A Novel Method of Resin-Embedding Thin, Flexible Polymer Films for TEM Sectioning

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Abstract

The use of a Transmission Electron Microscope (TEM) for cross-sectional analysis of thin polymer film product packaging is essential when determining the uniformity of distribution of components within the film and inspecting for possible defects or damage that could affect the structure and integrity of the film and decrease its effectiveness. Unlike most samples that are prepared for the TEM, the thin polymer films in this study were flexible, resulting in folding, twisting and bending during the resin embedding stage of TEM sample preparation. With no information available in the literature regarding embedding techniques for this type of material, the goal of the authors was to develop a method that would maintain the integrity of the sample, while lending it additional support and rigidity during preparation. The results that follow successfully met the criteria desired in a new sample preparation procedure for thin, flexible polymer films, including: increased film rigidity and support during resin embedding; good adhesion of the resin to the film; and TEM cross-sections that did not contain folds or damage to the edges, as was observed when using previous methods. This new method resulted in improved analysis of these thin barrier films by preventing damage which had previously occurred during embedding and ultramicrotomy using standard procedures.

Introduction

Coated thin polymer films are found in numerous food, drink and medical packaging systems and are used as a barrier to slow migration of outside contaminants into the product, or the product out of the packaging. Structural visualization is critical to elucidating coating-polymer interactions and in examining the product out of the packaging. Therefore, methods to increase the rigidity and support during resin embedding; good adhesion of the resin to the film; and TEM cross-sections that did not contain folds or damage to the edges, as was observed when using previous methods. This new method resulted in improved analysis of these thin barrier films by preventing damage which had previously occurred during embedding and ultramicrotomy using standard procedures.

Materials & Methods

A 2cm x 6cm strip was cut from the sheet of film. The strip was carefully rolled around a wooden pick (film x 2mm). Once tightly rolled, one end of the film was folded over (approximately 1cm from the top) and taped, using standard laboratory labeling tape, to allow the film to retain a rigid tube shape. The opposite end was trimmed to ensure that the film edge was even. An Epon-substitute epoxy resin (EMBed 812, Electron Microscopy Sciences) was prepared and pipetted into a BEEM capsule for each sample. The rolled film was then lowered into the center of the BEEM capsule using forceps. The samples were placed in a vacuum desiccator under 25m of Hg for 30 minutes and then cured in a 60°C oven for 48 hours. Cured samples were then trimmed for sectioning.

The rolled film was clearly visible in the block face (Figure 2A). Depending on the area sectioned, a single block face could contain a single piece (Figure 2B) or multiple pieces of film (Figure 2C). Samples were sectioned using an ultramicrotome at a speed of 1.20 mms⁻¹ and a feed of 100nm, collected on 200 mesh copper TEM grids and imaged with a JEOL JEM 2100 at 200 kV.

Results & Conclusion

Comparison of samples prepared using traditional techniques (Figure 1) to those prepared using the method described above (Figure 3) shows a maintained (or greater) integrity of the film edge and elimination of folding using the proposed techniques. A major concern with prior methods of embedding thin films was the resin’s lack of adhesion to the inner and outer surfaces of the film, partially because there was no support to keep the thin polymer film rigid during the curing process. This resulted in an unstable block and during the process of sectioning on the ultramicrotome, the resin pulled away from the film, causing the edges to roll or tear as the block face was cut.

Using the preparation method above, it was observed that both edges of the film remain flat against the resin (Figures 3B and C), allowing for analysis of the entire cross-section of the film. It is believed that the process of rolling the film allowed for an additional layer of support during the curing process, keeping the film in place with limited movement and improving adhesion between the film and resin. This allowed the visualization of possible film contaminants. Samples from the edges, that would have otherwise been destroyed by the previous embedding methods (Figure 4B and C).

The ability to analyze the entire cross-section of thin and flexible films allows for determination of composition, layering and contamination related to the synthesis and uniformity of these films.

References


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Figure 1: TEM images of film prepared using conventional techniques (dark area is film). (A& B) Show folding that occurs at the edges of the film. (C) Shows damage that occurs due to the flexibility of the film when multiple strips are stacked. Arrows show areas of folding or damage.

Figure 2: Stereomicroscope images of trimmed resin blocks containing the rolled film. (A) An entire film tube in the block face. (B) Sample containing one film strip in the cutting window. (C) Sample containing 3 film strips in the cutting window. Arrows show film in the resin block

Figure 3: TEM images of film after undergoing the described method. (A) Low magnification image of a film cross-section containing none of the damage found in Figure 1. (B&C) Left and right edges of the film remaining attached to the resin with no damage. Arrows show boundary regions between the resin and the film.

Figure 4: TEM cross-sectional analysis of thin, flexible polymer films for possible contaminants within the film. (A) Region of film with possible contaminants observed in the center of the sample. (B) Region of film with possible contaminant along the outer edge of the film (highlighted in red). (C) Higher magnification image of the contaminant along the edge of the film.