FeO and containing some pro-eutectoids when cooling rapidly, while composed of a mixture of Fe_3O_4 and Fe when cooling slowly. For the coiling temperature range of 350 °C ~600 °C, the oxygen content of FeO decreases sharply, and the FeO is unstable. When cooling to low temperature to coiling, the oxygen content in the FeO layer gradually reaches the supersaturation state. Pre-

eutectoid reaction formed by FeO near the original Fe_3O_4 layer, the reaction is as shown in formula 1. At the same time, with the decrease of temperature, the oxygen supersaturation in the FeO layer is further increased, and the driving force of the eutectoid reaction increases. With the precipitation of the pre-eutectoid Fe_3O_4 , a relatively oxygen-poor zone is formed around the pre-eutectoid Fe_3O_4 . A relatively oxygen-rich zone is formed at relatively far away. For the FeO which does not undergo the pro-eutectoid reaction was formed an iron-rich region. When the temperature drops to a critical temperature, the formation of elemental Fe nucleus begins to appear, and at the same time, Fe_3O_4 appears in the oxygen-rich region, which form a crystal nucleus of the eutectoid reaction product. The nucleation of the eutectoid reaction product continues to grow and finally a lamellar Fe_3O_4 /Fe eutectoid transformation product is formed, the eutectoid reaction is as shown in Formula 2[7~10].



The failure stress of FeO is about 0.4 MPa, the failure stress of Fe_3O_4 is about 40 MPa. Fe_3O_4 is good for maintaining the adhesion between the oxide scale and the substrate. However, if the majority of the oxide layer is the pre-eutectoid Fe_3O_4 , it is easy to be broken during the straightening process to form a powder [11~12].

When there was an external force, the micro-cracks of the oxide scale extend. When extending to FeO which containing a large amount of defects or lamellar Fe_3O_4 /Fe eutectoid transformation product, the diffusion of micro-cracks is terminated. Therefore, the optimal oxide scale structure is lamellar Fe_3O_4 /Fe eutectoid transformation product with a small amount of FeO exists, and FeO has a uniform island-like distribution in Fe_3O_4 . This is mainly due to the combination of soft and hard phases, which can effectively absorb the damage stress caused by straightening and avoids the breakage of the scale. Therefore, reasonable controlling of the proportion and morphology of FeO and Fe_3O_4 in the structure of oxide layer can achieve the purpose of controlling the peeling of oxide scale.

As the rolling speed increases, the oxidation time in the high temperature decreases, and the thickness of the oxide scale decreases. Therefore, the rough rolling temperature should be low and the finish rolling temperature should be high to obtain a high speed. It can be seen from the above that reasonable control of the coiling temperature to achieve the ideal control of the oxide scale structure is the key to avoid falling powder. When the coiling temperature was too low would affect the plasticity of the product. Based on ensuring the mechanical properties of the product, combined with controlling the structure of the oxide scale, the coiling temperature was set at about 570 °C. The mechanical properties and the surface quality can meet the user's requirements under process 2.

5. Material and experimental procedures

- 1) The “black ash” was mainly consisted of massive oxide scale and with a small amount of powdered oxide scale. The phase of the “black ash” is composed of Feo, Fe_3O_4 , and Fe_2O_3 , their proportion were 41.9%, 55.6% and 2.5%, especially.
- 2) The optimal mechanical properties were obtained when the finish rolling temperature and coiling temperature were 840 °C and 600 °C, while existing oxide scale peeling off. There were no oxide scale desquamates when the finish rolling temperature and coiling temperature were 900 °C and 570 °C.
- 3) When the finish rolling temperature and coiling temperature were 840 °C and 600 °C, the thickness were about 9μm, the proportion of Feo, Fe_3O_4 and Fe_2O_3 were 45.8%, 53.4% and 0.9%, respectively. When the finish rolling temperature and coiling temperature changed to 900 °C and 570 °C, the thickness was reduced to less than 7.5μm, the proportion of Feo, Fe_3O_4 and Fe_2O_3 were 7.0%, 86.2% and 6.8%, especially.
- 4) Reasonable controlling of the proportion and morphology of FeO and Fe_3O_4 in the structure of oxide layer can prevent oxide scale peeling. The optimal oxide scale structure is lamellar

$\text{Fe}_3\text{O}_4/\text{Fe}$ eutectoid transformation product with a small amount of FeO exists, and FeO has a uniform island-like distribution in Fe_3O_4 .

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