

# Failure analysis on metal automotive production parts

## Coating defect assessment using the Axia ChemiSEM

### Introduction

Sheet steel has been the dominant structural material in the automobile industry since the 1920s. Steel is strong and stiff, making for durable chassis and welded parts. Nowadays, steel is being employed in a continuously growing number of applications, ranging from construction (gears and machinery parts), food (i.e., cans), and wear-resistant tools (cutting tools, springs, high-strength wire). In addition, when compared to other materials, steel has shown itself to be an unbeatable value for mass production applications.

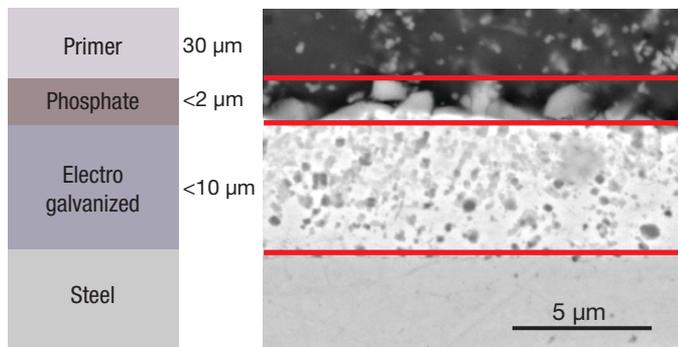


Figure 1. Schematic (left) and SEM image (right) of the different layers.

While the many advantages of steel are clear, there is one main disadvantage: low corrosion resistance. Rust on new or used vehicles has always been a problem. As recently as the 1970s, improvements in base metal and galvanized coating quality were needed to further inhibit rust. The presence of rust not only negatively affects the external appearance, but it also compromises the mechanical structure. Rust may also lead to an early structural failure that compromises the safety of the vehicle. In the 1970s, one partial solution to steel corrosion was simply to make the components thicker. However, in today's world of energy-efficient lightweight materials, heavy components are no longer a viable option.

Because the life of a vehicle depends in large part on the corrosion resistance of its body, the automobile industry worked to improve hot-dip galvanized or electrogalvanized zinc coatings for oxidation resistance. The electrogalvanized zinc layer is applied in a series of electrolytic cells, with the sheet steel being immersed in an alkaline zinc solution. This acts as a cathode,

and when an electric current passes through the solution, zinc ions are reduced to metallic zinc on the steel surface, creating a dense and uniform protective layer bonded to the steel. On top of the zinc metal layer are then deposited a phosphate layer and a primer for the paint, with a structure similar to Figure 1. The zinc phosphate film is applied to ensure further protection of the base metal but also to improve the adhesion of the primer, and, for this reason, it is porous.

The standard electro galvanized coating thickness varies between 5  $\mu\text{m}$  and 8  $\mu\text{m}$ , and its homogeneity is important to provide premium adhesive behavior, both of the upper layers to the coating and of the coating to the metal. The flatness of the sheet steel surface is then, obviously, of high importance to be able to offer a proper substrate for a high-quality coating. If the surface of the rolled steel is altered or imperfect, this can affect the electro-galvanizing and the subsequent adhesion of the zinc phosphate and primer, eventually leading to lower corrosion protection.



Figure 2. Defective coating on car door.

## Materials and methods

The defective coating analyzed in this application note comes from a car chassis component, specifically a car door, where ultra-low-carbon, titanium-stabilized steel has been used. As Figure 2 shows, the coating is peeling off, and this requires understanding what is causing the paint to create flakes and progressively detach from the steel's surface.

In this application note, a fast and easy workflow is presented both to identify the root cause of the automotive coating defect in Figure 2 and to ensure any defects are removed before the final product reaches customers. To achieve these goals, it is important to characterize the structural and compositional details via scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). With the Thermo Scientific™ Axia™ ChemiSEM, and owing to its integrated and always-on EDS, you have instant access to the compositional data necessary for accurate failure analysis, and the qualitative elemental information is directly linked to the SEM image, allowing for significant time savings.

## Analysis

The analysis has been initially conducted on the surface of a defective part of the sample of interest. The red square in Figure 3 shows where the surface characterization has been performed.

