Nucleation and Growth Process of Sticking Particles in Ferritic Stainless Steel

Won JIN, Jeom-Yong CHOI and Yun-Yong LEE

Stainless Steel Research Group, Technical Research Laboratories, Pohang Iron & Steel Co., Ltd., Pohang, Koedong-dong, Pohang-Shi, Kyungbuk, Korea. E-mail: pc543552@posco.co.kr.

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Nucleation and growth process of sticking particle in ferritic stainless steels was investigated using a two-disk type hot rolling simulator. The sticking behavior was strongly dependent on the surface roughness of a high speed steel roll (HSS) and the oxidation resistance of the ferritic stainless steels. A sticking occurrence was more sensitive for the steels containing higher chromium. There was a critical value which was found to be 3μm of scale thickness in the suppression of the sticking phenomenon. It was also illustrated that the initial sticking particles were nucleated at the scratches formed on the roll surface and were served as the sticking growth sites. As rolling proceeded, the sticking particles grew by the process that the previous sticking particles provided the sticking growth sites.

KEY WORDS: ferritic stainless steel; sticking behavior; hot rolling; high speed steel roll.

1. Introduction
The sticking phenomenon occurs frequently during the hot rolling of ferritic stainless steels, causing surface defects on the mill product and scoring on the roll surface. The sticking of the bare metal, exposed by scale breakaway during hot rolling, to the roll surface is affected by both hot rolling conditions and the stainless steel properties. According to the previous studies1–3) on sticking phenomenon under the various hot rolling conditions, the sticking behavior is strongly dependent on hot rolling conditions such as temperature, contact stress and backward slip ratio in the arc of contact. Also, it is known that the sticking is heavily influenced by the roll materials and the high temperature tensile properties and the oxidation resistance of the stainless steels.4)

By refer to the earlier studies, the sticking behavior is attributable to the growth of flake-like particles on the roll surface.5,6) The flake-like particles generated on the steel surface by plastic failure of surface layer are transferred to the roll surface, on which similar particles generated subsequently by repeated friction between the steel and the roll are accumulated as the rolling proceeds, thus causing the particles on the roll surface to grow. Recently, it was also reported that the rolled materials are relatively homogeneously stuck on the entire roll surface away from the usual expectation that the sticking initiates primarily in the hard region such as the coarse carbides of roll surface and that the nucleation of the sticking depends on the high temperature properties of the rolled materials rather than the microstructure of roll.6) However, despite of numerous studies, there still remain many questions of sticking occurrence mechanism in hot rolling of ferritic stainless steels, especially about the nucleation site on the roll surface.

In the present study, the sticking behavior according to the roll surface conditions and the thickness of scale layer of ferritic stainless steels were investigated. In addition, in order to determine the nucleation and growth process of sticking particle, the sticking simulation with the roll having the artificially formed scratches on the surface was employed. Five ferritic stainless steels with various Cr content from 11 to 26% were used as the rolled material and the high speed steel (HSS) roll as the roll material. Sticking simulation were conducted by using a two-disk type hot rolling simulator, which can empirically simulate the actual rolling conditions.

2. Experiments
Figure 1 shows the schematic diagram of the sticking simulator. The simulation was carried out by using a two-disk type hot rolling simulator which consisted of a high frequency induction heater, water cooling and temperature measuring system, equipped with a contact loading system between the roll and the mating material. The mating material was heated in the sticking simulator by a high frequency induction heater with a heating rate of 20°C/sec to the desired temperature. The experimental conditions of sticking simulation are given in Table 1. The chemical compositions of steels used in this study are shown in Table 2. Also, the chemical compositions of the roll material is shown in Table 3. The sticking weight was estimated as the difference between the initial roll weight and that obtained after simulation.

The roll disc surfaces polished and etched by Nital echant after sticking simulation were observed by optical
microscope. The cross section of the roll disc was examined by scanning electron microscope (SEM) and the oxide layer formed on sticking particles was analyzed by energy dispersive spectroscopy (EDS).

