

Model 1060 SEM Mill

Cross-section sample preparation using argon ion milling of a diphase paint coating on a polymer substrate

A method for cross-section sample preparation using the Fischione Instruments Model 1060 SEM Mill of a self-stratifying, intumescent paint coating deposited on polycarbonate substrate.

Epoxy/silicon-based paint coatings on polycarbonate or steel substrates are of interest to many industries. Current areas of research include the development of new types of coatings, such as self-stratifying, intumescent, self-healing and self-stratifying weather-corrosion resistant, for construction, furniture, automotive, and shipbuilding applications [1-6].

The flame retardant properties of intumescent coatings are of interest for construction applications because the coatings can reduce fire risk in combustible building materials, such as wood and plastics.

It is important to understand the structure, substrate adhesion, mechanical properties, and degradation mechanisms of the coatings. Scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS) investigations are key to better understanding the epoxy/silicon-based coatings – from deposition to application.

The ability to prepare a cross-section sample of a substrate with a coating without damaging the coating is a critical to the development of next generation protective paint layers.

The primary difficulty in cross-section preparation of substrates with paint coatings is keeping the coating on the substrate, which enables the characterization of the coating's structure, as well as the interface between the coating and substrate.

Sample preparation by mechanical polishing alone is not suitable for this type of sample because it typically damages or removes the coating from the substrate (see Figure 1). Electrochemical etching is also not a suitable method because of the non-conductive nature of the sample material. Argon ion milling, however, is uniquely suited for cross-section sample preparation of epoxy/silicon-based coatings on a wide variety of substrates.

This application note describes a method for cross-section sample preparation using the Fischione Instruments Model 1060 SEM Mill of a self-stratifying, intumescent, diphase coating deposited on a polycarbonate substrate. The coating and substrate were observed before and after the ion milling process using a SEM microscope equipped with a backscatter electron (BSE) detector. The observation was made at 5 kV acceleration voltages; EDS maps were collected at 10 kV.

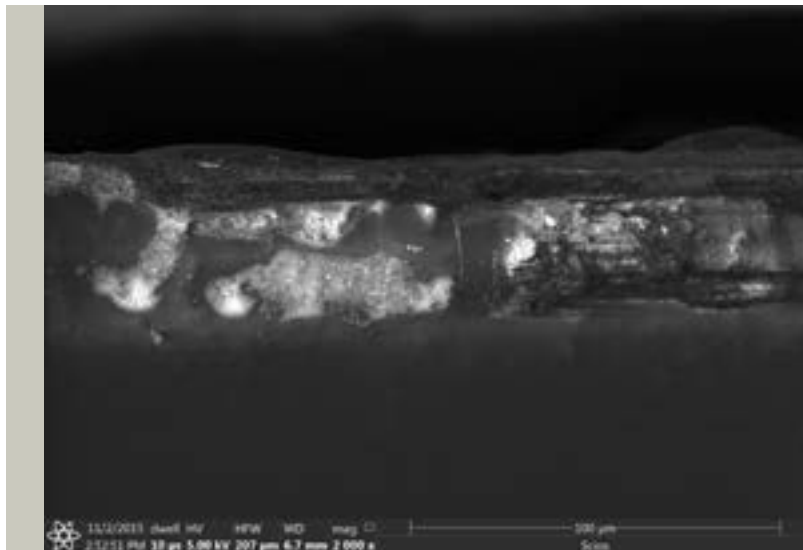


Figure 1. Cross section of a self-stratifying, intumescent, diphase coating deposited on a polycarbonate substrate. Significant damage to the coating due to mechanical polishing is observed.

Cross-section preparation

The sample was polished mechanically using alumina 0.3 μm lapping paper to make the cross section of the sample as flat as possible and to remove artifacts acquired during sample cutting. This step should be accomplished quickly (< 10 minutes) to avoid degradation of the coating. The goal was to obtain a flat surface prior to the ion milling process. Figure 1 shows the cross-section sample following mechanical polishing.

