APPLICATION NOTE

Failure analysis of corroded steel surface

Root cause assessment using the Axia ChemiSEM

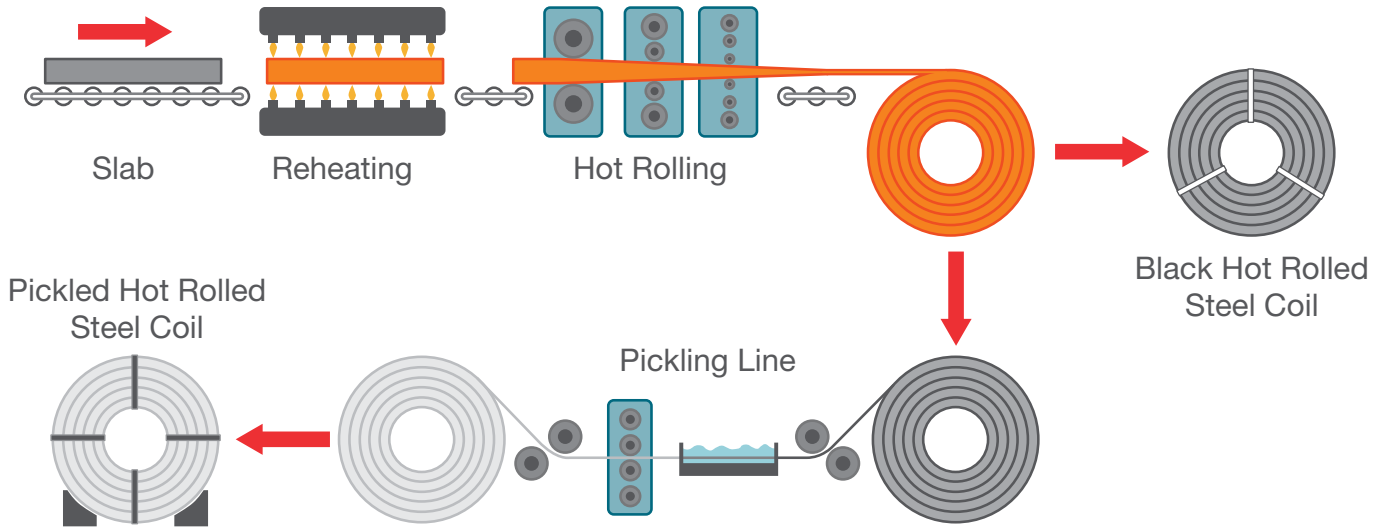

Figure 1. Hot-rolled steel sheet production process.

Introduction

Hot rolling processes (**Figure 1**) produce an oxide layer (also known as scale) on the surface of the steel strip. This scale must be removed before further processing is performed. Shot-blasting may be used to remove scale from discreet parts or for some sheet processing such as stainless steel. An acid bath dissolution process, called pickling, is the most common method for oxide scale removal on carbon steels.

The pickling consists of the immersion of the hot-rolled steel sheet in a highly concentrated, temperature-controlled acid solution in order to remove surface oxides (visible in **Figure 2**, left). Acids commonly used for pickling include hydrochloric, sulfuric, nitric, and hydrofluoric, among others. A combination of acids may be used to optimize the material cleaning while preserving the base metal of a wide range of steel alloys. The etched steel strip is then rinsed in water multiple times to ensure no pickling residues are left on the surface and then dried in warm air.

The sample characterized in this application note is a structural steel that underwent a pickling process and has been left in the etched condition (no oil, phosphate, or primer coating). However, stains have been found after the drying step, requiring further investigations to assess the cause.

Figure 2. Hot-rolled coils or "black bands" in the left image show black oxide scale formed during reheating and red rust which formed at ambient temperature. The right image shows a hot-rolled steel coil after pickling.

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Methods

The root cause assessment was achieved using the Thermo Scientific™ Axia™ ChemiSEM, a newly designed scanning electron microscope (SEM) that provides live quantitative elemental mapping through its always-on energy dispersive X-ray spectroscopy (EDS). This means the operator can view all the needed elemental information at any time without switching techniques.