3. Results and Discussion

3.1. Effect of the Surface Roughness of Roll and the Thickness of Scale Layer

The sticking simulation was carried out in order to investigate the effect of surface roughness of roll on the sticking behavior of ferritic stainless steels. Figure 2 shows the effect of surface roughness of HSS roll on the changes of sticking weight for five ferritic stainless steels. The rolling of 20 roll revolutions was carried out after holding for 10 sec at 1050°C. The sticking did not occur when the surface roughness of HSS roll was lower than about 0.5 μm. But, it exhibited that a drastical increase followed by gradual increase in sticking weight at the critical roughness of the roll for the most of the steels except Type 409L. In case of Type 409L, the sticking did not occur at the all range of surface roughness. The sticking phenomenon was more severe for the steels containing higher Cr content.

Figure 3 shows the optical micrographs of HSS roll surfaces which was polished and Nital etched after sticking simulation of Type 444 under the rolling conditions described above. It can be seen that a number of scratches formed on the roll surface increased with increasing surface roughness of the HSS roll and the white particles transferred from the stainless steel strip during hot rolling test were always existed on the scratches. The number of sticking particles increased with increasing surface roughness of the HSS roll. The sticking tendency shown in Fig. 2 corresponded well to Kato’s results where sticking occurred immediately if the specific conditions of occurrence are satisfied. Accordingly, it is thought that there is the critical value of surface roughness of HSS roll in order to nucleate the initial sticking particles and the specific conditions of nucleation were satisfied only beyond the critical value. The fact that the sticking particles were always found on scratches indicated that the scratches provided the nucleation site of the sticking particle.

Figure 4 shows the relationship between scale thickness and sticking behavior of five different grade of ferritic stainless steels according to holding time at 1050°C. The scale thickness of lower Cr steels which are Type 409L and 430 increased rapidly with increasing holding time while the one of the higher Cr steels which are Type 445 and 446 increased slightly, respectively. It also shows that the sticking occurs under the conditions of the thin scale formation irrespective of different steel grade. In case of Type 446 having relatively thinner scale layer, sticking occurs in all conditions regardless of holding time, on the contrary, there is no sticking occurrence in Type 409L having thicker scale layer. The previous report indicated that the sticking is heavily influenced by the high temperature tensile properties and the oxidation resistance of the stainless steels. It was also reported that the existence of lubricants in the contact arc is effective to prevent the sticking and the friction at the work-tool interface during hot working is influenced
by both the thickness and the bulk properties of the scale layer. From the result of Fig. 4, it is deduced that the presence of a substantial layer of scale on the rolling material has the role of lubricant and decreases the opportunity for direct contact between the roll and bare metal during hot rolling. On the other hand, there is a critical value which is found to be about 3.0 \( \mu \text{m} \) of scale thickness in the suppression of the sticking phenomenon.

**Figure 5** shows the sticking conditions in relation with the surface roughness of HSS roll and the holding time at 1050°C for each grade of steels. It can be seen that the steels having higher Cr content show a large area of sticking region, namely, lower resistance against sticking. This indicates that the higher Cr steels need a relatively longer holding time to suppress the sticking by forming thicker scale. On the contrary, the lower Cr steels show a better sticking resistance because of lower oxidation resistance. Conclusively, the tendency of sticking occurrence is strongly dependent on the surface roughness of roll and the thickness of scale layer of rolling materials.

### 3.2. Nucleation and Growth Process of Sticking Particle

The nucleation and growth process of sticking particle was investigated by the sticking simulation of the roll having the artificially formed scratches on roll surface in order to verify that the nucleation site of the sticking particle is a scratch on the roll surface.

