Effect of Al_2O_3 on Evolution of Oxide Inclusions in Tire Cord Steel during Hot Rolling

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The evolution of oxide inclusions with different concentrations of Al_2O_3 in tire cord steel during hot rolling was investigated by industrial trials using an automatic scanning electron microscope. Changes in chemical composition, particle size, and number density of oxide inclusions in the entire hot rolling process were studied with systematical samplings. The maximum diameter of most inclusions is less than 5 μm , a value independent of the Al_2O_3 concentration; the number density of oxide inclusions increases with the increase of Al_2O_3 , when this concentration is lower than 50%. The average chemical composition of oxide inclusions changed little during the hot rolling process when the concentration of Al_2O_3 was approximately 35%. Deformation and fracture of oxide inclusions occur simultaneously during the entire hot rolling process causing changes in the average diameter, number density and area fraction. Higher deformation in oxide inclusions was observed when the concentration of Al_2O_3 was in the range of 20%–25%. This behavior was explained due to a decrease in their melting point.

KEY WORDS: inclusions; hot rolling; evolution; deformation.

1. Introduction

Tire cord steel is widely used in tires. It is made by twisting the wire rods after a high-speed cold drawing process. Oxide inclusions in tire cord steels are one of the main reasons for wire fracture during the drawing process.^{1–4)} The requirements of higher strength and smaller diameter in tire cord steel demand wire rods with higher cleanliness as well as with excellent drawability.

Tire cord steels is typically a silico-manganese deoxidized steel. Because the ferroalloy used in actual production contains a certain amount of impurity elements together with the influence of refractory and slag, the composition of inclusions in molten steel will inevitably change and almost all inclusions in tire cord steels correspond to the $\text{SiO}_2\text{--MnO--Al}_2\text{O}_3\text{--CaO}$ system.^{2–5)} In view of the fact that it is impossible to remove the inclusions completely and, on the other hand, deformable inclusions with low melting point are beneficial for the drawability of steel wires, more researches^{2,6–15)} are focused on the influence of liquid steel composition, refining slag, and refractories on inclusions of $\text{SiO}_2\text{--MnO--Al}_2\text{O}_3\text{--CaO}$ system, which is expected to control the inclusions in the low melting point regions.

Considering the transformation of oxide inclusions dur-

ing hot rolling, a large number of experimental investigations^{5,16–25)} have been reported. Iwai²⁶⁾ proved that decreasing the melting point and a more homogeneous chemical composition, increased the degree of deformation of the inclusions. Kimura²⁷⁾ suggested that all the oxide inclusions (alumina, zirconia, zircon and silica) were fractured during hot rolling and its magnitude could be predicted from the Young's modulus and the mean atomic volume of the oxides. Wang^{5,28)} found that the formation of SiO_2 -type inclusions in wire rods was due to the precipitation of SiO_2 in the formed $\text{MnO--SiO}_2\text{--Al}_2\text{O}_3$ inclusion during casting and then separated during rolling. Zhang *et al.*^{29,30)} proposed that deformation is related to the chemical composition and values of the Young's module for the inclusions. Deformation increases by decreasing the Young's module. However, there are few investigations on the transformation mechanism in the $\text{SiO}_2\text{--MnO--Al}_2\text{O}_3\text{--CaO}$ system with different content of Al_2O_3 during the industrial production of tire cord steels.

In this paper, the changes in chemical composition, particle size and number density of inclusions in the $\text{SiO}_2\text{--MnO--Al}_2\text{O}_3\text{--CaO}$ system during hot rolling of industrial production was analyzed, and the influence of Al_2O_3 content on the deformation of oxide inclusions in tire cord steels during hot rolling was discussed.

