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Application Note

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Polymer Sample Preparation using the Atlas® Constant Thickness Film Maker Part 1: Reproducibility of the Polymer Films

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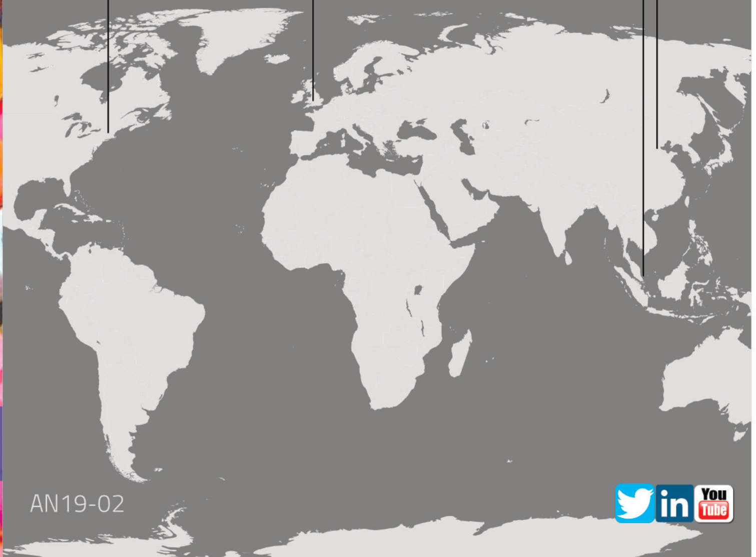

Inside: See how you can form reproducible constant thickness films

Introduction

Polymeric materials are ubiquitous in manufacturing and require analysis to ensure manufacturing conformity. It is therefore imperative that quick and reliable sample preparation and analytical methods are used for quality assurance to ascertain the conformity of these products at every stage of production.

Traditional methods for forming films, such as solution casting, are often long involved processes, with poor sample thickness control and the potential for sample contamination. Hot pressing of polymers to form a thin film overcomes many of these challenges. This application note demonstrates the reproducibility of films produced using the Constant Thickness Film Maker (CTFM).

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 under 15 minutes
- Suitable for Qualitative and Quantitative analysis
- Suitable for polymers with meting points up to 300 °C

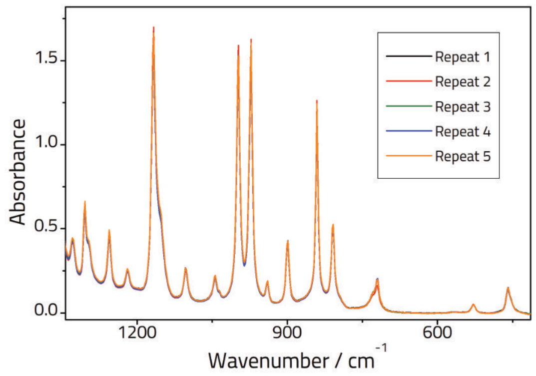

Figure 1: Spectra recorded from different films of PP with a nominal film thickness of 25 µm.

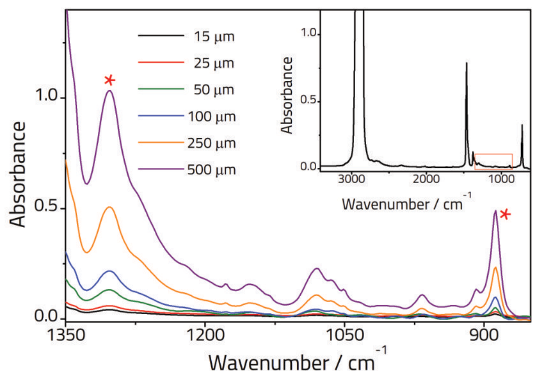

Figure 2: Spectra recorded from films of PE with a variable film thickness. Inset: Full spectrum of the 15 µm film. The red box shows the region displayed in the main panel.

