Curing Monitoring of Two-Component Epoxy Adhesives at THz Frequencies

A. Soltani, T. Probst, S. Sommer and M. Koch

Department of Physics and Materials Sciences Center, Philipps-Universität Marburg, Renthof 5, 35032 Marburg, Germany

soltanim@physik.uni-marburg.de

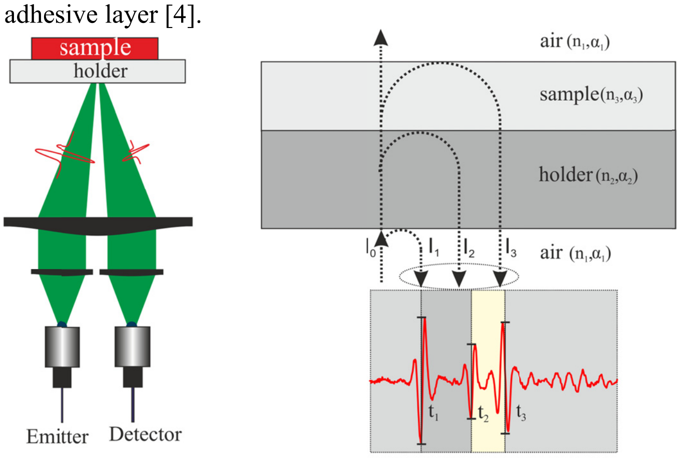
***Abstract*— We demonstrate real time monitoring of the curing of two-component epoxy adhesives at THz frequencies. Our method is based on THz time domain spectroscopy (TDS) in reflection geometry. We monitor the curing of two-component adhesive over ten hours and extract refractive index, absorption and thickness.**

I. INTRODUCTION

Transmission geometry is more convenient for nondestructive testing in the THz range [1]–[3] . Yet, there are cases when it may be neither convenient nor possible to access both sides of the material to be tested and then reflection geometry is the only option. Here we use THz time-domain spectroscopy in simple and straightforward reflection geometry to monitor the curing of two-component epoxies.

II. DESCRIPTION OF THE SETUP

The measurement setup which is schematically shown in Fig. 1 (a) is described in detail in ref [4]. A schematic of the sample holder with the adhesive sample and the THz pulses reflected from each interface is shown in Fig. 1b. The first three reflections originate from the interfaces between air and PE-HD (layers 1 and 2), PE-HD and the sample under test (layers 2 and 3), and the sample and air (layers 3 and 4), respectively. By using Fresnel's equations, the amplitudes and time position of each pulse $(t_1, t_2\ , t_3)$ we are able to monitor refractive index, absorption and thickness of the adhesive layer [4].



(a) (b)

Figure1: (a) schematic of the THz reflection geometry; (b) reflected THz pulses from air, holder and sample interfaces.

III. MATERIAL PARAMETER CALCULATION

In order to calculate optical parameters of the adhesive layer, we considered intensities (I_1, I_2 and I_3) which is proportional to peak-peak amplitude of the detected THz pulses (Fig. 1b). By following the formulation in [5] one could write:

$$n_{adhesive} = n_{sample} = \frac{n_2\sqrt{\frac{I_2}{I_0}}e^{-\alpha_2 d_2}\sqrt{\frac{1}{T_{21}T_{12}}+1}}{1-\sqrt{\frac{I_2}{I_0}}e^{-\alpha_2 d_2}\sqrt{\frac{1}{T_{21}T_{12}}}} \qquad (1)$$

where d_2 is the known thickness of the holder layer. By considering the zero crossing points of the 2nd and 3rd reflected pulses ($\Delta t = t_3 - t_2$), one may conclude:

$$d_{adhesive} = \frac{\Delta t \times c_0}{2 \times n_{adhesive}} \qquad (2)$$

Finally, by considering $n_{adhesive}$ and $d_{adhesive}$, we could calculate the absorption of the adhesive layer:

$$\alpha_{adhesive} = -\frac{1}{2d_3}\ln\left(\frac{\left(\frac{I_3}{I_2}\right)\times\Gamma_{23}}{T_{32}\times\Gamma_{31}\times T_{23}}\right) \qquad (3)$$

where $\Gamma_{ij}$is the Fresnel reflection coefficient between two interfaces. The described method supports us to calculate refractive index, absorption coefficient and thickness of the sample directly from time domain signals. The main condition for this method is to consider all the mediums non-dispersive. Here we used PE-HD for holder, which is non-dispersive in THz range [6], and also provided narrow band measurement to fulfill this condition. This method is limited when the optical parameters of the holder are close to the sample, because of the non-detectable reflection from this interface.