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2. Experimental Procedure

The experimental work was carried out in a steel plant producing tire cord steels. The process consisted of the following stages: “Basic Oxygen Furnace (BOF) → Ladle Furnace (LF) refining → Continuous Casting (CC)”. During tapping from the BOF, ferroalloys were added into molten steel for deoxidizing and alloying and then the molten steel was transferred to the refining (LF) station. Low basicity pre-melted slag (CaO/SiO_2 approximately 1.0) was used and molten steel was stirred by bottom argon blowing with a proper flow during the LF refining process. The pre-melted slag with fixed chemical composition was added during the LF process. The weight and composition of slag in different heats changed little. The slag composition in the refining process of different heats is shown in **Table 1**, differences in slag chemical composition from different heats was negligible.

After a certain soft blowing time, the molten steel that met the temperature and composition requirements was transported to the continuous casting platform, which had protective measures to prevent reoxidation of liquid steel. The differences in total aluminum for the three heats shown in **Table 2** occur as a consequence of small variations in the chemical composition of SiMn, this in turn affected changes in the Al_2O_3 content in the billets obtained ($150 \times 150 \text{ mm}^2$). The contents of total oxygen (T.O) in the steel were measured using N/O analyzers, the concentration of soluble aluminum (Als) was measured by inductive coupled plasma-mass spectrometer (ICP-MS).

The billets were then hot rolled, at the rolling temperature of 950–1 050°C, into wire rods with $\phi 5.5 \text{ mm}$ in diameter through dozens of passes during the rolling process. Steel

samples were taken from billets and rods with diameters of $\phi 75$, $\phi 33$, $\phi 17$, and $\phi 5.5 \text{ mm}$ in different rolling stages, schematically shown in **Fig. 1**, defined as R1, R2, R3 and R4, respectively. Inclusions were evaluated from the section at 1/4 of the thickness of billets from the loose side along the casting direction. The longitudinal section at the center of the cross section of wire rod ($\phi 5.5 \text{ mm}$) was investigated, for the other diameters the sampling position was located at one-quarter of the diameter. Approximately 20 mm^2 for each sample were examined using an automatic SEM apparatus after grinding and mirror polished, in order to obtain the following information about non-metallic inclusions; chemical composition, number density, particle size, and morphology during the hot rolling process. The minimum, maximum, average diameters and area of the inclusions were measured.³⁰⁾ Particularly, the inclusions with a diameter larger than $1 \mu\text{m}$ were taken into account during the detection.

3. Oxide Inclusions in Billet

As already mentioned, almost all inclusions in tire cord steels correspond to the $\text{SiO}_2\text{--Al}_2\text{O}_3\text{--CaO--MnO}$ system. The oxide inclusions were classified into two systems: if the mass fraction of CaO was larger than that of MnO, they were assigned to the $\text{SiO}_2\text{--CaO--Al}_2\text{O}_3$ system otherwise it was classified as part of the $\text{SiO}_2\text{--MnO--Al}_2\text{O}_3$ system. This division was made in order to simplify its study using ternary phase diagrams. The compositions of oxide inclusions in the billets for different heats are shown in **Fig. 2**. The size of circles in Fig. 2 indicates the maximum diameter (D) of inclusions. The symbol D_{max} and D_{min} in Fig. 2 indicate the maximum and minimum values, respectively. The red stars represent the average chemical composition. The liquidus in the diagram was calculated by FactSage7.2. The distribution of oxide inclusion composition in different heats showed significant differences, the average Al_2O_3 composition of inclusions in heats 1, 2 and 3 was 34.29%, 23.48% and 7.37%, respectively. In Heat 1, most of the oxide inclusions were in the $\text{SiO}_2\text{--CaO--Al}_2\text{O}_3$ system with approximately 40% of Al_2O_3 , the other inclusions were in $\text{SiO}_2\text{--MnO--Al}_2\text{O}_3$ system and contained approximately 35% of Al_2O_3 . As for Heat 2, most inclusions belonged to the $\text{SiO}_2\text{--MnO--Al}_2\text{O}_3$ system, and the average content of Al_2O_3 dropped below 30%. However, in Heat 3, almost all oxide inclusions were in the $\text{SiO}_2\text{--MnO--Al}_2\text{O}_3$ system with the average content of Al_2O_3 less than 15%. In addition, the location of oxide inclusions in Heat 1 and Heat 2 were closer to the 1 873 K liquidus while the oxide inclusions in Heat 3 kept away from low melting regions.