Discussion

Figure 3 shows a picture of the defective surface. Scanning electron microscopy combined with the chemical information provided by EDS is needed to investigate the defect to assess the presence of morphological abnormalities and to study any possible compositional differences in comparison with the clean surface.

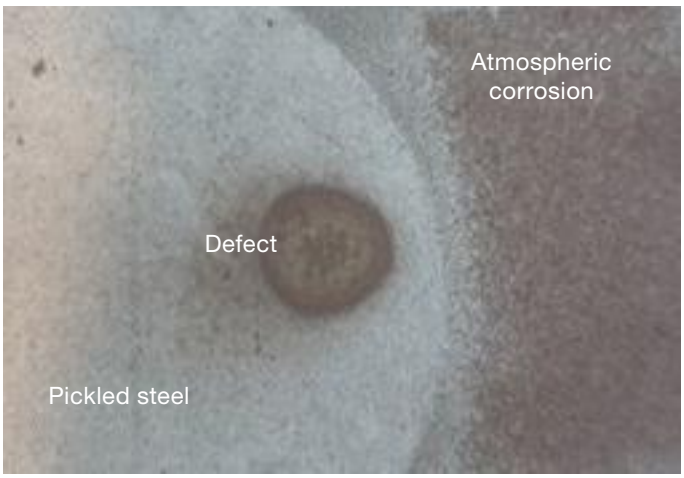

Figure 3. Defective sample.

Figure 4 presents a typical surface of an etched steel. To assess the presence of different morphological features, an image with the same magnification and acquisition parameters has been acquired inside the stain spot and is shown in Figure 5.

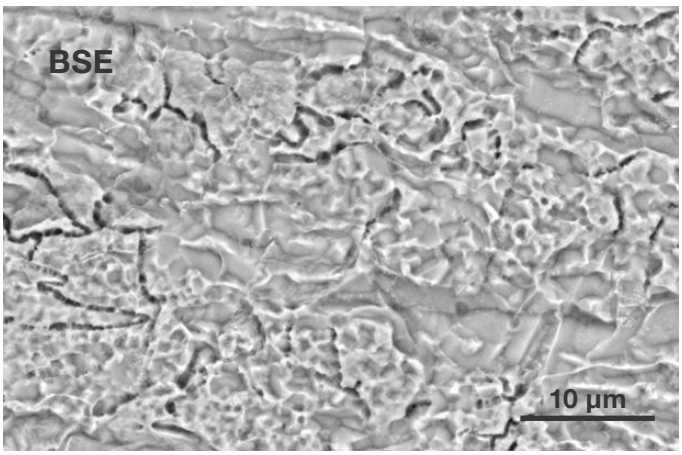

Figure 4. Typical surface of an etched steel (Acc voltage 15 keV, beam current 0.44 nA).

A foreign particle is visible on the analyzed surface. It is also visible in the secondary electron image, where the particle appears to be at a different height than the rest of the surface. However, as we notice from the backscattered electron image, the compositional contrast provided by the conventional SEM imaging is not enough to confirm that it is an unknown object and, most importantly, to disclose its composition.

