Specac Application Note

UK: +44 (0) 1689 892902 US: +1 866 726 1126

WWW.SPECAC.COM
Contact us for more information

Polymer Sample Preparation using the Atlas® Constant Thickness Film Maker Part 2: Separating Strongly Adhering Polymer-Foil Sandwiches Using $NaOH_{(aq)}$

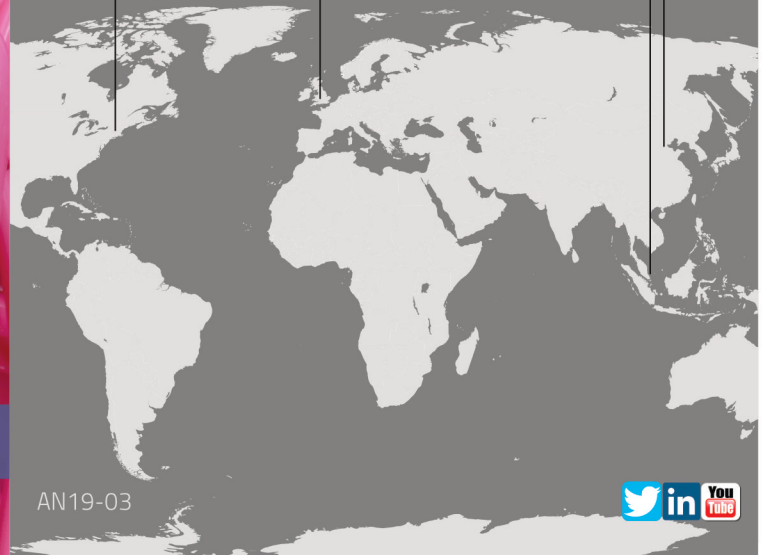
THE QUEEN'S AWARDS FOR ENTERPRISE 2018

London,
England
+44 (0)1689 892902

Beijing,
China
frank.li@specac.com

Fort Washington,
America
+1 866 726 1126

Singapore,
Asia
kamhar.woo@specac.com



AN19-03

Inside: Learn how to release strongly adhering polymers from the Al foil when it will not simply peel away.

Introduction

Polymeric materials are ubiquitous in manufacturing and require analysis to ensure manufacturing conformity. The huge variation in sample form poses a challenge to the analyst!

Hot pressing of polymers to form a thin film is one of the most commonly used techniques for the analysis of polymers by FTIR.

Although most polymers are easily separated from the Al foil used in the molding process, some (such as polyamides) can be a challenge to prepare as a film with issues of thermal degradation and adhesion to the heated surfaces.

Designed for use with the Heated Platens and 15T Manual Hydraulic Press, the Atlas® Constant Thickness Film Maker accessory ensures that a constant and reproducible thickness of film is obtained for reliable transmission FTIR measurements.

- Reproducible constant film thickness
- Quickly and easily change the film thickness with choice of 15, 25, 50, 100, 250 and 500 µm
- Sample turn around time of just15 minutes
- Suitable for Qualitative and Quantitative analysis
- Suitable for polymers with meting points up to 300 °C

Acknowledgement: The data presented here was originally obtained by Dr. A. Afran of Specac Ltd and Dr J.E. Newbury of Greenwich University.

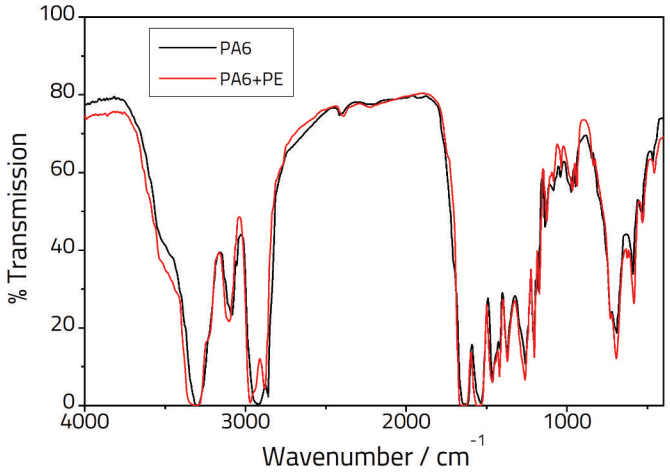


Figure 1: Transmission Spectra of Polyamide PA6 (black line) and PA6 + 20% Polyethylene filler prepared films (red line)