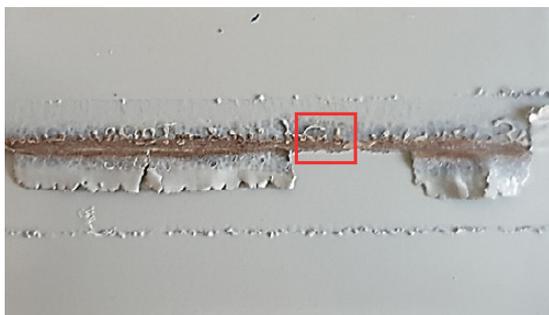


Figure 3. Sample surface.

Figure 4 shows a low-magnification view of the surface of interest, which, as is already visible in the grayscale image, is not protected by any of the coatings. A close-up image has been acquired to further investigate the morphology of the defects but also to acquire elemental information. In fact, one of the main advantages of the Axia ChemiSEM is the direct link between the SEM image and EDS signal acquisition.

During a conventional SEM imaging session, X-rays are collected in the background and can then be shown immediately. The results are available on the spot, with no need to switch to another software application—as with a conventional EDS system—where the operator would need to re-acquire the

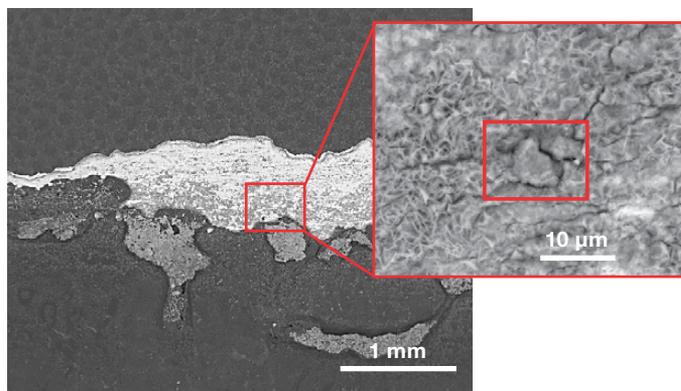


Figure 4. Low-magnification view (left) of the sample surface and a higher magnification of the area of interest (right). (Acc voltage 12 keV, beam current 0.85 nA).

image, start a new acquisition of the EDS data, and, after that, post-process the signal, taking care of the peak overlaps and background removal, among other things.

With a simple click, the qualitative elemental information of the area imaged in Figure 4 is shown, along with the spectrum. Both highlight the presence of a foreign particle. To clarify the nature of the particle, you can decide which element needs to be shown or hidden for a better understanding of the elemental distribution and particle composition. These steps are easily achieved within the same image acquisition, and they provide qualitative information, as the collected signal is post-processed (i.e., the background is subtracted and peak overlaps are considered) prior to being presented.



Figure 5. ChemiSEM image showing the involved elements (all activated and shown in this view) and their distribution. (Acc voltage 15 keV, beam current 0.85 nA, acquisition time 60 s).