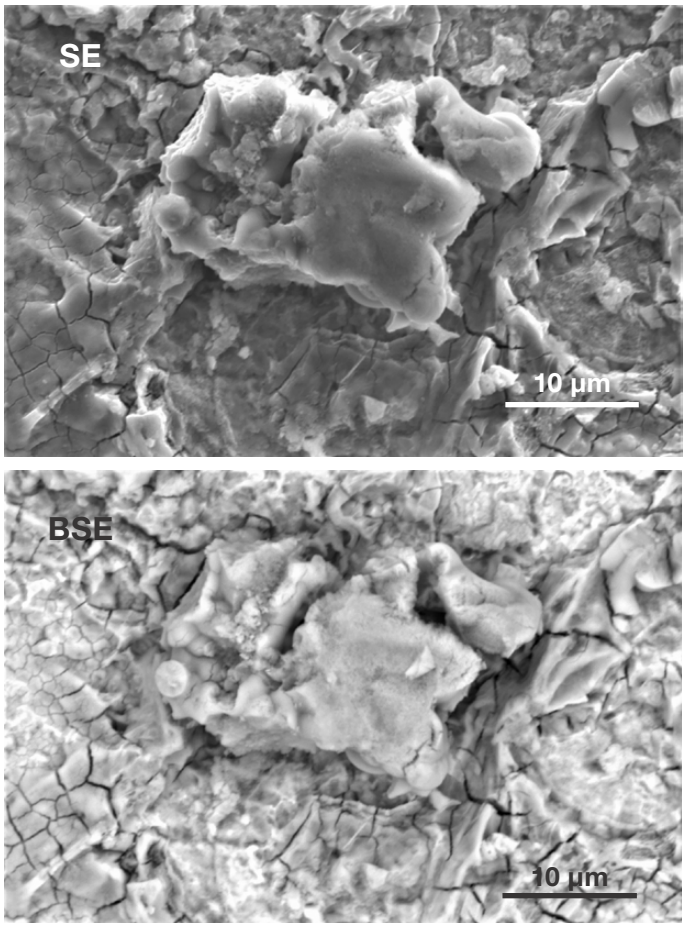

Figure 5. Secondary electron image (top) acquired inside the defective area. It shows the presence of a particle, higher than the surrounding surface, confirming it can be identified as a foreign object. Backscattered electron image (bottom) shows the compositional contrast (Acc voltage 15 keV, beam current 0.44 nA).

Thanks to the new approach provided by the Axia ChemiSEM, the quantitative elemental information of the area of interest is collected during the acquisition of the SEM images and can be displayed, with a single click, on the preferred image (either the secondary electron or the backscattered electron image).

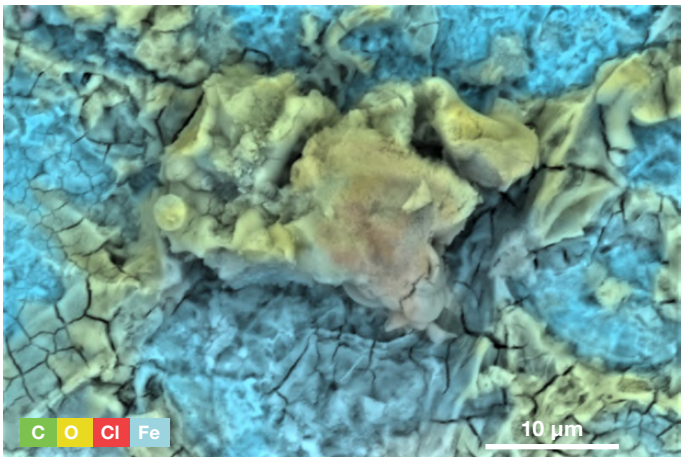

Figure 6. ChemiSEM image of the area displayed in **Figure 5** (Acc voltage 15 keV, beam current 0.44 nA, acquisition time 60 s).

The ChemiSEM image in Figure 6 shows the distribution of all the present elements. The presence of iron is expected, while the presence of oxygen highlights where the sample is oxidized. The presence of chlorine, however, is unexpected and needs further investigation. For an instant confirmation of the chlorine distribution, the Axia ChemiSEM allows you to hide certain elements in order to show only the element of interest (Cl in this case).

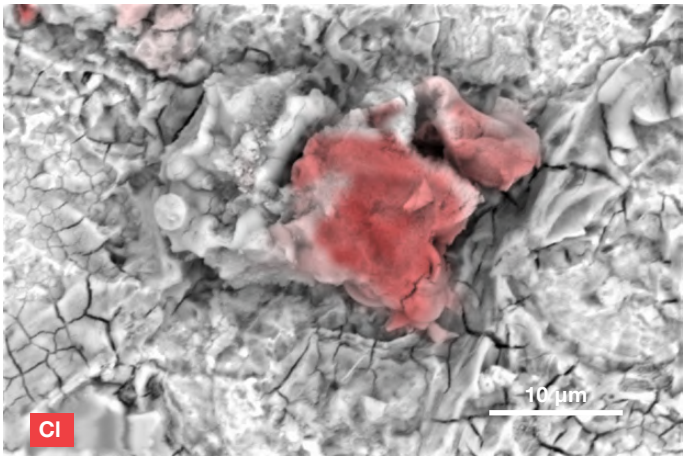

Figure 7. ChemiSEM image of the Cl distribution (Acc voltage 15 keV, beam current 0.44 nA, acquisition time 60 s).

