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Key Words

Inks

• FT-IR/polymers

Macros\Basic

HATR/Quant

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• Paints & Pigments/

Monitoring the UV Cure Process of a Polymer Based Ink by FT-IR

Introduction

The degree of cure of a polymer-based ink applied to a Mylar film is readily determined by FT-IR. The ink is screened onto the Mylar film and then exposed to UV light to cure the ink. The determination of percent cure is an important quality control (QC) tool and may also be used to optimize the product manufacturing process.

Experimental

Spectra were collected using a Thermo Scientific Nicolet[™] FT-IR spectrometer and a Smart Multi-Bounce horizontal ATR accessory with a zinc selenide crystal. The ATR sampling technique was chosen for this analysis to enhance the spectral response of the inked surface and to minimize the response of the bulk Mylar film base. The inked side of the Mylar film sample was simply pressed onto the surface of the ZnSe crystal of the ATR accessory. No sample preparation was required. Spectra were collected at 4 cm⁻¹ with 32 sample scans (40 second sample collection time).



Figure 1

Results

Figure 1 shows the spectrum of the sample prior to UV irradiation.

For this particular ink, the peak of interest is at 810 cm⁻¹ and is related to free acrylate monomer. As the ink cures, there will be less of this free monomer, so the intensity of this peak will decrease. In order to quantify the degree of polymerization, an internal standard peak is needed. For this ink, the peak at 830 cm⁻¹ is unrelated to the cure chemistry and remains unchanged. It is a simple exercise to monitor the ratio of these two peak heights in order to determine the amount of polymerization that has occurred during the cure process.

Figure 2 shows six spectra from the polymerization process with percent of cure ranging from 0 to 87%. Note that for these six spectra autoscaled on the 830 cm⁻¹ absorbance, the band at 810 cm⁻¹ varies in intensity. Where the peak heights of the 830 cm⁻¹ and 810 cm⁻¹ bands are nearly identical, the ink is uncured. Where the peak height of the 810 cm⁻¹ band is lowest with respect to the 830 cm⁻¹ absorbance the ink is 87% cured.



Figure 2

Figure 3 shows a simple macro, written using Thermo Scientific OMNIC[™] Macros\Basic[™] software, that computes the ratioed peak height of 810 cm⁻¹/830 cm⁻¹ for each

sample spectrum using a base-line correction point at 895 cm⁻¹.



Figure 3



The plot in Figure 4 shows a linear relationship between percent cure and the ratioed peak area. The physical properties of the cured ink are then related to the percent cure to determine the optimal manufacturing process for the UV cured ink based upon the QC determination of the spectral band ratios.

Conclusion

The use of FT-IR with ATR sampling provides a fast and easy determination of the quality and state of UV curable polymerization in inks.

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90 80 70 60 50 % Cure 40 30 20 10 0 1.02 0.90 .0.72 0.66 0.52 0.47 Band Ratio 810 cm⁻¹/830 cm⁻¹ Figure 4

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