GladiATR Application Note

(High Performance Diamond ATR)

PIKE社 *“GladiATR ”* 은 다이아몬드 Crystal를 사용하여 시료에 가하는 압력을 30,000 psi 까지 올려도 흠집이나 파손의 염려가 없으므로 특히 금속, Polymer등 단단한 재질의 시료를 측정하는데 탁월한 성능을 발휘합니다. 또한 250℃까지 온도조절이 가능하고 LCD 모니터로 측정 부위를 확인하며 측정 할 수 있습니다.

GladiATR

GladiATR Vision

- 모든 종류의 시료 측정 (고체,액체,분말)
- 높은 광학배율 (110x)
- Cooling & Heating (250℃)
- 고효율 광학 에너지
- Mid-IR부터 Far-IR까지 측정
- 높은 압력(~30,000 psi)에서 사용
- Viewing LCD size : 80 x 60mm
- USP로 PC 연결
- Ge, Silicone crystal 공급

➢Monitoring Cure Characteristics of a Thermoset Expoxy

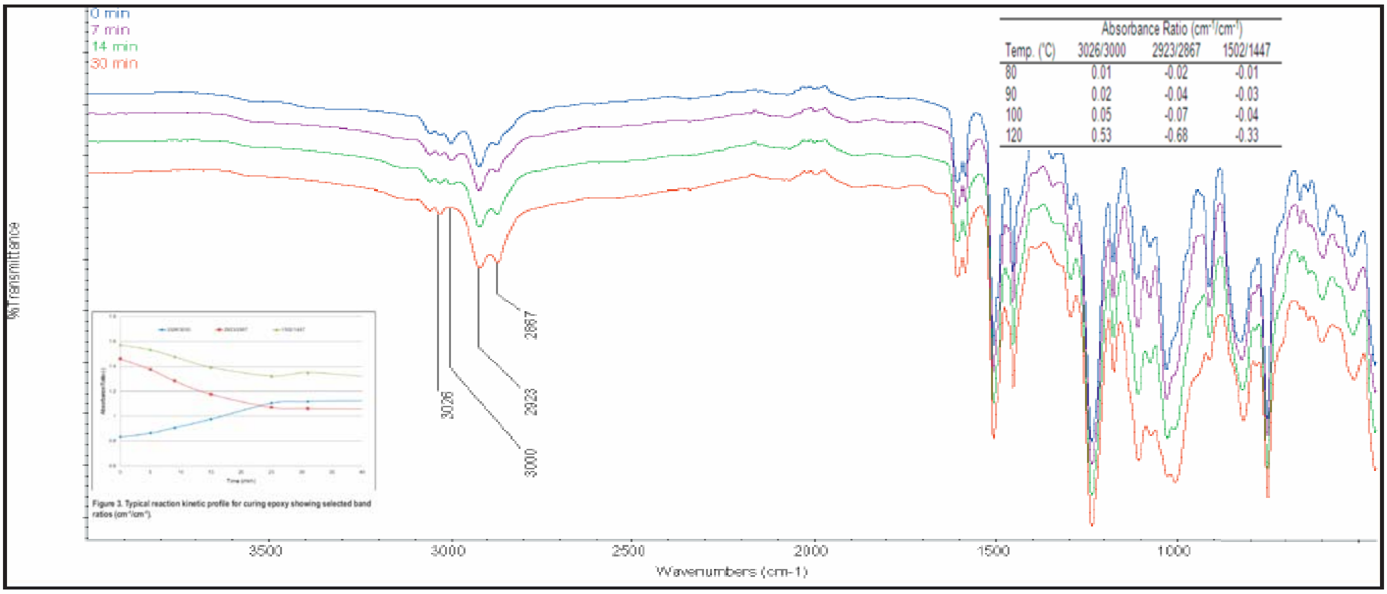

Figure 2. Typical spectral evolution during curing.