The size distribution of inclusions in different heats is

Table 1. Composition of slag in different heats (Mass percentage).

Heat	CaO	SiO ₂	MgO	MnO	Al ₂ O ₃	FeO	CaF ₂
1	40.10	38.95	7.91	4.18	3.08	4.56	1.21
2	38.73	41.32	4.75	6.80	3.40	3.75	1.26
3	39.28	39.48	5.74	9.03	3.20	2.09	1.18

Table 2. Composition of tire cord steels in different heats (Mass percentage).

Heat	C	Si	Mn	P	S	Al _L	Al _S	T.O	Fe
1	0.72	0.20	0.52	0.007	0.005	0.0009	0.0005	0.0014	balance
2	0.73	0.22	0.52	0.008	0.007	0.0007	0.0005	0.0015	balance
3	0.74	0.21	0.55	0.011	0.003	0.0005	<0.0005	0.0016	balance

Fig. 1. Schematic of hot-rolling process.

Fig. 2. Composition of oxide inclusions in the billet of different heats: (a) Heat 1, (b) Heat 2, (c) Heat 3. (Online version in color.)

shown in Fig. 3. The number density of oxide inclusions in Heat 1 was the largest, and the number density of oxide inclusions with maximum diameter in the range of 1–2 μm and 2–3 μm was close to 10/mm². As the maximum diameter increased, the number density gradually decreased and was even lower for inclusions larger than 5 μm . Compared with Heat 1, the number density of oxide inclusions in Heat 2 decreased significantly. Although the maximum diameter of oxide inclusions was also in the range of 1–4 μm , the proportion of oxide inclusions with the maximum diameter in the range of 2–3 μm was higher. The number density of oxide inclusions in Heat 3 was even lower than those of heat 1 and 2. The maximum diameter of oxide inclusions was in the range of 2–3 μm with a number density less than 3/mm². Comparing the maximum diameter distribution of oxide inclusions of billets in different heats, it was found that this value is lower than 5 μm . According to Fig. 2, decreasing

the concentration of Al₂O₃ in oxide inclusions, the number density also decreased.

4. Evolution of Oxide Inclusions during Hot Rolling

The transformation in the average composition of oxide inclusions during hot rolling is shown in Fig. 4. Figure 4(a) shows the transformation in the average composition of oxide inclusions during the hot rolling in Heat 1. During the whole rolling process, the content of SiO₂ and Al₂O₃ fluctuated slightly within the range of 35%–40%. As the rolling progressed, the content of MnO showed a downward trend, while the content of CaO increased first and then decreased. Figure 4(b) shows the transformation in the average composition of oxide inclusions of Heat 2. Different from Heat 1, the content of SiO₂ in oxide inclusions is the highest with a mass fraction that exceeded 50%. During the subsequent

Fig. 3. Maximum diameter distribution of oxide inclusions in the billet of different heats.

Fig. 4. Variation of average composition of oxide inclusions in the billet of different heats: (a) Heat 1, (b) Heat 2, (c) Heat 3. (Online version in color.)

rolling process, the content of SiO_2 gradually increased from 50% to approximately 65%; the content of Al_2O_3 in oxide inclusions gradually decreases from 23% to 18% in the

rolling process, and the contents of MnO and CaO fluctuated from 10% to 15%. The transformation in the average composition of oxide inclusions of Heat 3 is shown in Fig. 4(c), the content of SiO_2 in oxide inclusions was close to 70%. During the entire rolling process, the content of SiO_2 decreased first and then increased, and the content of MnO increased first and then decreased; the content of Al_2O_3 in the oxide inclusions continued to decrease throughout the rolling process, while there was no obvious change in the contents of MgO and CaO due to the low content during the whole rolling process.

