AN-NIR-089

Quality Control of Laminates

Improved PCB production testing with NIR spectroscopy

Summary

In the semiconductor industry, thermoset resins combined with fabric or paper are used as an intermediate layer between substrates of printed circuit boards (PCB). These polymer-based sheets (laminates) are chosen depending on thickness and their thermomechanical and electrical characteristics. Important quality parameters are tensile and shear strength, the glass transition temperature, expansion coefficient, and dielectric constant.

Near infrared spectroscopy (NIRS) is a fast, non-destructive and easy-to-use analytical method which allows the measurement of multiple parameters in less than a minute. The following Application Note describes the determination of the transition time of PCB laminates by NIRS, a parameter correlating with the thickness, glass transition temperature, and tensile strength of the material.

Experimental Equipment

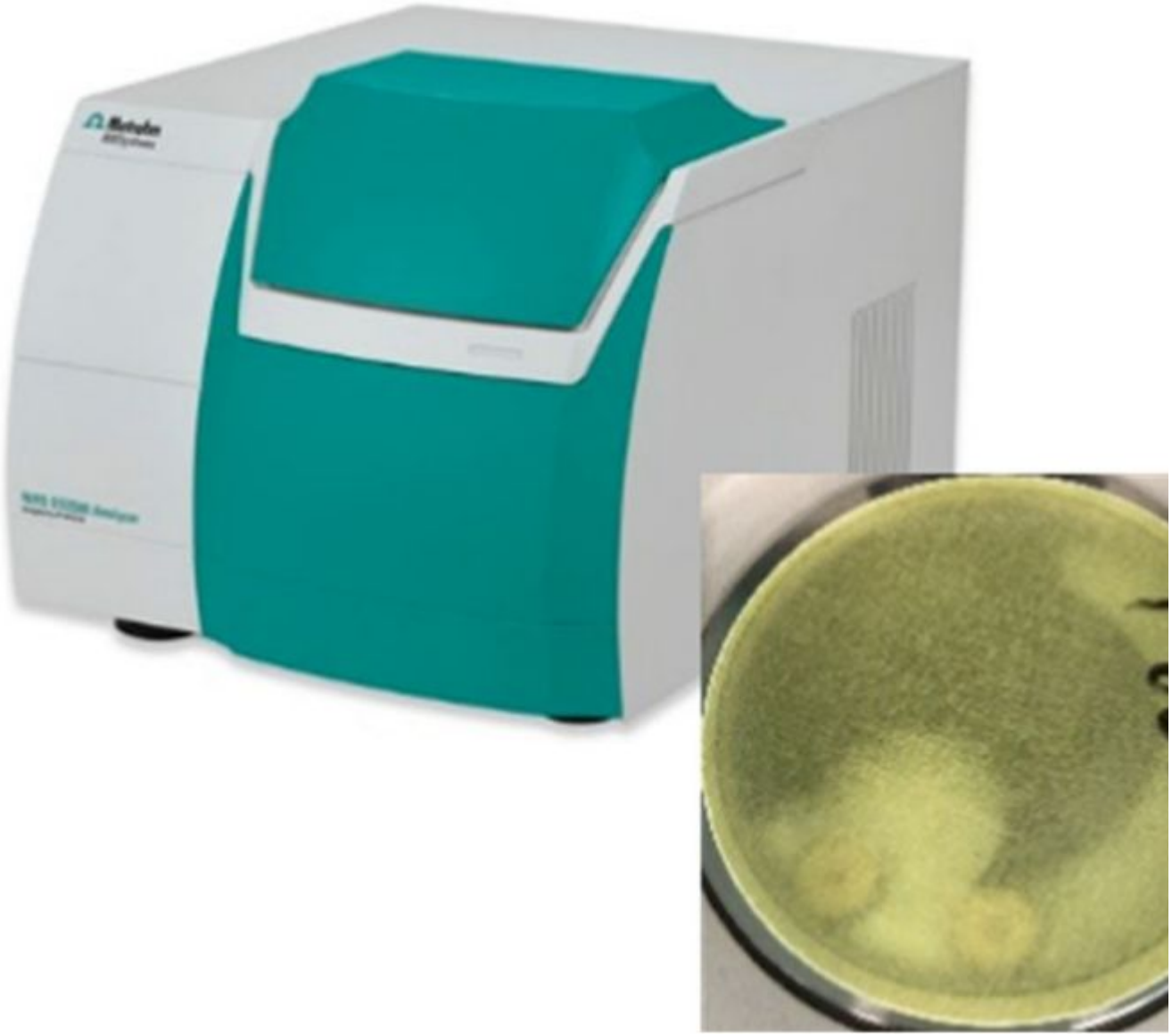

Figure 1. DS2500 Solid Analyzer and a polymer sheet resin.