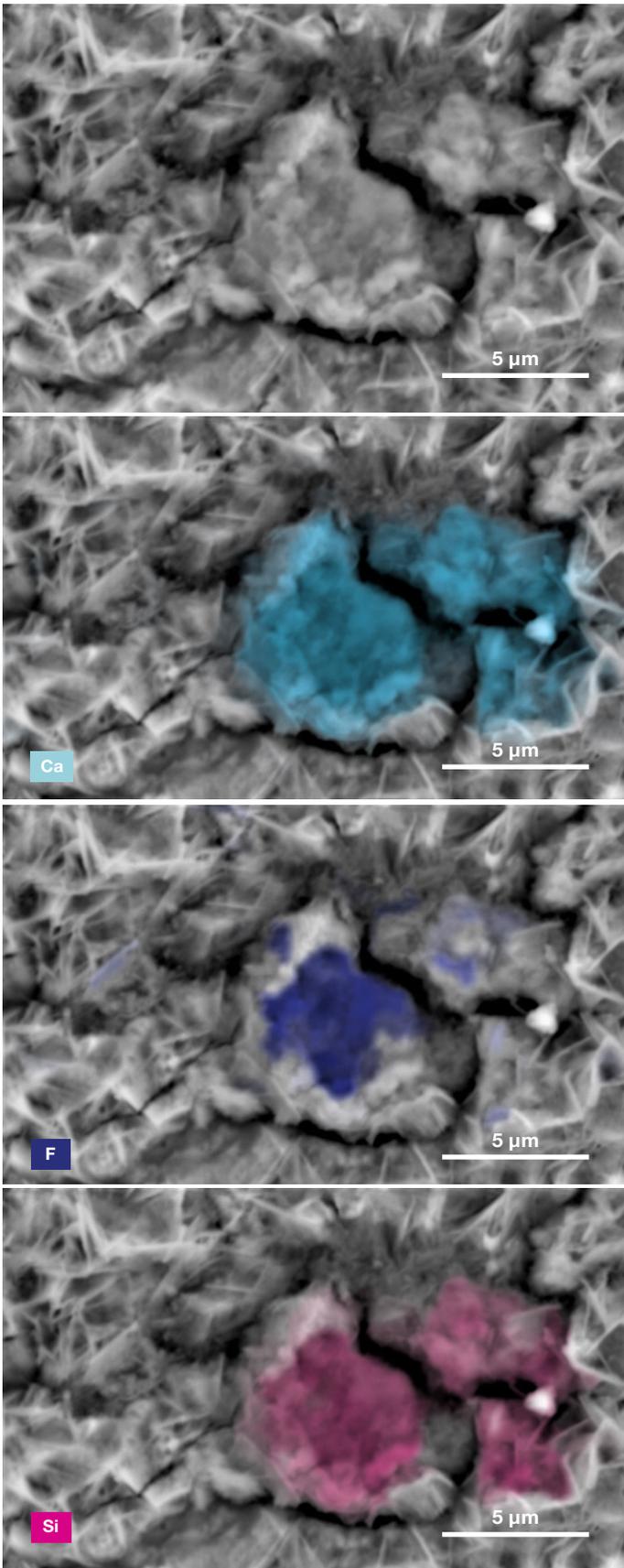


Figure 6. ChemiSEM images of the foreign particle. From top to bottom: backscattered electron image, calcium, fluorine, and silicon distribution. (Acc voltage 15 keV, beam current 0.85 nA, acquisition time 60 s).

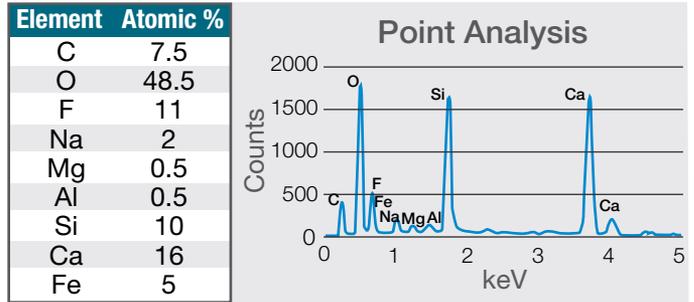


Figure 7. Quantification (left) and spectrum (right) of a 30-second area analysis acquired on the defect (Acc voltage 15 keV, beam current 0.44 nA).

The particle contains calcium, silicon, and fluorine as the most abundant elements. A 30-second area analysis of the defect was acquired, and its spectrum with related quantification is shown in Figure 7.

Both spectrum and quantification of the defect align well with a possible residue of mold powder from the continuous casting process, employed during steel production. The mold fluxes are synthetic slags constituted by a complex mix of oxides, mainly silica ( $\text{SiO}_2$ ) and calcium oxide ( $\text{CaO}$ ). The ( $\text{CaO}/\text{SiO}_2$ ) ratios range between 0.7 to 1.3, with fluorspar ( $\text{CaF}_2$ ) and soda ( $\text{Na}_2\text{O}$ ) added to reduce the viscosity. Carbonaceous materials are also added to their composition.

Steel production makes use of mold powders for different reasons:

1. **Prevent oxidation**—The mold flux constitutes a barrier to avoid steel re-oxidation by contact with air.
2. **Control heat**—Heat transfer in the mold must be controlled.
3. **Lubricate the mold**—A good lubrication is the most important function of the mold fluxes, as a bad lubrication can promote future cracking of the solidifying steel shell.

The presence of surface inhomogeneities prevents the zinc protective coating from adhering to the steel, leading to future corrosion. In addition, exogenous inclusions, such as liquid mold powder droplets, may enter steel by turbulent metal flow, and, for this reason, further characterization of the ROI's cross section has been executed. Figure 8 confirms the presence of sub-surface inclusions.

