

Reflections on 2005 by Scott Little, President

Welcome to the Winter 2006 edition of the PIKE Reflections newsletter. It's hard to believe 2005 has already passed. At the risk of sounding like a "broken record" (old phrase from the 70's when CD's were called records), 2005 was another incredible year for PIKE Technologies. In fact, thanks to you, we have broken every record we have (not meaning of the musical variety this time). Not only did we set new records for order and shipment levels, but we also beat the old records by the largest margin ever. It's with great humility that all of us at PIKE say thank you for making this all possible and for trusting us with your accessory needs. From the looks of it, we think 2006 will be even better!

As you can imagine, keeping up with this level of growth results in a lot of changes, such as adding to our staff, adding to our inventory and updating our facilities. But one thing that will never change at PIKE is our pro-customer philosophy and our will-

PITTCON 2006 - What's New from PIKE? by Ken Kempfert

This year's PITTCON is already turning out to be a busy time for PIKE. We have plans to launch 8 new products and product enhancements at this year's event in Orlando. We hope that you will be there to visit our booth, discuss your applications and see our new products!

So, what new products will you see in the PIKE booth at PITTCON 2006?

We will be showing 2 new options for our μ MAX IR Microscope - the μ Find X,Y sample stage and the EZSet transmission

ingness to bend over backwards to help you. Responding to all your calls and emails remains our #1 priority and favorite thing to do. Perhaps the biggest advantage that comes from this growth is the fact that we now have even more resources to apply to developing new accessories for meeting your spectroscopy challenges of the future.

When we sent out the Summer edition of PIKE Reflections a few months ago, we mentioned the need for additional parking. Fortunately, that project was completed just in time to welcome Matt Easley and Jessica Grimm, our newest members of the PIKE team. Matt is a new Product Specialist in the Heated and Automated Products manufacturing center. His previous experience in the automation group of Stoughton Trailers, has made him a perfect fit. You may already have talked with Jessica as she is the newest member of the PIKE sales team. Jessica is handling customer correspondence, order entry and order confirma-

aperture. These 2 new features will make the μ MAX IR Microscope even easier to use. We will be showing our new HT/HP Heat Chamber for our DiffusIR diffuse reflectance accessory. We will introduce new far-IR ATR crystal plates for our very popular MIRacle ATR and our VeeMAX with ATR accessories.

For hyphenated FTIR sampling we will introduce our new sample compartment TGA/FTIR accessory. This accessory fits most FTIR spectrometers and offers interface with most TGA instruments.

For our integrating sphere product line we will introduce our automated tablet analyzer option for our NIR Integrating Sphere. This



Matt Easley and Jessica Grimm - newest members of the PIKE team.



tions and has been a very busy lady since joining us.

We again wish to thank you for your business and the opportunity to help with your spectroscopy applications in 2006 as well. All of us at PIKE hope that you have a very **Happy and Healthy New Year!**

P.S. Please stop and see us at PITTCON! Our booth number is 2305.

new option provides the ability to do automated transmission analysis of pharmaceutical tablets.

We hope you can come to Orlando to see us and our products in person. See you there!



New options for the μ MAX IR Microscope

Use of Single-Reflection ATR for the Monitoring of UV - Cure Adhesives by Gabor Kemeny, PhD.

Ultraviolet radiation to enhance polymerization or other photochemical processes is gaining more importance in industrial production. Thermal cure of adhesives holding optical elements can introduce thermal stresses, distort the assemblies and misalign the optical systems [1]. UV curing of epoxies, silicones, urethanes and acrylates on the other hand offers a fast, well-controlled and easy way to achieve adhesion in industrial, military and space applications. New adhesives and new UV curing techniques [2] are introduced and thus there is a need to objectively monitor the cure characteristics. ATR is one of the preferred techniques for the monitoring of chemical reactions in situ. Pressure sensitive adhesives have been studied by real-time FTIR-ATR techniques [3]. Other non-destructive techniques, such as near-infrared and fluorescence have also been utilized [4, 5]. Concentrations of photo initiators, adhesion promoters, the cross linked adhesives and residual monomers need to be monitored to measure the rate of adhesive cure to assure that the adhesives are fully cured and to avoid introduced stress to optical assemblies [6].

