New Cross-Section Sample Preparation Method Applied to Microstructural and Chemical Investigation of Steel Coatings using FE-SEM

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Abstract: Steel strips coated with Al-43.5Zn-1.5Si (Galvalume) alloy exhibit superior corrosion resistance as compared to Zn galvanized steel strips. The continuous hot-dip coating process used to produce such coatings entails a metallurgical reaction between the steel strip and Al-Zn-Si liquid alloy that leads to formation of an intermetallic compound layer at the steel-coating interface. Formability of the coated strip depends strongly on the morphology, dimensions (thickness) and chemical nature of this intermetallic layer. Proper characterization of the intermetallic layer structure and chemistry and the nucleation sites on the steel surface is therefore of paramount importance for the development of formable Galvalume coated steel strips. This requires preparation of artifact free cross-sectional samples. Such samples can be obtained using JEOL Cross-section Polisher (CP). Unlike mechanical sample preparation techniques that introduce significant amount of strain and possible artifacts due to preferential etching of various constituents, the CP uses a broad Ar beam and a rocking stage that minimize possible preferential etching and produces strain free cross-sections. In this paper, SEM images as well as chemical (EDS) data characterizing the interface layer between the steel strip and the Galvalume coating prepared using Cross-sectional Polisher are presented.

Introduction

SEM observation of a specimen cross section can provide important information for research and development as well as failure analysis. In many cases, surface observation alone cannot compare to the cross sectional image of granular materials, layered materials, fibrous materials, and metallic coatings, etc. Preparing highly-polished cross sections of these materials is both a science and an art.

Typically, a cross section is prepared using mechanical means like conventional mechanical polishing methods or a microtome. The sample is first embedded in a holder or device, and then polished to achieve a flat cross section. In some cases, an etching procedure is used to highlight a specific component of the sample. Such methods can be lengthy procedures that require a great deal of skill, and can introduce artifacts into soft materials, deform the material around voids, or compress layers of soft and hard materials in composite samples. Mechanical polishing can distort fine details such as hairline cracks, and present a challenge to water-soluble phases.

In other cases, a Focused Ion Beam (FIB) System is used when precise positioning of the cross section is required, such as in the case of thin film or micro area specimen preparation. However, the size of the resulting cross section is limited, and the heavy gallium ions in the beam can damage the sample surface.

A new precision Ar ion beam cross section polisher simplifies the preparation of samples and makes it possible to prepare truly representative cross sections of samples free of artifacts and distortion. Use of the broad Ar ion beam eliminates the problems associated with conventional polishing and allows for larger specimens to be prepared with precision.

The JEOL Cross Section Polisher (CP) consists of a specimen chamber with a turbo pump vacuum system, an optical microscope for specimen positioning, and controls for the vacuum system and stationary ion beam (Fig. 1). The specimen stage in the chamber features a holder and masking plate.

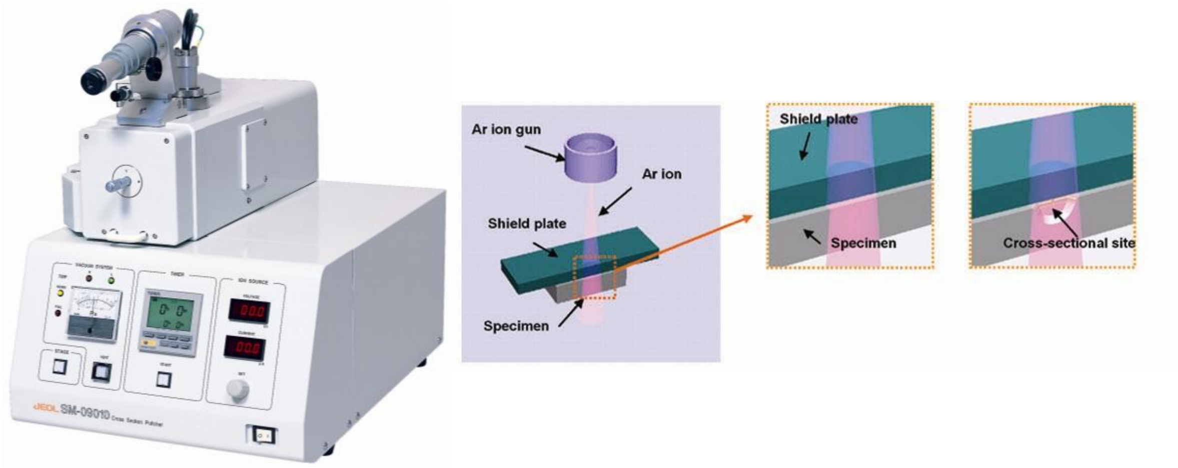

Figure 1 JEOL Cross Section Polisher (CP) and principle of operation

To produce a cross section using the CP, the specimen is placed in the holder, and the region of the sample to be cross sectioned is selected under the optical microscope. The masking plate is placed across the selected region. After evacuating the specimen chamber, the region is irradiated with the broad Ar ion beam with a selectable accelerating voltage range of 2 to 6kV.