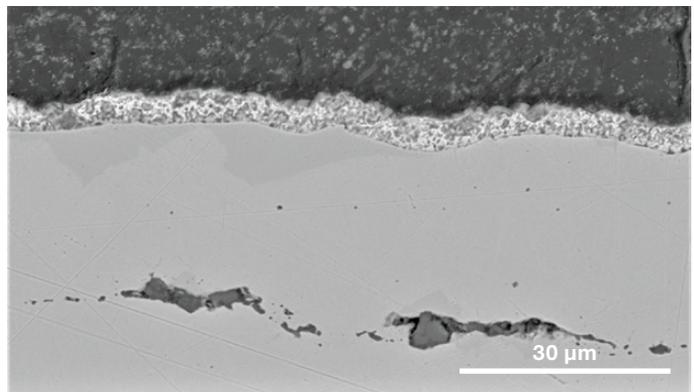
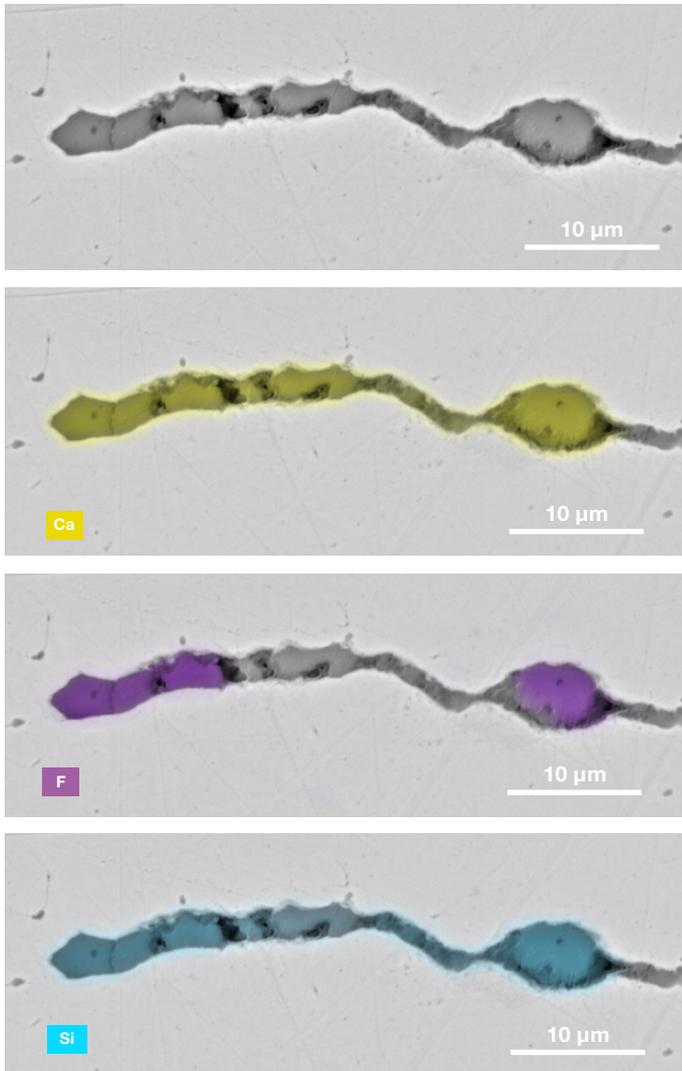


Figure 8. Cross section of the steel showing the coatings involved and the presence of sub-surface inclusions. (Acc voltage 15 keV, beam current 0.44 nA).



The Axia ChemiSEM's characterization of one of the sub-surface inclusions shows (Figure 9), as the foreign particle on the surface, the presence of calcium, silicon, and fluorine as the most abundant elements.

**Conclusion**

High durability and corrosion resistance of automotive exposed steel panels (such as doors, roofs, and quarter panels) are the principal concerns for vehicle manufacturers. All these components must guarantee a high resistance to atmospheric corrosion and maintain their performance over years. For this reason, final products are always inspected if they present defects in any of the protective coating layers, to understand the root cause of the defect and to prevent it in the future.

In this application note, we presented a procedure that allows you to quickly investigate the presence of foreign particles in combination with a fast and easy assessment of their composition. The Axia ChemiSEM, unlike traditional SEMs, provides instant access to all the needed elemental information for accurate failure analysis and defect discovery.

Figure 9. ChemiSEM images of one of the sub-surface inclusions. From top left: backscattered electron image, calcium, fluorine, and silicon maps showing their distribution within the inclusions (Acc voltage 15 keV, beam current 0.44 nA).

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