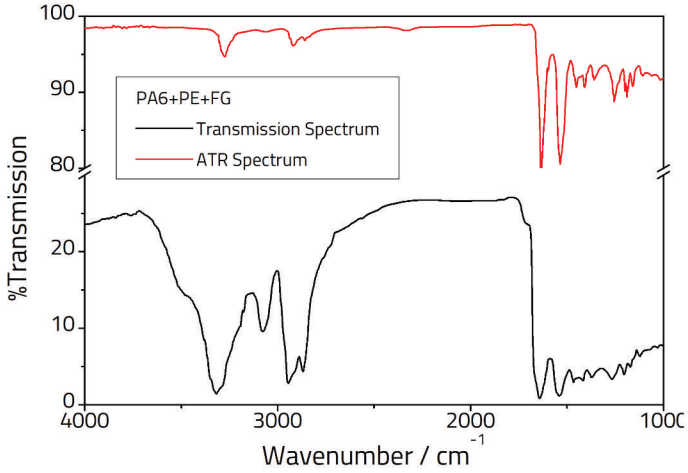


Figure 2: Spectrum of Polyamide PA6 + 5% PE + 30% Fibreglass film analyzed by transmission (black line) and ATR (red line)

A problem commonly associated with preparation of polyamide films is their strong adherence to metal surfaces, making the release of the polymer film very difficult. Commercial releasing agents may be applied to metallic surfaces before pressing to prevent adhesion, though these organic sprays interfere with the polymer film spectral band assignment.

Experimental

Three polyamide samples were selected for film preparation analysis:

- Polyamide (Nylon) 6 (PA6)
- PA6 + 20% polyethylene (PA6+PE)
- PA6 + 5% PE + 30% reinforced fiberglass (PA6+PE+FG)

The samples were prepared by cutting into small pieces and placing in the Constant Thickness Film Maker (CTFM) fitted with a 50 µm spacer ring. The polymer pieces were sandwiched between two aluminium foil discs to avoid the samples adhering to the polished metal surface of the CTFM. The CTFM was placed within an Atlas® 15 T Manual Hydraulic Press fitted with Heated Platens preheated to 150 °C.

To avoid polymer degradation due to overheating, the pressing temperature was carefully increased in 10 °C steps in pressing trials starting from 150 °C to determine the polymer softening temperature. The ideal sample pressing was found to be at 200 °C under 4 tons load for 2-3 minutes. It was not possible to separate the polymer film/aluminium sandwich by normal separation procedures, so the sample was immersed in 3M $NaOH_{(aq)}$ at 40 °C to dissolve the aluminium off the polymer film. The film was removed with a pair of tweezers, washed several times with distilled water and allowed to dry.
Unpressed samples were analyzed using the SelectorTM diffuse reflectance accessory. The CTFM prepared polymer films were mounted in Specacards for transmission measurements and were also analyzed using a Specac ATR unit. All spectra were recorded on a commercially available spectrometer at 4 cm^{-1} resolution.

Results and Discussion

The spectra obtained from these films are illustrated in Figures 1 and 2. To ensure that no chemical effect had taken place during the NaOH dissolution phase, diffuse reflectance spectra of the unprocessed samples were converted into transmission spectra (not shown) and then compared to the transmission spectra of the CTFM prepared films. Both types of spectra were broadly identical indicating no chemical change. However, diffuse reflectance analysis is only semi-quantitative technique and cannot be used for the accurate analysis of fillers and additives. Though the transmission spectra from the CTFM prepared PA6 and PA6+PE films (Fig.1) give excellent band resolution, the fibreglass reinforced film (Fig. 2) exhibits strong scattering below 2000 cm^{-1} masking polymer band resolution. This effect is caused by scattering from the strongly absorbing glass material. Using the alternative ATR technique (Fig.2) overcomes this problem as the analysis pathlength is shorter than in a transmission measurement.

Conclusions

The aluminium dissolution technique has made it possible to extend the capability of the CTFM samples which adhere to metallic materials. By this technique the polyamide films produced may be quantitatively analysed for different levels of fillers and additives by either transmission or ATR methods.

References

[1] AN19-02: Reproducibility of Polymer Film Preparation for Infrared Analysis using the Constant Thickness Film Maker