During milling, the specimen stage can be automatically rocked +/- 30^o to prevent beam striations and ensure uniform etching of composite materials with different hardness, preventing the soft portions from being cut faster than the hard portions. As it is not a mechanical polishing method, abrasives are never embedded in the polished surface, and samples that are sensitive to heat can be prepared without distortion.

Advantages of the CP over other preparation techniques include:

- High quality cross sections of composites of soft and hard materials
- Minimum strain and distortion of the polished surface, enabling clear and easy observation of grain contrast (channeling contrast)
- Large cross section areas as compared to FIB (a single cut is typically 1.5 mm wide and several hundreds of microns deep)
- No particle embedding in the polished surface as compared to mechanical polishing
- Ease of operation

Cross-sectional observation is critical for manufacturing and determination of adhesion properties of coatings; however, typical mechanical sample preparation often smears the interface layers and makes such observations somewhat ambiguous. In this paper we address application of CP sample preparation method for investigation of Galvalume coatings on low-C steel strips. It has been shown that such coatings exhibit excellent corrosion resistance and therefore it is important to understand what factors govern the formation and the overall properties of such coatings. It has been also shown [3] that increasing amount of Si in the coating bath reduces the thickness of the initial intermetallic layer (~1µm) that forms between the steel and the final coating. Control over the amount of Si in the liquid alloy promotes more adherent coating and prevents excessive growth of the alloy layer. Typically, such samples are prepared via mechanical sample preparation an often are further etched to expose certain regions of the microstructure. Mechanical polishing and subsequent etching introduce artifacts into the structure and therefore make SEM observations somewhat ambiguous. The purpose of this paper is to show how the CP instrument can be utilized for preparation of strain-free and smear-free interfaces for more accurate microstructural and chemical (EDS/EBSD) analyses of the intermetallic compound interface formed during manufacturing of Galvalume coated steel strips.

Experimental Materials and Procedures

Material. The samples chosen for this investigation were strips of cold rolled low C steel (0.06 wt% C, 0.2 wt% Mn) coated with a Galvalume alloy of nominal composition Al- 47.5Zn-1.5Si (wt%). Coatings were produced by continuous hot-dipping in an industrial continuous galvanizing line. Samples had an additional very thin organic or chromate coating.

Sample preparation. Small sections of the samples (1x1 cm^2) were cut out using a diamond saw. The samples were then mounted in the CP instrument and polished using 5kV Ar^+ ion beam for 3-6 hrs. The overview of the resulting cross-section can be seen in Fig. 2. The samples were imaged using JEOL JSM-7000F Schottky field emission SEM in combination with Oxford INCA EDS and HKL EBSD systems.

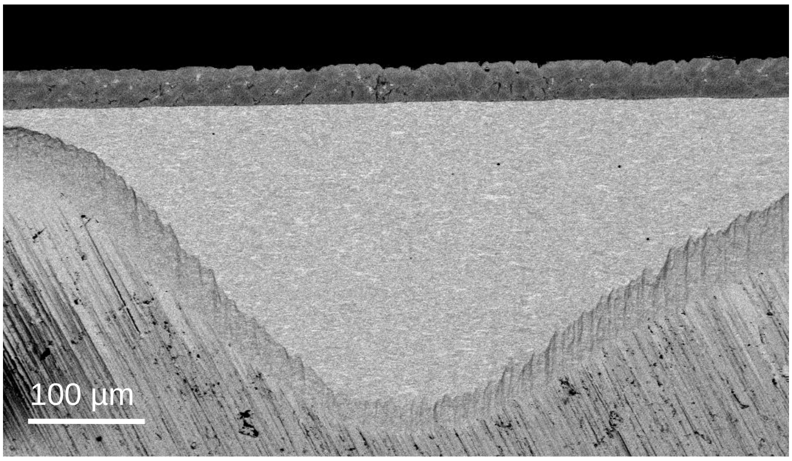

Figure 2 Overview of the cross-section of a Galvalume coated steel strip prepared with the CP Polisher.

Results and Discussion

Fig. 3 shows typical SEM image and corresponding EDS analysis of a sample prepared via mechanical polishing. The various features in the coating as well as the intermetallic region are not very clearly resolved in such a sample, primarily due to inadequate sample preparation.