IV. RESULTS

Fig. 2 (a) displays the intensities of the reflected pulses for two different samples over time. Fig. 2 (b-d) shows the calculated refractive index, thickness and THz absorption respectively for Collopox 902 and Collopox 903 monitored over ten hours. The data suggest that adhesive layer needs at least two hours to reach their stable structure.

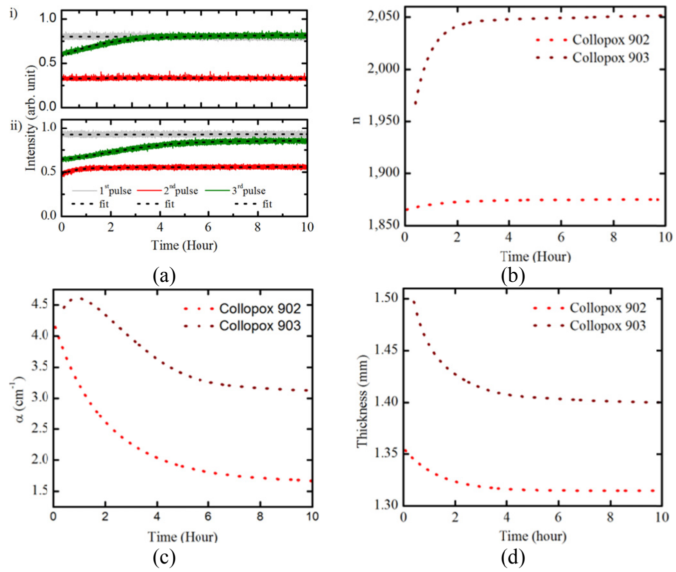


Figure 2: (a) Intensity of the I_1, I_2 and I_3 for two samples, (b-d) monitored refractive index, absoroption and thickness over 10 hours [4].

V. Conclusion

Simple but robust reflection geometry is used to monitor the real time curing of epoxy adhesives. A key feature of the method is the novel and robust signal processing method, based on the peak-to-peak amplitude and pulse position of reflections from known and unknown interfaces. This design is well suited for NDT applications in industrial environment.

References

[1] F.-P. Liu, R.-Z. Li, J. Li, H.-G. Chen, and C.-C. Yang, "Group Time Delay in the Reflection of P-Polarized Electromagnetic Plane Waves at a Stratigraphic Interface," *Math. Geosci.*, vol. 40, no. 7, pp. 813–829, Mar. 2008.

[2] C. D. Stoik, M. J. Bohn, and J. L. Blackshire, "Nondestructive evaluation of aircraft composites using transmissive terahertz time domain spectroscopy," *Opt. Express*, vol. 16, no. 21, pp. 17039–17051, 2008.

[3] E. K. Rahani, T. Kundu, Z. Wu, and H. Xin, "Mechanical damage detection in polymer tiles by THz radiation," *Sensors Journal, IEEE*, vol. 11, no. 8, pp. 1720–1725, 2011.

[4] T. Probst, S. Sommer, A. Soltani, E. Kraus, B. Baudrit, G. E. Town, and M. Koch, "Monitoring the Polymerization of Two-Component Epoxy Adhesives Using a Terahertz Time Domain Reflection System," *J. Infrared, Millimeter, Terahertz Waves*, vol. 36, no. 6, pp. 569–577, 2015.

[5] C. a Balanis, *Advanced Engineering Electromagnetics*, vol. 52. Wiley Online Library, 1989.

[6] S. Wietzke, C. Jansen, F. Rutz, D. M. Mittleman, and M. Koch, "Determination of additive content in polymeric compounds with terahertz time-domain spectroscopy," *Polym. Test.*, vol. 26, no. 5, pp. 614–618, Aug. 2007.