The sample was then polished using the Model 1060 SEM Mill with the milling parameters shown in Table 1.

Table 1: Model 1060 SEM Mill milling parameters.

Parameter	Setting
Number of ion sources	One
Energy	4 kV
Beam angle	3°
Rocking angle	15°
Milling time	3 hours

Results

Following the ion milling process, the sample was imaged using a BSE detector at 5 kV. Figure 2 shows the coating cross section after the ion milling process. The interface between the substrate and the coating was clearly observed (Figure 2a). The observations at higher magnification showed that the ion milling process revealed the structural details of the analyzed layer (Figures 2b-c). From BSE images, one can clearly observe two main phases: epoxy-based (C-rich area, Figure 3b) and silicon-based (Si-rich area, Figure 3d) resins, which were confirmed by EDS analyses (Figure 3b-f). Figure 4 presents the EDS map sum spectrum from the analyzed area. The white particles above the silicon-based resin were identified as rich in iron (Figure 3c). Figure 2c shows an interphase region between coating layer phases.

The improved surface quality following the ion milling process revealed a very thin

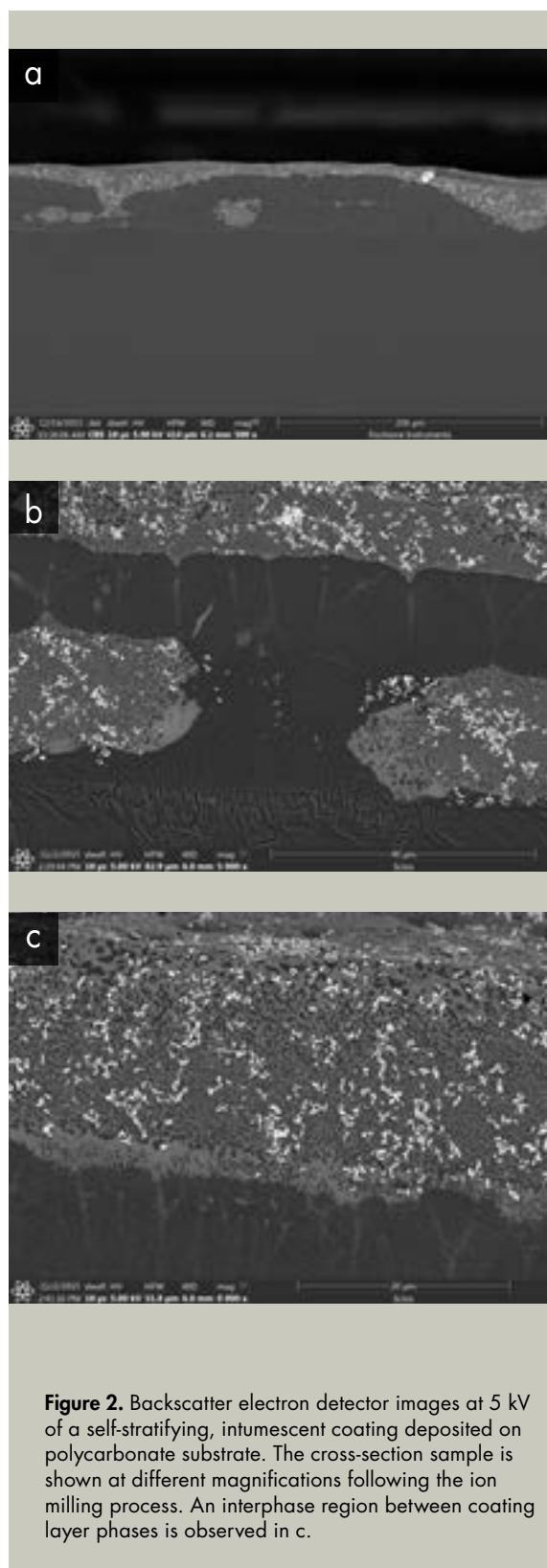


Figure 2. Backscatter electron detector images at 5 kV of a self-stratifying, intumescent coating deposited on polycarbonate substrate. The cross-section sample is shown at different magnifications following the ion milling process. An interphase region between coating layer phases is observed in c.

