2514
doi:10.1017/S1431927621008965
Microsc. Microanal. 27 (Suppl 1), 2021

Correlating Microscopy Techniques for Understanding Root Cause of Defects in Coatings

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The development of next generation coatings with improved properties is a key goal of PPG, but often new coatings will experience coating defects. In order to move forward with a new coating formulation, fundamental understanding of the defect mechanism is required. The use of microscopy and microanalysis analytical tools can greatly aid in this endeavor. Best practices using microscopy to understand many common defects have been well established (1-3). However, unusual defect types often require more advanced analytical tools or methodologies for complete analysis. This paper will highlight one industrial coating defect and the related microscopy techniques that were used to understand the origin of the defect and how it was correlated to the properties of the film.

Coatings are routinely applied to metal coils by a coil coating process and then fabricated into the final part. These coatings have to be both extremely flexible and durable to survive the fabrication processes, which are often complex. In this work, a new coating was being applied on an aluminum coil and was found to produce a significant number of a fiber defect that would prohibit the use of this coating commercially. It was hypothesized that the fiber itself may be the result of coating damage during the fabrication process, or could be produced from coatings that were either not fully cured or were cured but with incompatible components (both of which could make a coating less durable). A fundamental microscopy analysis revealed that the fibers were formed during the first few steps of the fabrication process, and revealed key information about the size and shape of the fibers, as well as the location where the fibers were being created. These results helped to drive next steps in the formulation of this and related coatings.

Scanning electron microscopy (SEM) was used to identify the location of the fiber and to compare coating damage between different types of coil coatings after the fabrication process. Using a systematic approach, it was determined that the fibers were being generated from the very edge of the metal part. To better understand the shape and morphology of the fiber, X-ray nano-radiography was performed. Fibers sourced from different coating systems were analyzed to generate a series of 2D projections at different angles to gain a multi-perspective view of each. These projections revealed that the fibers were laterally compressed, and that they were not hollow or porous. Additionally, heated stage confocal laser scanning microscopy (CLSM) experiments were performed to understand if the fiber was the result of an undercured film. A series of images were captured while the fiber was heated, monitoring any changes in shape or length. Little change to the fiber was observed upon heating above the coating's Tg, indicating that the coating was cured completely, ruling out this mechanism for creation of the fiber defect.

Finally, atomic force microscopy (AFM) measurements were performed at varying relative humidity levels to look for the presence of polymer domains in the coating that may indicate a decrease in coating integrity. Polymer domain formation could be attributed to a coating incompatibility that may result in a coating more prone to producing fibers. However, no trend in polymer domain formation was observed upon measuring multiple coating types, indicating that this was not the cause of the fiber.

In conclusion, multiple microscopy and microanalysis methods were correlated to determine root cause of a fiber defect. A combination of SEM, X-ray nano-radiography, CLSM and AFM data was used to understand the location, shape, and process for producing the fiber. The results helped to identify the root cause of the defect, determining how and where the fibers were formed while also ruling out key theories related to coating incompatibility. This work helped to inform next steps for coating formulation in order to improve on this product and reduce the formation of the fiber defect.

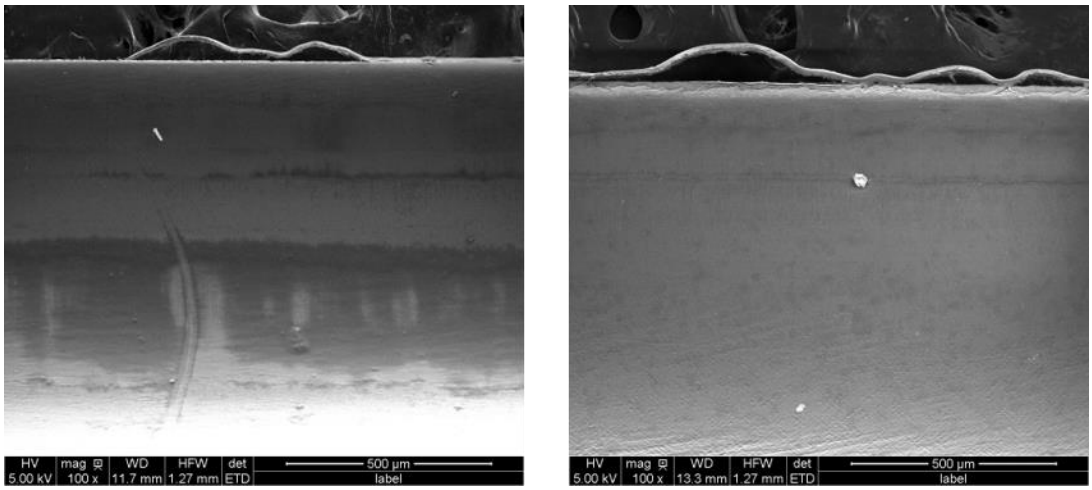

Figure 1. SE micrographs of two different types of fiber defect.

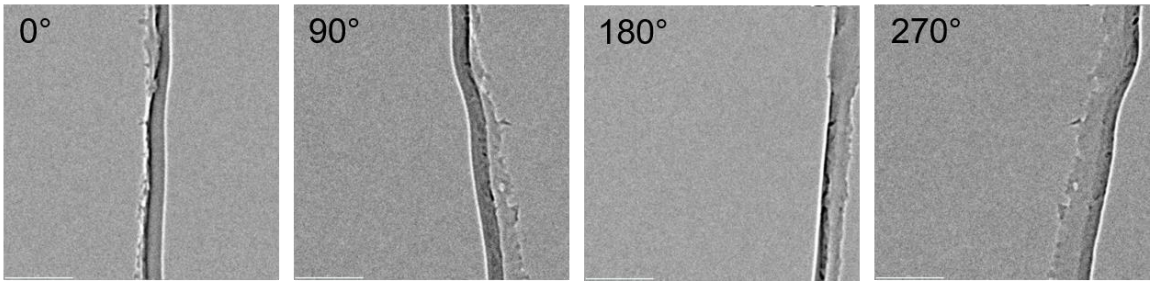

Figure 2. Nano-X-ray 2D radiographs collected at different angles of rotation of an individual fiber. Scale bars are approximately 48 µm.

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https://doi.org/10.1017/S1431927621008965 Published online by Cambridge University Press