EXPERIMENTAL

A Miracle™ ATR was placed in the sample compartment of an FTIR spectrometer. A single-reflection Diamond was chosen as the ATR element because it is chemically inert, and because it will resist the mechanical stress that will be necessary to remove the cured adhesives at the end of the experiments. A drop of the optical adhesive was placed on the diamond crystal to cover the ATR element with a thin, approximately half millimeter layer.

Curing sources are usually referred to as visible (above 400 nm), UV-A (350-400nm), UV-B (300-350 nm) or UV-C (below 300 nm). In our experimental setup a Model LS-7CB UV Lamp (Raytech Industries, Middletown, CT) was used with the illuminating lamps held just above the adhesive samples. The module has separate 4 W lamps for 365nm and for 254nm wavelengths. For the high inten-

sity visible illumination a Cuda Products (Jacksonville, FL) Model I-150 Fiber Optic Light Source was used, with the fiber optic probe held close to the sampling point.



Fig.1 PIKE Miracle™ with heated diamond ATR crystal. UV source with 254nm and 365nm was placed above the sample in close proximity.

Recommended UV cure wavelength was long-wave ultraviolet (320-400nm) for the first material tested. After sample placement, automatic scanning was started, with each spectrum point being an average of 2 scans and approximately 4 sec apart. The spectrum was unchanged during the first 20 sec until the lamp was turned on. The UV source producing 365nm has a few seconds warm-up time after which significant changes were observed in the spectra. Fig 2 indicates that the ATR spectra were detecting the disappearance of the free urethane and the forma-

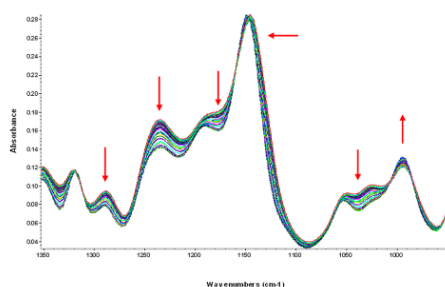


Fig 2 Changes observed on the ATR spectra of commercially available UV-cure optical adhesive. Arrows indicate observed spectral changes from onset. Total elapsed time was about 3 minutes.

tion of the cross linked species. Some bands did not change in intensity but shifted as the UV- cure progressed. In order to establish the kinetics of the UV-cure, either the spectral features of the appearance of the cross-links or disappearance of the oligomer / monomer could be used. The integrated area of the absorbance between 960 – 864 cm⁻¹ was increasing over the 3 minutes of the experiment, leveling off towards the 3 minute mark. The kinetics can be assessed by plotting the logarithm of the concentration change as a function of time - shown in Figure 3.

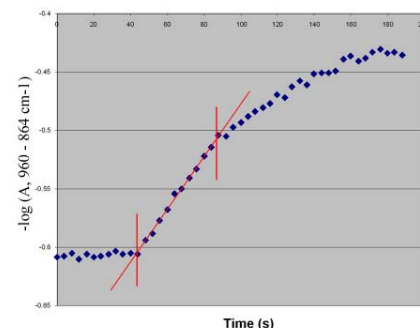


Fig 3. Increase in integrated area between 960-864 cm⁻¹. The logarithm of the area increases linearly between the red markers.

The first 40-45 seconds show a linear relationship followed by a slowing down of the cross-linking. The first part of the kinetics is linear with ample mobile and available moieties. As soon as the cross-linking starts to limit the mobility of the urethane groups, the progress of the cure slows down, indicated by the deviation from the first order kinetics. Please note that a relatively weak UV source was used. In industrial assembly, a 75W UV gun would be used with concentrated UV radiation directed at the parts to be attached. This high intensity illumination results in a few seconds of congealing time.