The typical morphologies of some large oxide inclusions at different stages of hot rolling in different heats are shown in Fig. 5. As shown in Fig. 5, oxide inclusions of $\text{SiO}_2\text{--MnO--CaO--Al}_2\text{O}_3$ system with different content of Al_2O_3 in tire cord steel were deformed or broken along the rolling direction during the hot rolling process due to the large reduction. Some oxide inclusions elongated during the first two rolling stages due to the relatively small rolling reduction, while some oxide inclusions fragmented at the last two rolling stages. From Fig. 5, it is clearly shown that the deformation of inclusions at the first two rolling stages in Heat 3 was the lowest. Comparing different stages of hot rolling in Heat 1 and Heat 2, the deformation of inclusions during the first two rolling stages was similar and the deformation of inclusions at the last two stages of hot rolling in Heat 2 was higher than that in Heat 1. The variation in the average chemical composition is seen in Fig. 4 which is related to the deformation behavior. The elongation of oxide inclusions affected the average composition little because the only change in deformation was the shape of oxide inclusions, but the fragmentation of inclusions promoted changes in its average chemical composition due to the inhomogeneity of those inclusions. Comparing the changes of oxide inclusions during hot rolling in different heats it was observed that the content of SiO_2 in oxide inclusions had a little increase at the end of hot rolling, which was consistent with the research results of Wang.⁵⁾ When the content of Al_2O_3 of oxide inclusions in the billet was close to 35%, the hot rolling process had little effect on the average composition of oxide inclusions. When the content of Al_2O_3 of oxide inclusions in the billet was approximately 23% and 8%, the average Al_2O_3 content of oxide inclusions showed a decreased trend in the hot rolling process. Only trace content of MgO was detected in oxide inclusions, and the content of CaO and MnO of oxide inclusions were affected by that of Al_2O_3 and SiO_2 .

Changes in the average diameter of oxide inclusions during hot rolling are shown in Fig. 6. The average diameter of oxide inclusions decreased throughout the whole hot rolling process; the average diameter of oxide inclusions in Heat 1 decreased during the rolling process but showed large fluctuations in comparison with Heat 2 and Heat 3. Figure 7 shows the change in the number density of oxide inclusions during the rolling process. After stage R1, the number density of Heat 1 decreased, while Heat 2 and Heat 3 showed an upward trend. In the subsequent rolling stages, the number density in different heats all showed the trend of first decreasing and then increasing, which was caused by the broken inclusions during hot rolling. Figure 8 shows the transformation of the area fraction of oxide inclusions

Fig. 5. Typical morphologies of some large oxide inclusions at different stage of hot rolling in different heats: (a) Heat 1, (b) Heat 2, (c) Heat 3.

Fig. 6. Variation of average diameter of oxide inclusions during hot rolling in different heats. (Online version in color.)

Fig. 8. Variation of area fraction of oxide inclusions during hot rolling in different heats. (Online version in color.)

Fig. 7. Variation of number density of oxide inclusions during hot rolling in different heats. (Online version in color.)

during hot rolling. Different heats had similar results; the area fraction decreased at first and rose in the later stage of hot rolling. In fact, the deformation and elongation of oxide inclusions during hot rolling cause the increase of average diameter, and the fragmentation of oxide inclusions increases the number density and area fraction, and decreases the average size. As shown in Figs. 6, 7, and 8, oxide inclusions were fragmented due to continuous deformation during hot rolling, especially when the reduction ratio was relatively large in the late rolling pass. There was an increase in the average diameter in the early stage of hot rolling when the content of Al_2O_3 in oxide inclusions was approximately 30%, which meant that there were a large number of oxide inclusions with better deformation ability in Heat 1.