Fig. 4 shows typical SEM backscatter images of a sample prepared using the CP. The images show very clearly various features in the coating, such as the intermetallic layers and grains, Si-rich needles, Zn/Al/Si eutectic and grain structure of the steel substrate. From the images it can be deduced that the coating grows as a single grain. The intermetallic layer shows presence of Zn-rich particles (white particles in the images). The channeling contrast (grain structure) observed in the steel substrate is indicative of a strain-free sample preparation.

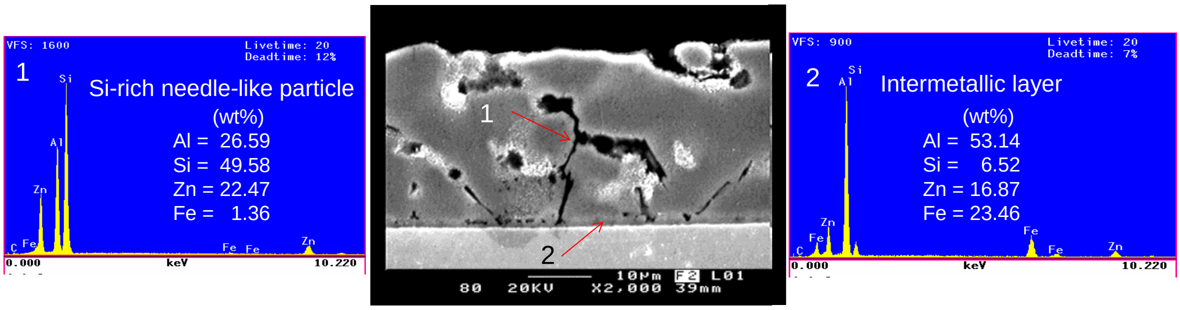

Figure 3 Typical SEM image and corresponding EDS analysis of a Galvalume coated steel strip sample prepared via mechanical polishing.

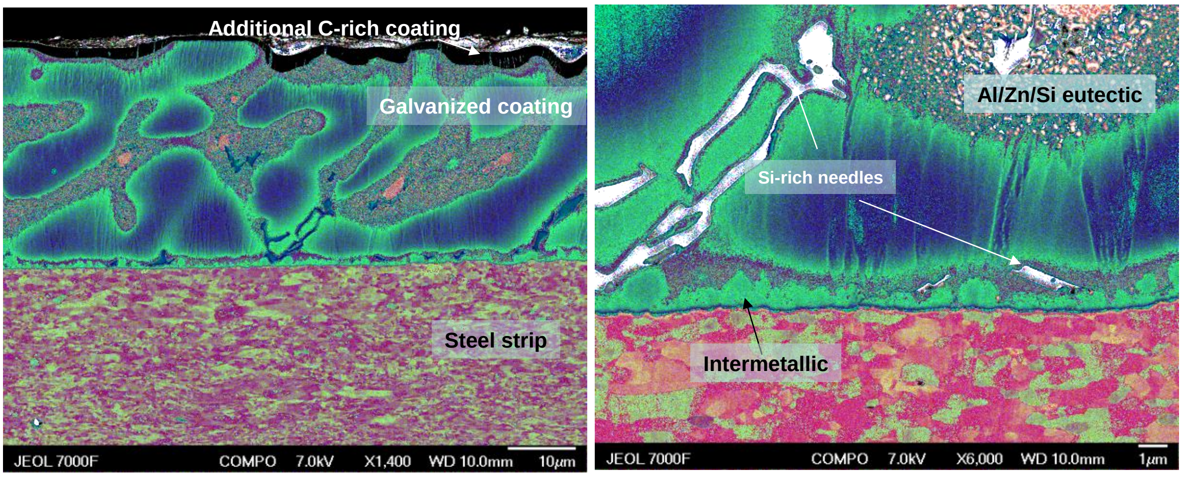

Figure 4 Typical backscattered electro SEM image of a Galvalume coated steel strip sample prepared using the CP.

Closer examination of the intermetallic interface region of the coating (Fig. 5a) allows clear observation of the steel grain structure, delamination between the steel and the coating and the lack of adherence as a result of surface contamination on the steel. Similarly, the eutectic nature of microstructure in the interdendritic region of the coating overlay can clearly be observed in Fig. 5b.