ATR kinetic measurements are useful for the comparison of different products and cure rates. Fig.4 shows the normalized kinetics of three commercially available optical adhesives. Using the same light flux, the infrared spectrum was measured every 10 seconds for a duration of 8 minutes. 5 scans with 4 cm⁻¹ resolution were averaged. Product 1 and 2 were very similar, whereas the cure of Product 3 was slower. The composition of the products examined were differ-

Spectroscopic Creativity - our Customer's Perspective

Dr. Heather A. Bullen, assistant professor of Chemistry at Northern Kentucky University, uses several PIKE ATR systems in her research. Dr. Bullen is an analytical chemist with various research interests in bioanalytical, nanomaterials, and surface chemistry. Utilizing the PIKE HATR flow cell system the Bullen research group investigates the role of different types of siderophores, organic molecules produced by many bacteria that have a high affinity for iron, in the initial adhesion of bacteria to metal oxide surfaces. By placing metal oxide sol-gel films directly placed onto the surface of the ZnSe ATR crystal and incorporating the flow cell siderophore/metal oxide interactions can be directly observed. A detailed study of the interaction of model catecholate siderophores with metal oxide substrates will be presented at the Spring 2006 ACS meeting in Atlanta.

UV Cure - cont.

ent, but all of them showed clear transition when exposed to the UV light indicated by the appearance, disappearance of multiple bands and wavelength shifts in the fingerprint region.

Cure kinetics with UV B, UV-C and Visible source revealed that the adhesive practically only cures with the 365 nm source. Some cross linking was observed with the visible source, probably due to a small fraction of the light output being below 400 nm. Optical adhesives are extensively used in optical assemblies, thus UV-C radiation cure for these products would not be practical because most common optical glasses would absorb the short wave UV radiation and prevent complete cure. Visible light on the other hand would cause premature binding of the optical components before the alignment is complete. Some UV-cure adhesives are also heat curable. In this experiment we did not change the temperature of the sample although the particular diamond ATR was the temperature controlled version, as seen on Fig 1. No heating of the samples was detected by the use of either illumination source or wavelength that could introduce

The knowledge gained from this work will advance our fundamental understanding of biofilm development and assist in developing materials capable of preventing biofilm formation. Collaborative research with Dr. Martines, Biological Sciences NKU, involves using the VeeMAX II ATR flow cell system to evaluate the potential of customizable dendrimers to serve as drug delivery agents across the blood brain barrier (BBB). Depth profiles probe the interaction of the dendrimers with model BBB. Additionally, the Bullen research group has developed a collaboration with Dr. Barton, Biological Sciences, and Dr. Bertog, Geology, at NKU to evaluate microbial processes and interactions in cave environments. Metabolic interactions of organisms in oligotrophic environments have been evaluated using the MIRacle Si ATR fitted with a high pressure clamp. Analysis of unreacted and reacted CaCO_3 surfaces indicate that organisms living in low biomass environments change the local geochemistry of the environment with identifiable metabolic processes.



Dr. Heather Bullen (left) with undergraduate research assistant Heather Gulley (right) who received an ACS Division of Analytical Chemistry I. M. Kolthoff award to present her research using a PIKE HATR at the Spring 2006 ACS Meeting.

These research projects are supported by NSF, Merck/AAAS Undergraduate Science Research Program, and the Center for Integrated Natural Science and Mathematics at Northern Kentucky University. For more information on the above research you can email Dr. Bullen at bullenh1@nku.edu or contact her at her office 859-572-5411.

a bias to the UV cure kinetics. Other uses for the monitoring of photochemistry with a single-bounce ATR are general photochemistry, photo-bleaching, light activated tooth whitening gels and for any other UV induced reactions that have infrared active molecular groups participating in the photo reactions.