The deformation of inclusions during hot rolling depends on different factors including dimensions, distribution, rolling parameters (temperature, reduction ratio, *etc.*), homogeneity and melting points which depend on the composition of inclusions.^{16,21,26,27,31)} From Fig. 3, it can be seen that the size of most oxide inclusions is less than 10 μm . In this study, it is assumed that the deformation of steel during the rolling process was uniform and the rolling parameters in different heats were similar. Wang^{5,28)} *et al.* proposed that there are dual-phase inclusions ($\text{MnO-SiO}_2\text{-Al}_2\text{O}_3$) in tire cord steel, and these inclusions with different deformability became gradually separated from each other during hot rolling. Therefore, the main factors affecting the evolution of oxide inclusions in different heats during hot rolling were the differences related to homogeneity and melting points. The low melting point inclusions with better deformability were elongated into a thin strip, while the inclusions with poor deformability deformed little or got broken. However, both homogeneous and heterogeneous inclusions could be classified into 4 types according with its deformation behavior during the rolling process: (a) non-deformed inclusions with small sizes (b) deformed homogeneous inclusions (c) fragile homogeneous inclusions (d) composite inclusion, as shown in Fig. 9. If (a) and (c) type inclusions account for a high proportion, the number density and area fraction of inclusions during rolling process increases and the average size correspondingly decreased. When the proportion of (b) type inclusions was higher, in the case of small reduction ratio, the number density and area fraction of inclusions during the rolling process increased little. Type (d) consists of a mixture of plastic and hard sections which upon deformation break and separate. This fragmentation causes an increase in the average concentration of the hardest component during hot rolling.

5. Effect of Al_2O_3 on Evolution of Oxide Inclusions during Hot Rolling

As mentioned earlier, the deformation of inclusions during

hot rolling is affected by homogeneity and melting points, these parameters are mainly determined by the composition of inclusions. It has been shown that oxide inclusions were fragmented due to continuous deformation during hot rolling, especially when the reduction ratio was relatively large in the R3 and R4 pass. Therefore, the deformation of oxide inclusions at R1 and R2 pass in different heats was studied. In addition to this, the results for the same pass in different heats should be studied simultaneously because the reduction ratio for the same pass in different heats was identical. A deformation index is defined by the aspect ratio λ . An increase in this ratio indicates higher deformability. The aspect ratio λ can be defined as the ratio of the major axis to the minor axis of the inclusion,³²⁾ however due to the irregular geometry of the inclusions the minor axis can be quite small and give high values for the aspect ratio. To avoid this, the aspect ratio defined as the ratio of the maximum diameter of the inclusion to the equivalent diameter has been employed.³⁰⁾

$$\lambda = \frac{d_{\max}}{d_{\text{equ}}}$$

$$d_{\text{equ}} = \sqrt{\frac{4A}{\pi}}$$

where A is the area of the inclusion detected by automatic SEM apparatus (μm^2).

The relationship between the deformation of oxide inclusions and the content of Al_2O_3 at R1 pass during hot rolling process is shown in Fig. 10. The content of Al_2O_3 of oxide inclusions fluctuated in the range from 0 to 50%. Maximum deformability was observed when the content of Al_2O_3 in oxide inclusions was in the range of 20%–25%. When the content of Al_2O_3 is below this range, the deformation capacity of oxide inclusions increased from 2.08 to 3.06 with the increase of the content of Al_2O_3 . The deformation capacity of oxide inclusions decreased when the content of Al_2O_3 in oxide inclusions increased from 25% to 50%. Figure 11 shows the relationship between the deformation of oxide

Fig. 9. Schematic of deformation of inclusions during rolling: (a) inclusions with small size (b) deformed homogeneous inclusions (c) fragile homogeneous inclusions (d) composite inclusion.

Fig. 10. Relationship between the deformation of oxide inclusions and the content of Al₂O₃ at R1 pass during hot rolling process.