Monitoring Cure Characteristics of a Thermoset Epoxy by ATR/FTIR

Introduction

Attenuated total reflection (ATR) has become a primary FTIR sampling technique because of its versatility and ease of use, often entailing no sample preparation. Some materials and applications, however, require an especially robust ATR accessory. Examples include

- Rigid polymers and plastics
- Coated metals and wires
- High temperature thermoset resins
- High temperature surface reactions

Figure 1. GladiATR with heated crystal plate and temperature controller.

Experimental Methods

The PIKE Technologies GladiATR (Figure 1) equipped with a resistively heated plate was used in this investigation. Operational temperature of the heated plate is from ambient to 210 ˚C. Its monolithic diamond, a scratch and fracture resistant crystal, provides a platform to withstand applied pressures of greater than 30,000 psi. This attribute is imperative to assure intimate crystal-sample contact of mechanically rigid samples, achieving the highest quality spectra. In addition, the GladiATR utilizes optimized reflective optics for maximum energy throughput, which maximizes signal to noise ratio. This, in turn, allows for higher precision for low absorbing or low concentration components within a sample.

All spectra were collected at 8 cm^{-1} spectral resolution co-averaging 5 scans. The 2-part thermoset epoxy investigated is suitable for applications such as bonding metal, glasses, and ceramics. Epoxy components A and B were mixed per the manufacturer's specifications at room temperature, and applied directly onto the GladiATR crystal equilibrated at a prescribed temperature (80, 90, 100, 120 ˚C). Curing characteristics were monitored via FTIR spectra as a function of time and temperature. Once the reaction came to completion, the residual was aggressively scraped off using a scalpel without harm to the monolithic diamond due to the diamond crystal's extreme durability.

Results and Discussion

ATR is an optimal FTIR accessory for evaluating cure characteristics of adhesives. These data are useful for formulation development as well as for quality control. Comparing spectra as a function of time may be used to determine reaction kinetics by evaluating absorbance band depletion and evolution as the reaction proceeds. This method also may be used to evaluate cure time by determining the time required to reach steady-state conditions.

Figure 2 shows spectra collected using the GladiATR accessory. By plotting the ratio of selected absorbance bands as a function of time, reaction kinetics may be evaluated (Figure 3). At each temperature step, a brief lag period was observed followed by a decrease or increase in the value of the band ratio and eventually achieving steady-state conditions. Assuming a linear fit during the reaction phase, a kinetic constant was determined (Table 1). These data exhibited an exponential increase or decay with increasing temperatures.

Summary

A high temperature epoxy curing study is presented as an example application where a high performance, robust ATR accessory is desirable. Through FTIR data, reaction kinetics were determined. PIKE Technologies' GladiATR provides exceptional quality data for demanding and high temperature applications.

Table 1. Kinetic constant (min^{-1}) for curing epoxy.

	Absorbance Ratio (cm^{-1}/cm^{-1})		
Temp. (°C)	3026/3000	2923/2867	1502/1447
80	0.01	-0.02	-0.01
90	0.02	-0.04	-0.03
100	0.05	-0.07	-0.04
120	0.53	-0.68	-0.33