References:

1. Bachmann A, Arnold J, Langer N.: "Movement Between Bonded Optics", Dymax Corp. Tech. Bulletin, Sept. 2001
2. Panico L.: "Pulsed UV Curing Provides User-Friendly Solutions to Tough Problems", Adhesives Age, January 1997
3. Scherzer, T., Tauber, A, Mehnert R.: "UV Curing of Pressure Sensitive Adhesives Studied by Real-Time FTIR-ATR Spectroscopy", Vib. Spec., **29** (1), pp. 125-131 (2002).
4. Santos A.F. et al: "Monitoring and Control of Polymerization Reactors Using NIR Spectroscopy" Polymer-Plastics Technology and Engineering" **44** (1), pp 1-61 (2005)

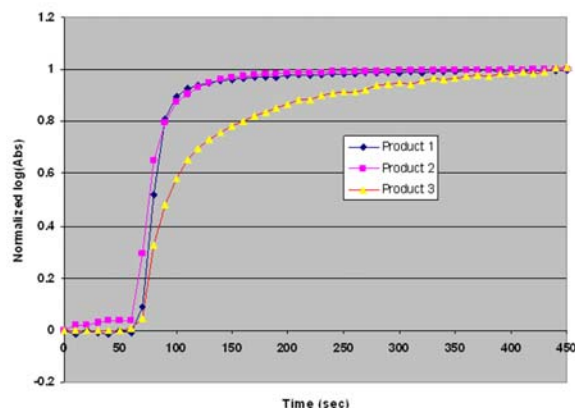


Fig 4. Comparison of the area of increasing band intensities in the 1300-850 cm^{-1} region for three commercially available UV-cure adhesives. 365nm illumination was used with the same light intensity for all samples. The source was turned on approximately 1 minute after scanning started.

5. Peinado C. et al: "Fluorescent Probes for Monitoring the UV Curing of Acrylic Adhesives" Macromol. Chem. Phys., 202(9), pp 1924-1934 (2001)
6. Bachmann, A: "Advances in Light Curing Adhesives", SPIE Conference, Paper #4444-20, August 2001.

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- *ATR Monitoring of UV - Cure Adhesives*
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New offers for Spectroscopy Sampling from PIKE! by Krista Garcia

Yes, I am a new Mom and loving every minute of it! You can see my son, Davian in the photo in our Events Calendar. Sorry for straying off the subject - I just can't help myself!

As many of you took advantage of the MIRacle promotion the end of last year, we realized promotions are a great way to show appreciation to our valued customers. So here we go again!

We know many of you use our MIRacle and HATR accessories on a regular basis.

With regular use, sometimes these crystals need to be refurbished by replacing the used crystal with a new one. We do our best to refurbish your crystals as quickly as possible, usually within a few days. If you need even faster service, we have a great new opportunity for you! If you purchase a new crystal as a back-up plate in addition to the refurbishment of your existing crystal, we will give you the new plate at the same price as the refurbished one. What a great offer!! This means you will have zero down time to restore your MIRacle or HATR accessory. But hurry, this is a limited-time offer!!

You have heard of our new in-sample compartment microscope - the μ MAX. We have received a lot of interest in the μ MAX and are providing a great introductory offer. If you purchase a new μ MAX, you can receive the ATR objective for FREE!!



Please contact me directly at garcia@piketech.com or by phone (608) 274-2721 to find out more about these exciting offers!

\$1,000 Question & Answer

Thank you for all of your replies to our last \$1,000 questions. Provide 3 reasons why CsI is not a good material for ATR? The correct answers include:

- It's too soft*
- Its refractive index is too low*
- It is water soluble*
- It reacts with many organic compounds*

We had numerous correct responses and our judges panel recognized that we had an exceptionally brilliant additional answer provided by Leo Gauthier, Jr. from Johns Hopkins University. Leo added that CsI

as an ATR crystal would be confused with a new CSI forensic series! Thank you, Leo for your wit and congratulations - you are our winner!

Here is our new \$1,000 question; something to think about for ATR!

What volume of liquid is required to provide full ATR depth of penetration on the MIRacle single reflection diamond crystal?

Send your answers to sales@piketech.com and you could be the winner of a \$1,000 accessory discount.

PIKE Events Calendar

Irene is a Grandmother!
 Jacob Philip born to Elizabeth on November 25, 2005



Krista is a Mom!
 Davian born on October 21, 2005



PITTCON 2006
 March 13 - 17
 Orlando, FL



Please stop by our booth (#2305). We hope to see you there!