

Reaction Tracking of UV-Curable Resin by Rapid Scan

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User Benefits

- ◆ The IRXross FTIR permits acquisition of infrared spectra at a rate of up to 20 spectra/second.
- ◆ Detailed observation of curing reaction processes with reaction completion times of several seconds is possible.
- ◆ Rapid Scan measurement software has various analytical functions such as reaction rate calculation and is an extremely effective technique for tracking chemical reactions and changes.

Introduction

Polymer materials have a molecular structure which is altered by mixing, heating, or irradiation with light. In some cases, those changes are completed in several seconds, while in others they proceed slowly over several hours. Because the Fourier transform infrared spectrophotometer (FTIR) enables high-speed measurement, FTIR is an extremely effective technique for tracking chemical reactions and changes that proceed very quickly. This article introduces the results of measurement of the reaction process of a UV-curable resin using the Rapid Scan function of IRXross, a new FTIR from Shimadzu Corporation.

UV Curing Reaction and Rapid Scan

Acrylate and other UV-curable resins can be hardened (cured) in a comparatively short time by radical polymerization when irradiated with ultraviolet (UV) light. Although the curing reaction proceeds slowly under an ordinary indoor fluorescent light, a nearly complete curing reaction can be achieved in only a few seconds by using strong UV irradiation from a commercial UV lamp. An FTIR with a high-speed scanning function is necessary in order to track reactions of this type, which proceed in a very short time. Because the Rapid Scan feature of the IRXross FTIR permits acquisition of up to 20 infrared spectra per second, even curing reaction processes that are completed in only seconds can be observed in detail. Fig.1 shows the appearance of the IRXross equipped with the Rapid Scan function.



Fig. 1 Appearance of IRXross

Measurement

In this experiment, a thin layer of a commercially-available acrylate-based UV-curable resin was coated on a metal plate, and the infrared spectra generated during the curing reaction process were measured by the specular reflection method.

After a background measurement using the metal plate as the reference, the plate was coated with the resin sample thinly enough to avoid peak saturation.

The measurement was then started in the Rapid Scan measurement mode at an acquisition rate of 20 spectra/second, and UV irradiation was begun after approximately 5 seconds. Fig. 2 shows the scene during UV irradiation of the sample.

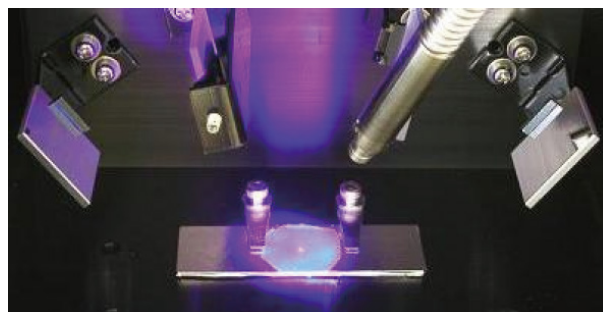


Fig. 2 UV Irradiation of Sample

Table 1 shows the FTIR measurement conditions and the UV irradiation conditions of UV lamp.

Table 1 Measurement Conditions

| | |
|----------------------|------------------------------------------------------------------------------------------------------------------------------|
| Instruments | : IRXross Fourier transform infrared spectrophotometer (KBr window) SRM-8000 special model specular reflectance accessory |
| Resolution | : 16 cm ⁻¹ |
| Accumulation | : 1 time |
| Apodization function | : SqrTriangle |
| Detector | : MCT |
| UV lamp | : HAMAMATSU L9588 Power < 45 mW |

Fig. 3 shows a 3-dimensional (3D) representation of the infrared spectra acquired in the wavenumber range of 1700 to 700 cm⁻¹.

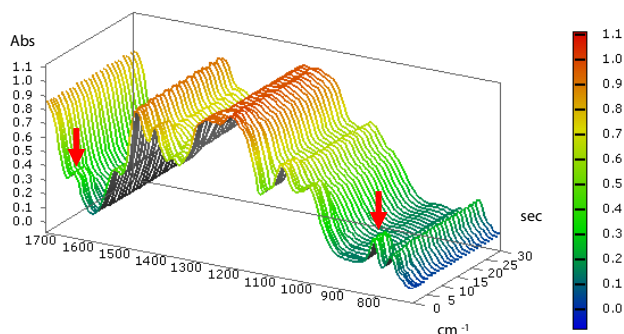


Fig. 3 Results of Rapid Scan Measurement of UV-Curable Resin (3D)

Fig. 4 and Fig. 5 show the expanded spectra of the peaks around 1635 cm⁻¹ and 810 cm⁻¹ in Fig. 3 for a period of approximately 2 seconds immediately after the start of UV irradiation as 2-dimensional (2D) and 3D representations. The peaks at 1635 cm⁻¹ and 810 cm⁻¹ are considered to originate from C=C stretching vibration and out-of-plane C-H bending vibration of the vinyl group, respectively. Here, it can be understood that the peaks associated with the vinyl group decreased abruptly within a short time after the start of UV irradiation.