**Figure 6** shows the shape of scratches artificially formed by a sharp diamond cone on the polished HSS roll surface (Fig. 6(a)) and the photograph of those scratches (Fig. 6(b)). The depth of scratches was 5~6 \( \mu \text{m} \), and the width was 200~250 \( \mu \text{m} \). **Figure 7** shows particle stuck on the artificial scratch after the sticking simulation for Type 446 stainless steel. The rolling of 20 roll revolutions was carried out after holding for 10 sec at 1050°C. The lightly polished and Nital etched roll surface (Fig. 7(a)) shows that sticking...
particle sticks on the artificial scratch and covers a whole shape of scratch, whereas any sticking particle is not found to stick on the polished roll surface. After strong polishing and Nital etching of sticking particle, it is found that the boundary of the artificially formed scratch is clearly shown, and the inside of scratch is fully covered with the sticking particle. This result indicates that the nucleation site of the sticking particle is a scratch on the roll surface, away from the results of previous reports that the sticking initiates primarily in the hard region such as the coarse carbides of roll surface or that the sticking particles are relatively homogeneously stuck on the entire roll surface. However, there are still the questions in what conditions of scratch is needed to be the nucleation site of the sticking particle and in the reason why the sticking particle sticks on the scratch. In order to verify the detail condition of scratch on the roll surface to be the nucleation site of the sticking particle, the additional studies are needed.

The more detail point of view of the sticking particle stuck on the artificial scratch was investigated in order to verify the growth process of sticking particle. Figure 8 shows the surface of sticking particle of Type 446 stainless steel stuck on the artificial scratch (Fig. 8(a)) and the schematic drawing of the sticking particle (Fig. 8(b)). The surface of sticking particle is divided into three regions with different lightness, dark grey, light grey, and white region which comes from the differences in scale thickness. This indicates that each region experienced the different oxidation procedure during hot rolling test. The dark grey region experienced more chance of direct contact to hot rolling material, and so, it had a relatively thicker scale layer during repeated hot rolling procedure. The dark grey region was stuck earlier on the artificial scratch of roll than other bright regions, whereas, the white region is the latest sticking particle compared to the previous sticking particles. This phenomenon is verified more clearly in the observation of cross section of sticking particle.

Figure 9 shows the scanning electron micrograph of the cross section of Type 446 sticking particle stuck on the artificial scratch (Fig. 9(a)), the schematic drawing of sticking particle (Fig. 9(b)) and the EDS (Energy Dispersive Spectroscopy) analysis result of the boundaries of scale layer (Fig. 9(c)). Figure 9(a) shows many slip lines which might be undergone considerable plastic deformation before they stuck on the roll surface. It also shows some boundaries crossing the sticking area in the inner side of the sticking area. These boundaries were analyzed by EDS. The result shows that these boundaries are typical Fe & Cr oxide layers. From the schematic drawing of the sticking area, it
is found that the sticking area consists of four fragments having a different stacking sequence such as ①, ②, ③ and ④, and the sticking fragment ① is the first one stuck on the artificial scratch. These results indicate that the sticking particle consists of many fragments that were formed at different stage of time, not in the same time during hot rolling simulation. The nucleation site of the first sticking fragment is obviously a scratch on the roll surface and the nucleation site of the next sticking fragments is the previous sticking fragments. Namely, the previous sticking fragments provide a source for growth sites of another sticking fragments.

The nucleation and growth process of sticking particle can be deduced from the previous results of Figs. 7, 8 and 9. **Figure 10** is the schematic diagram showing the nucleation and growth process of sticking particle of ferritic stainless steel. The initial sticking fragment is nucleated at the scratch on the roll surface, afterwards, as rolling proceeds, the sticking particle grows by the process that the previous sticking fragment provide the sticking growth site. The sticking phenomenon can be occurred only when the bare metal exposed by scale breakaway during hot rolling meets a scratch on the roll surface. If the specific conditions of occurrence are satisfied, the sticking phenomenon occurs immediately and the particle grows as rolling proceeds.

4. Conclusion

(1) The sticking phenomenon was heavily influenced by the surface roughness of HSS roll and the oxidation resistance of steels.

(2) The sticking phenomenon was more severe for the ferritic stainless steels containing higher Cr content because of its higher oxidation resistance.

(3) The scratch formed on the roll surface provides the nucleation site for the sticking particle.

(4) As rolling proceeded, the sticking particle grows by the process that the previous sticking fragment provides the sticking growth site for another sticking fragment.

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Fig. 9. The photograph showing the cross section of roll. a) Particle stuck on the artificial scratch of roll b) Schematic diagram of sticking particle c) EDS analysis result of scale layer in particle

Fig. 10. Schematic diagram showing the nucleation and growth process of sticking particle in ferritic stainless steel.