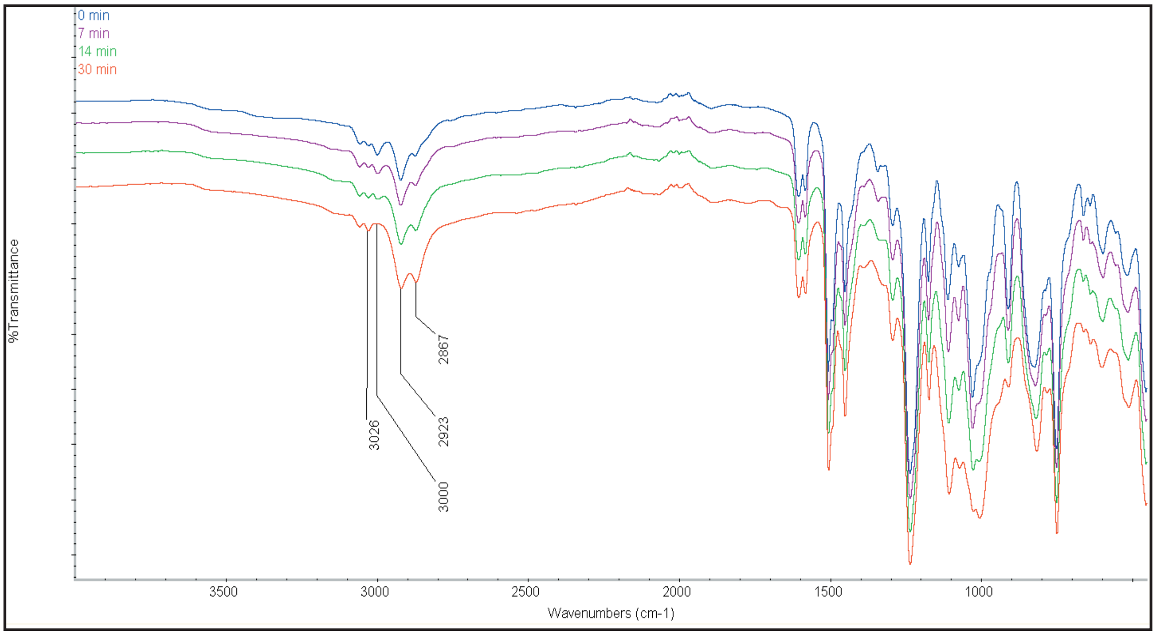

Figure 2. Typical spectral evolution during curing.

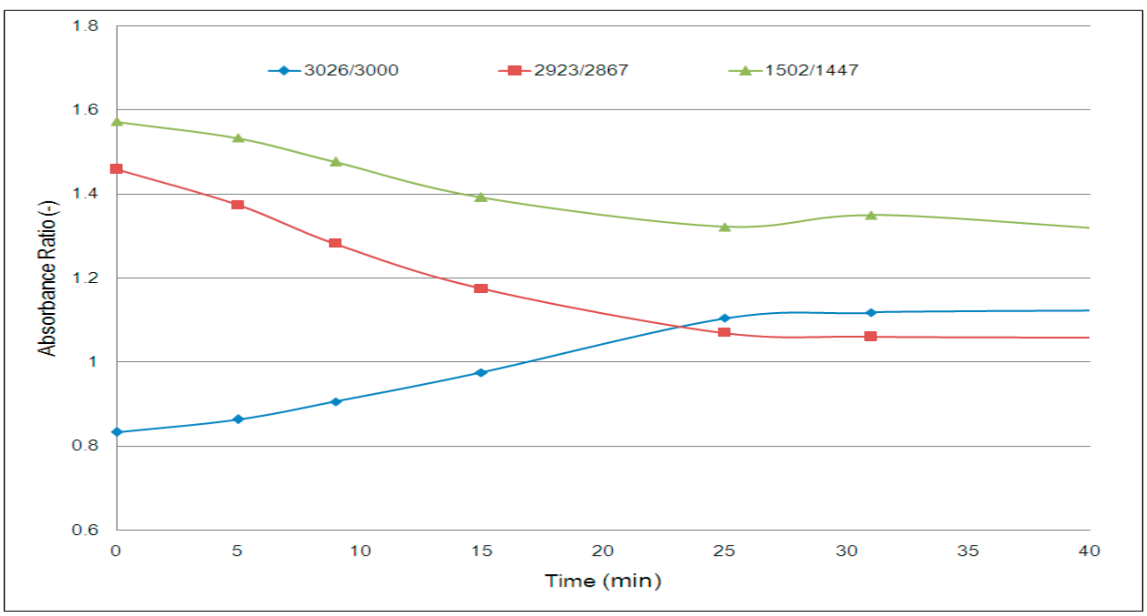

Figure 3. Typical reaction kinetic profile for curing epoxy showing selected band ratios (cm^{-1}/cm^{-1}).