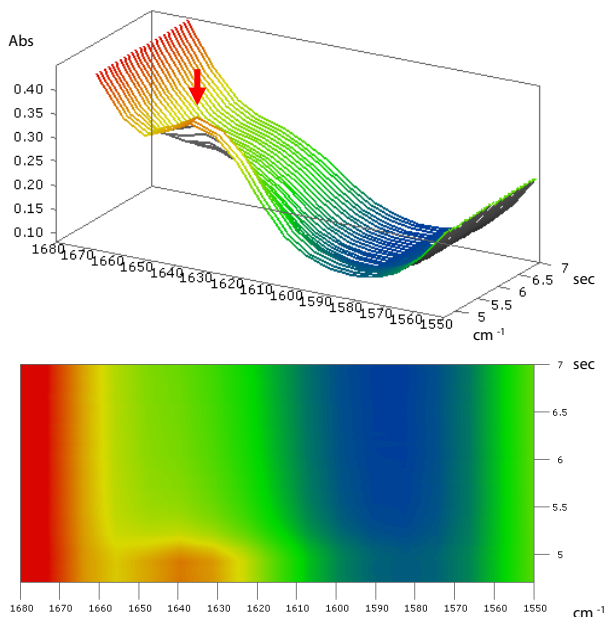


Fig. 4 Expanded Spectra of Peak around 1635 cm⁻¹ in Fig. 3 (Top: 3D, Bottom: 2D)

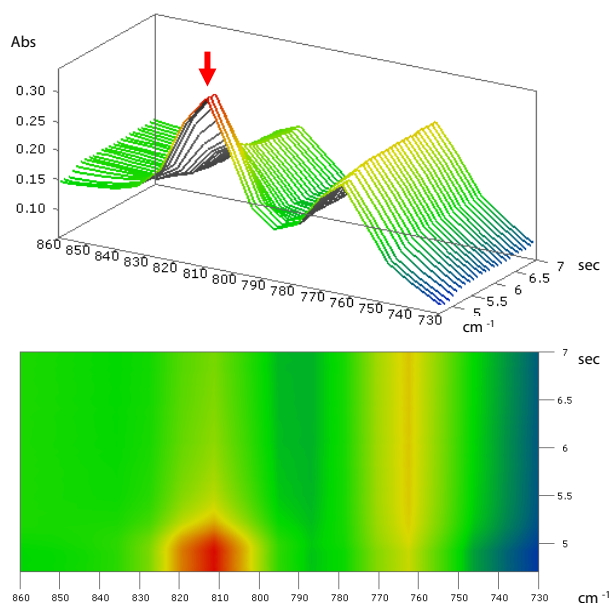


Fig. 5 Expanded Spectra of Peak around 810 cm⁻¹ in Fig. 3 (Top: 3D, Bottom: 2D)

This change is thought to show that the vinyl group decreases by the radical polymerization reaction of the acrylate-based resin (see Fig. 6).

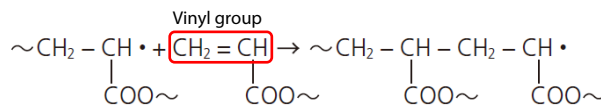


Fig. 6 Radical Polymerization Reaction of Acrylate-Based Resin

■ Time-Course Graph and Reaction Rate Calculation of Radical Polymerization Reaction

Fig. 7 is a time-course graph in which the x-axis shows the reaction time and the y-axis shows the peak areas for 1635 cm⁻¹ and 810 cm⁻¹. Both peaks decreased dramatically within a few seconds immediately after the start of UV irradiation. In this type of time-course graph, the wavenumbers of multiple peaks of interest can be specified before the start of measurement, enabling real-time observation of the changes in the target peaks during the measurement.

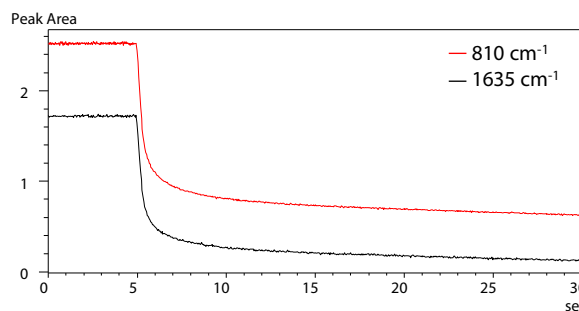


Fig. 7 Time-Course Graph of Peak Areas for 1635 cm⁻¹ and 810 cm⁻¹

Next, Fig. 8 is a time-course graph showing the reaction rate of the radical polymerization reaction using the peak area value of the 1635 cm⁻¹ peak. Here, it is assumed that the reaction rate is 0% before UV irradiation and 100% at the point where the peak disappears, in other words, at completion of the curing reaction. Following the start of UV irradiation (at 5 seconds on the graph), the reaction rate exceeded 50% in less than 1 s and reached 90% in an irradiation time of about 5 s, after which the reaction proceeded slowly. This reaction rate calculation is one example of the analytical functions included in the Rapid Scan measurement software.

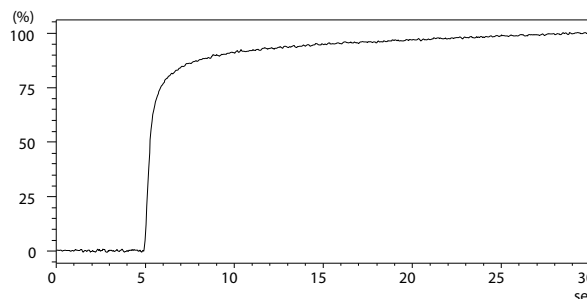


Fig. 8 Time-Course Graph Showing Reaction Rate of Radical Polymerization Reaction

■ Conclusion

The reaction process track of an acrylate-based UV-curable resin was measured using the IRXross FTIR. The Rapid Scan software feature of this new FTIR is an effective means of tracking chemical reactions and changes that proceed very quickly, and enables detailed observation of the reaction process.

It may be noted that the Time-Course measurement program can be used with samples which have a long reaction time. Appropriate use of the Rapid Scan and Time-Course programs according to the reaction time of the target sample is recommended.

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