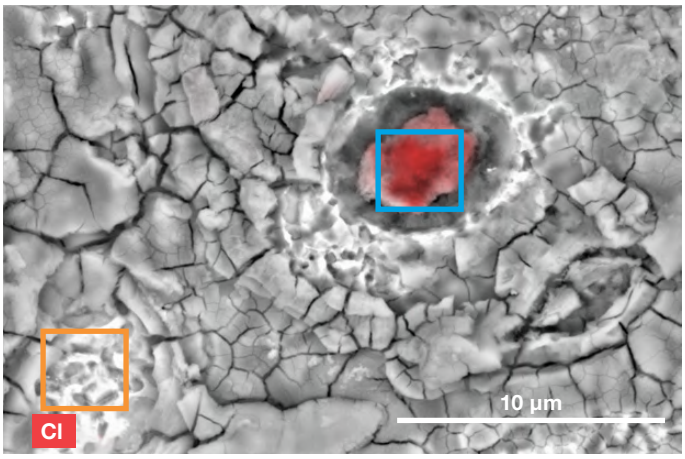

Figure 8. ChemiSEM image showing the two areas used for the region analyses.

Quick area analyses were then executed on another foreign particle and on a clean area of the etched steel (shown in Figure 8) to quantify the contaminants in the stain spot.

Both the spectrum (presented in Figure 9) and the quantifications (Table 1) confirm a clear difference between the two areas: the amount of oxygen and chlorine measured in the particle support the hypothesis of a foreign object.

Element	Foreign Particle Atomic %	Etched Steel Atomic %
O	57.9	9.5
Cl	9.8	–
Fe	32.3	90.5

Table 1. Quantification of the two areas of interest.

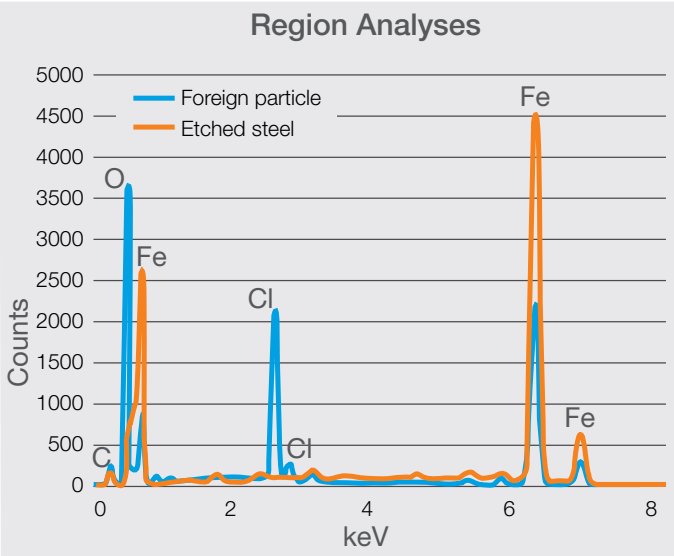

Figure 9. Spectra acquired on the foreign particle and on a clean etched steel surface. The comparison shows the presence of the chlorine peaks (the main peak (Kα) is visible at 2.621 keV) and a higher content of oxygen (Kα at 0.525 keV) in the foreign particle (Acc voltage 15 keV, beam current 0.44 nA, acquisition time 30 s).

The stain spots on the surface contain chlorides in different areas, suggesting that the sample treated with hydrochloric acid may not have been properly washed. Droplets of the pickling acid contacted the surface, causing the stain.

Conclusion

Pickling is a crucial steel surface treatment, especially when the steel is required to be uniform and free of surface stains or oxides. The process requires the use of an acid solution followed by a proper washing step to eliminate acid contamination of the steel surface.

In this application note, a contaminated surface of etched steel has been characterized in order to study the root cause of the contamination. The Axia ChemiSEM was used to investigate the surface morphology and chemical constituents of the defect. In about one minute of analysis time, a chemical map was created, where microscopic surface features were colored based on their composition. Here, the surface stain was quickly found to be rich in chlorides, suggesting that a droplet of acid contacted the surface after the cleaning step had been completed.

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Notes

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