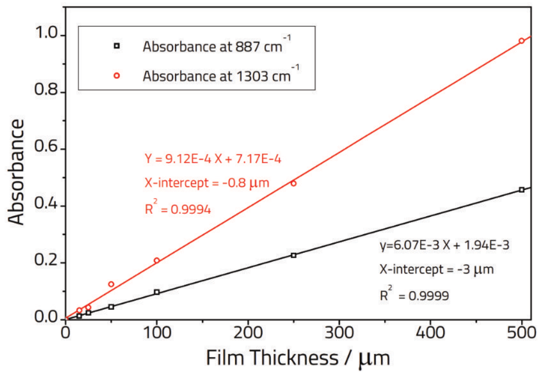

Figure 3: Calibration plots showing the variation of peak intensity (labelled with an asterix in Figure 2) with film thickness.

The CTFM has a series of spacer rings (labelled A to F) which produces film thicknesses of 15 to 500 µm at a set diameter of 29 mm. The unit incorporates a cooling chamber which considerably reduces sample preparation time. Used in conjunction with a hydraulic press and heated platens coupled to an automatic temperature controller, films can be routinely produced in about 8-12 minutes dependent on the type of polymer. This time could be further reduced using two CTFM's; whilst one is cooling the second can be loaded in the press and the polymer melting.

Experimental

Films of polyethylene (PE) and polypropylene (PP) were prepared in the Constant Thickness Film Maker (CTFM) fitted with various sizes of spacer rings. The polymers were sandwiched between two Al foil discs to avoid the samples adhering to the polished metal surface of the CTFM. The thickness of the foil discs supplied by Specac has already been taken into account in the design of the film maker rings and therefore does not introduce any errors into the thickness of the polymer discs produced. The CTFM was placed within an Atlas® 15 T Manual Hydraulic Press fitted with Heated Platens, preheated to 90 °C for PE and 170 °C for PP, and pressed under a load of 5 tons for 5 minutes. The CTFM was transferred to the cooling chamber for 5 minutes and then the polymer film extracted by peeling away the Al foil. Samples were mounted in a Specacard and analyzed in a commercially available spectrometer at a resolution of 4 cm^{-1}.

Results and Discussion

Spectra obtained from five films of PP formed with a thickness of 25 µm are shown in Figure 1. The spectra overlay one another with a very narrow distribution. For each peak maxima shown in the figure (16 peaks), a standard deviation was calculated and expressed as a percentage of the average peak intensity. The average standard deviation was 2.2% with a range of 1.9-2.4%, demonstrating highly reproducible film thicknesses.

PE films of various thickness were also prepared and the spectra obtained from these films are shown in Figure 2. As expected, the peaks increase in intensity with increasing thickness, obeying Lamberts law. The full spectra of the 15 µm film is shown in the inset. Figure 3 shows the calibration plot for peak intensity vs. film thickness for two of the peaks in Figure 2 (labelled with asterisks). A simple linear regression was performed, and a good fit was obtained with R2 values close to 1 and an x-axis intercept very close to 0 µm.

However, it is worth noting that in some circumstances the characteristics of the polymer may result in shrinkage or expansion after film preparation which can result in higher deviation from the expected film thickness. This should cause no problem in the calibration process since such changes will be uniform across the various rings, however, a calibration plot will result in a greater shift from the origin. where absolute values are required for such polymers, it is recommended that the true thickness of the films produced are measured with a micrometer. Some polymers may yield under pressure. Generally, good results can be obtained by maintaining the applied pressure until the polymer ceases to yield prior to transferring the accessory to the cooling chamber.

Conclusions

Highly reproducible films can be prepared quickly and easily using the Constant Thickness Film Maker. An excellent correlation was obtained between film thickness and peak intensity and the film-to-film spectral distribution for a fixed thickness has been demonstrated to be extremely narrow.

The Constant Thickness Film Maker offers the means of obtaining qualitative and quantitative information from polymer films without resorting to the use of internal standards or other more elaborate, time consuming and expensive methods.