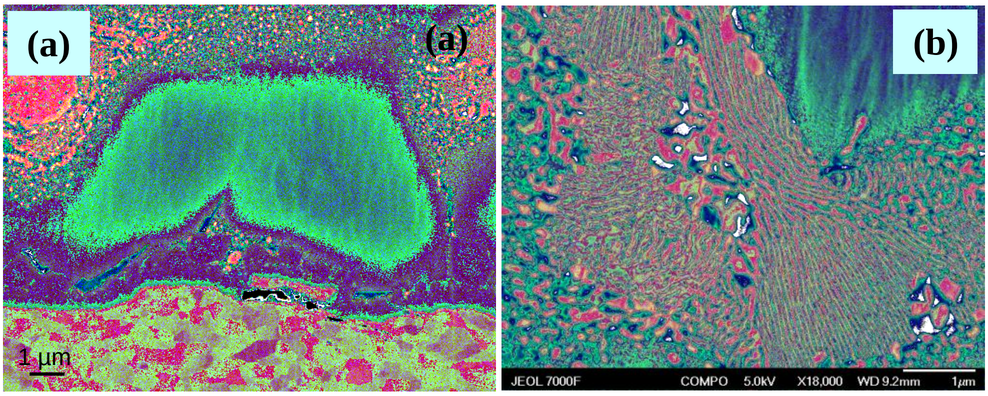

Figure 5 High magnification backscattered electron SEM images of (a) intermetallic interface region and (b) interdendritic region of the coating overlay.

Fig.6 shows a series of EDS maps of a region of the CP prepared sample illustrating the Al, Si, Fe and Zn elemental distributions. From the maps, it appears clear that the intermetallic layer is an AlSiFe intermetallic compound. In addition, these maps clearly show that excess Si in the coating bath results in formation of large needle-like Si-rich particles all over the coating overlay. This type of particles affect negatively the formability of the coating and can lead to cracking and reduced corrosion resistance during forming and service, respectively.

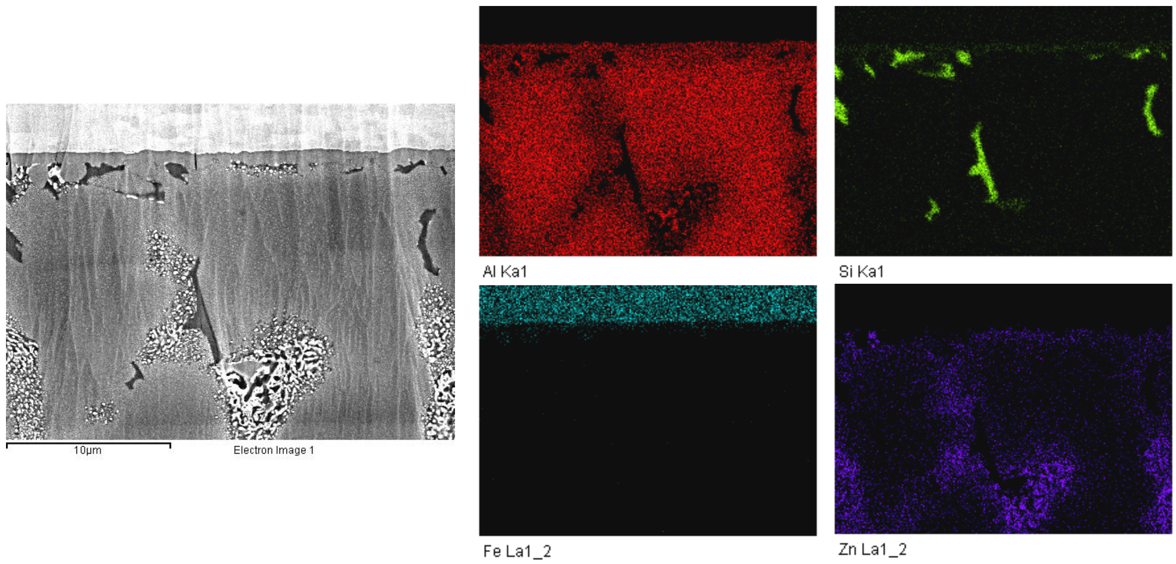

Figure 6 EDS maps of a region of the CP prepared sample illustrating the Al, Si, Fe and Zn elemental distributions in the microstructure of the Galvalume coated steel strip.

It should be noted that the maps were collected at 7 kV, which significantly reduces the electron/specimen interaction volume and therefore allows probing of smaller features in the specimen (Fig. 7). Low kV EDS analysis is difficult to conduct on mechanically polished samples due to some damage introduced to the sample surface during polishing and etching (the EDS results shown for mechanically polished specimens were done at 20kV). The CP preparation is damage and strain-free and it allows very low kV EDS, which in return can provide chemical analysis of very small volumes and allow unambiguous EDS mapping of interfaces.