Fig. 11. Relationship between the deformation of oxide inclusions and the content of Al₂O₃ at R2 pass during hot rolling process.

inclusions and the content of Al₂O₃ at R2 pass during hot rolling process. Similar to Fig. 10, the content of Al₂O₃ in oxide inclusions with a higher deformation is in the range of 20%–25%.

The melting points (liquidus temperatures) of oxide inclusions were calculated by Macro Processing of FactSage with Equilib Module (FcatPS, FToxid database). **Figure 12** shows the relationship between the melting points of oxide inclusions and the content of Al₂O₃ at R1 pass during the hot rolling process. The melting points of oxide inclusions decreased from 1656°C to 1298°C with the content of Al₂O₃ increased from 0 to 25% and increased again when the content of Al₂O₃ in oxide inclusions increased from 25% to 50%. **Figure 13** shows the relationship between the melting points of oxide inclusions and the content of Al₂O₃ at R2 pass during the hot rolling process. Similar to Fig. 12, when the content of Al₂O₃ in oxide inclusions increased from 0 to 50%, the melting points decreased first then increased. Lower melting points were observed when the content of Al₂O₃ in oxide inclusions was in the range of 20%–25%.

A comparison of the average deformation performance of oxide inclusions during the hot rolling process in different heats is shown in **Fig. 14**. The average deformation perfor-

Fig. 12. Relationship between the melting points of oxide inclusions and the content of Al₂O₃ at R1 pass during hot rolling process.

Fig. 13. Relationship between the melting points of oxide inclusions and the content of Al₂O₃ at R2 pass during hot rolling process.

Fig. 14. Variation of average aspect of oxide inclusions during hot rolling in different heats.

mance of oxide inclusions during hot rolling in Heat 3 was the smallest, while the average deformation performance of oxide inclusions during hot rolling in Heat 2 was the largest and Heat 1 was similar to Heat 2 with a larger fluctuation of average deformation performance of oxide inclusions during hot rolling. As mentioned earlier, elongation and

fracture of oxide inclusions co-exist simultaneously during the entire hot rolling process. With the continuous increase of reduction ratio in the hot rolling process, the fracture of oxide inclusions gradually dominated, which meant that the statistical results of the deformation performance of oxide inclusions continued to decrease. The comparison of the average deformation performance of oxide inclusions during hot rolling process in different heats shows that the average deformation performance of oxide inclusions in Heat 2 with an average content of Al_2O_3 approximately 23% was the largest during hot rolling, and the deformation performance deteriorates when the content of Al_2O_3 deviates from this value. The results are consistent with the previous discussion that lower melting points and higher deformation performance was observed when the content of Al_2O_3 in oxide inclusions was in the range of 20%–25%.

6. Conclusions

In the current study, industrial trials were performed to investigate the transformation of oxide inclusions during hot rolling in tire cord steels, deformation of oxide inclusions with different content of Al_2O_3 was discussed. The following conclusions were obtained:

(1) When the concentration of Al_2O_3 in SiO_2 – MnO – Al_2O_3 – CaO inclusions was less than 50%, its plasticity reached a maximum in the range of 20%–25%. In this range the melting point shows minimum values, increasing for lower and higher values in alumina.

(2) Regardless of the content of Al_2O_3 in oxide inclusions, the maximum diameter of most inclusions was less than 5 μm ; when the mass fraction of Al_2O_3 in oxide inclusions was in the range of 0–50%, the number density of oxide inclusions increased with the increase of Al_2O_3 content.

(3) When the average content of Al_2O_3 in oxide inclusions of billets was approximately 35%, the hot rolling process had little effect on the average composition of oxide inclusions; the average content of Al_2O_3 in oxide inclusions showed a decreasing trend in the hot rolling process when the content of Al_2O_3 in billets was approximately 23% and 8%.

(4) The reason for the statistical changes in the average diameter, number density and area fraction were that the elongation and fracture of oxide inclusions occur simultaneously during the entire hot rolling process. With the continu-

ous increase of the reduction ratio in the hot rolling process, the fracture of oxide inclusions gradually dominated.

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