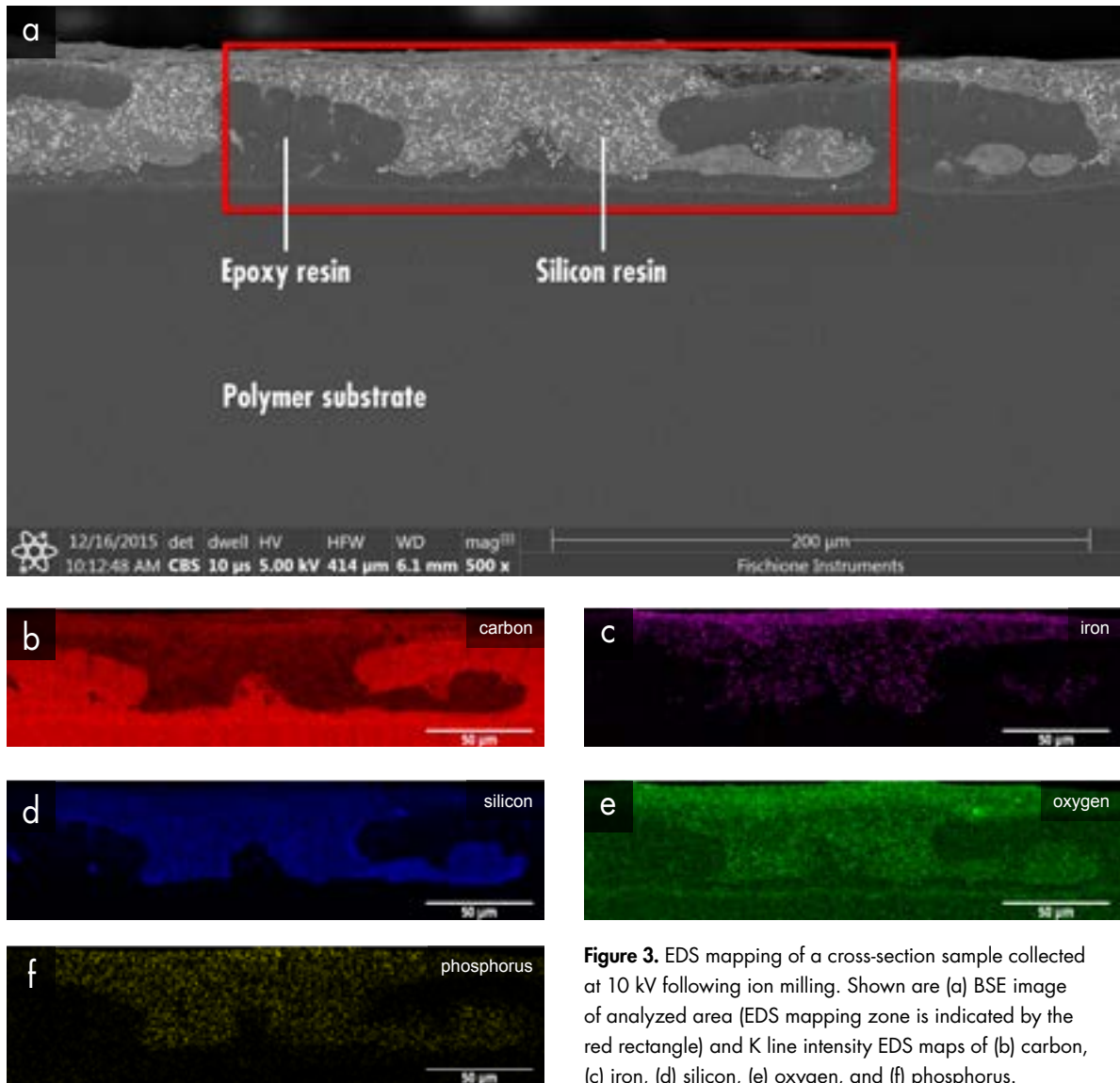


Figure 3. EDS mapping of a cross-section sample collected at 10 kV following ion milling. Shown are (a) BSE image of analyzed area (EDS mapping zone is indicated by the red rectangle) and K line intensity EDS maps of (b) carbon, (c) iron, (d) silicon, (e) oxygen, and (f) phosphorus.

and homogeneously spread structure within the silicon-based resin phase (Figure 2c). This detail was not observable following mechanical polishing. The EDS mapping revealed that the thin structure contained phosphorus (Figure 3f). Phosphorus promotes char formation, which protects the coated surface from burning [1]. The ability to evaluate the homogeneity of the phosphorous distribution

is critical when analyzing self-stratifying, intumescent coatings.

Conclusion