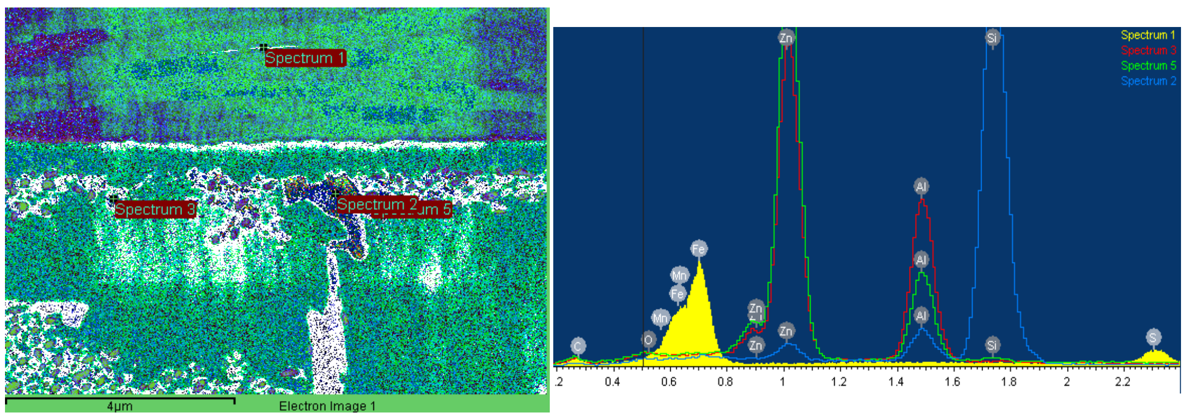

Figure 7 Point and ID EDS analysis of individual features in the intermetallic region of the coating.

Finally, Fig. 8 shows a micrograph of the cross section of a bubble defect formed on the paint coating of Galvalume coated pre-painted steel strip. It is evident that this type of sample could not be prepared by mechanical methods. The bubble formed as a result of deficient curing (short time and/or low temperature) of the primer layer of the paint coating. The curing temperature of the primer is lower than curing temperature of the paint. Thus, gas produced in the primer during curing of the paint layer can migrate to the paint and cause bubbles that break the paint layer and leave surface defects on the pre-painted strip.

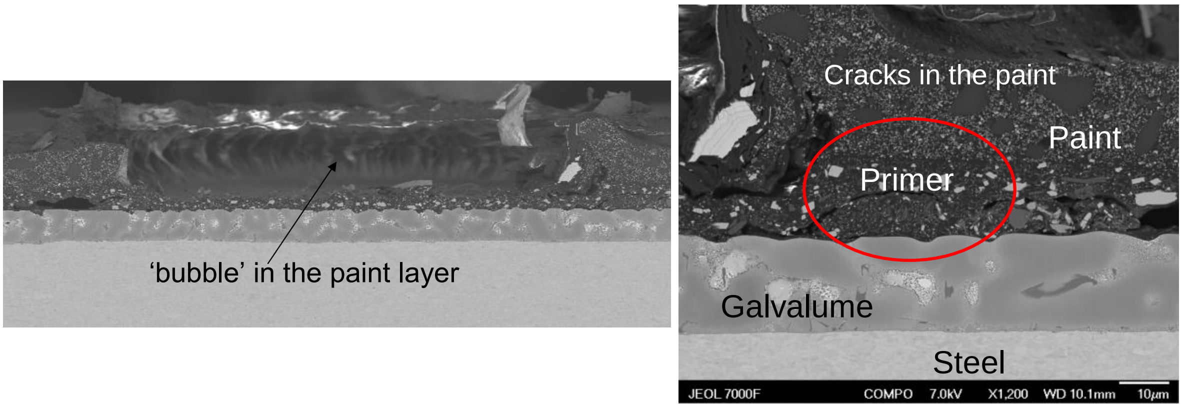

Figure 8 Micrograph of the cross section of a bubble defect formed on the paint coating of Galvalume coated pre-painted steel strip

Summary

Proper characterization of the intermetallic layer structure and chemistry and the nucleation sites on the steel surface is of paramount importance for the development of formable Galvalume coated steel strips. This requires preparation of artifact free cross-sectional samples. Such samples can be obtained using JEOL Cross-section Polisher (CP). Unlike mechanical sample preparation techniques that introduce significant amount of strain and possible artifacts due to preferential etching of various constituents, this technique uses a braod Ar^+ ion beam to polish samples 1 cm wide and several micrometers deep. The technique has been applied in the present work for the characterization of Galvalume coated and pre-painted (Galvalume coated) steel strips.

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