Accurate cross-section preparation of paint coating layers enables imaging and analyses of the coating's structure and properties. Use of the Model 1060 SEM Mill for cross-section sample preparation:

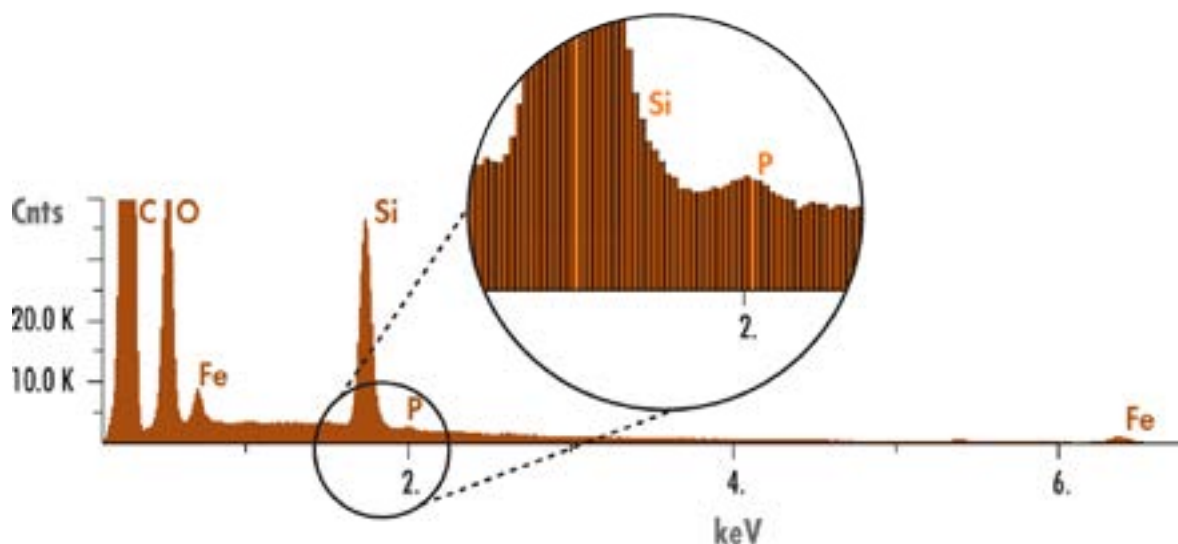


Figure 4. EDS map sum spectrum from the analyzed area (EDS mapping zone is indicated by the red rectangle in Figure 3a).

- enables surface preparation of difficult materials.
- exposes the structure of the coating layer on the polymer substrate.
- reveals the interface between the coating layer and the polymer substrate.
- exposes the interphase structure between the coating layer phases.
- provides the surface quality required for EDS analysis.

Acknowledgements

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References

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