520 spectra of samples were collected using a Metrohm DS2500 Solid Analyzer and the Vision Air Complete spectroscopy software. The laboratory values for the transition time were determined by melting the samples, and values between 60 and 126 seconds were obtained. The data set consisting of spectra and lab values was split into a calibration and validation set (1:1). Outlier detection was performed on pre-processed spectra (2^{nd} derivative and SNV) using a maximum distance algorithm. The NIR prediction model was created with the equipment described in **Table 1** and validated using the validation set.

Table 1. Hardware and software equipment overview

Equipment	Metrohm number
DS2500 Solid Analyzer	2.922.0010
DS2500 large sample cup	6.7402.050
Vision Air 2.0 Complete	6.6072.208

2.922.0010 - DS2500 Solid Analyzer

Robust near-infrared spectroscopy for quality control, not only in laboratories but also in production environments.The NIRS DS2500 Analyzer is the tried and tested, flexible solution for routine analysis of solids, creams, and optionally also liquids along the entire production chain. Its robust design makes the NIRS DS2500 Analyzer resistant to dust, moisture, vibrations, and temperature fluctuations, which means that it is eminently suited for use in harsh production environments.The NIRS DS2500 covers the full spectral range from 400 to 2500 nm and delivers accurate, reproducible results in less than one minute. The NIRS DS2500 Analyzer meets the demands of the pharmaceutical industry and supports users in their day-to-day routine tasks thanks to its simple operation.Thanks to accessories tailored perfectly to the instrument, optimum results are achieved with every sample type, no matter how challenging it is, e.g. coarse-grained solids such as granulates or semi-solid samples such as creams. The MultiSample Cup can help improve productivity when measuring solids, as it enables automated measurements of series containing up to nine samples.

6.7402.050 - DS2500 large sample cup

Large sample cup for the spectral recording of powders and granulates in reflection at various sample positions using the NIRS DS2500 Analyzer.

6.6072.208 - Vision Air 2.0 Complete

Vision Air - Universal spectroscopy software. Vision Air Complete is a modern and simple-to-operate software solution for use in a regulated environment.Overview of the advantages of Vision Air: Individual software applications with adapted user interfaces ensure intuitive and simple operation; Simple creation and maintenance of operating procedures; SQL database for secure and simple data management; The Vision Air Complete version (66072208) includes all applications for quality assurance using Vis-NIR spectroscopy: Application for instrument and data management; Application for method development; Application for routine analysis; Additional Vision Air Complete solutions: 66072207 (Vision Air Network Complete); 66072209 (Vision Air Pharma Complete); 66072210 (Vision Air Pharma Network Complete);

Results

The obtained correlation graph displays a high correlation (R^2 = 0.95) between transition times predicted by NIR and the primary lab method (**Figure 3**). The validity of the prediction model is confirmed by the figures of merit (Ratio SEC to SECV < 20%), confirming that NIR spectroscopy is a suitable analytical method to determine transition times of PCB laminates.

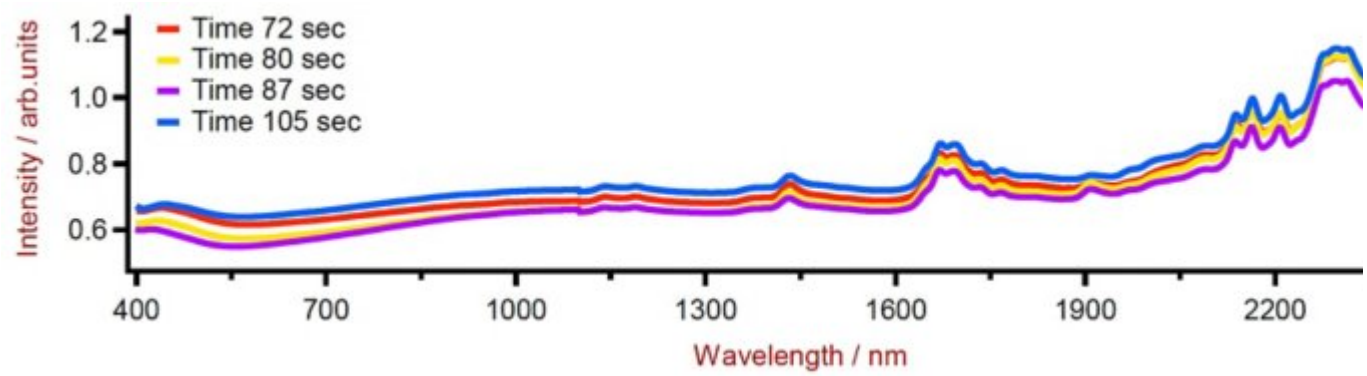

Figure 2. Vis-NIR spectra of polymer resins measured on a DS2500 Solid Analyzer.

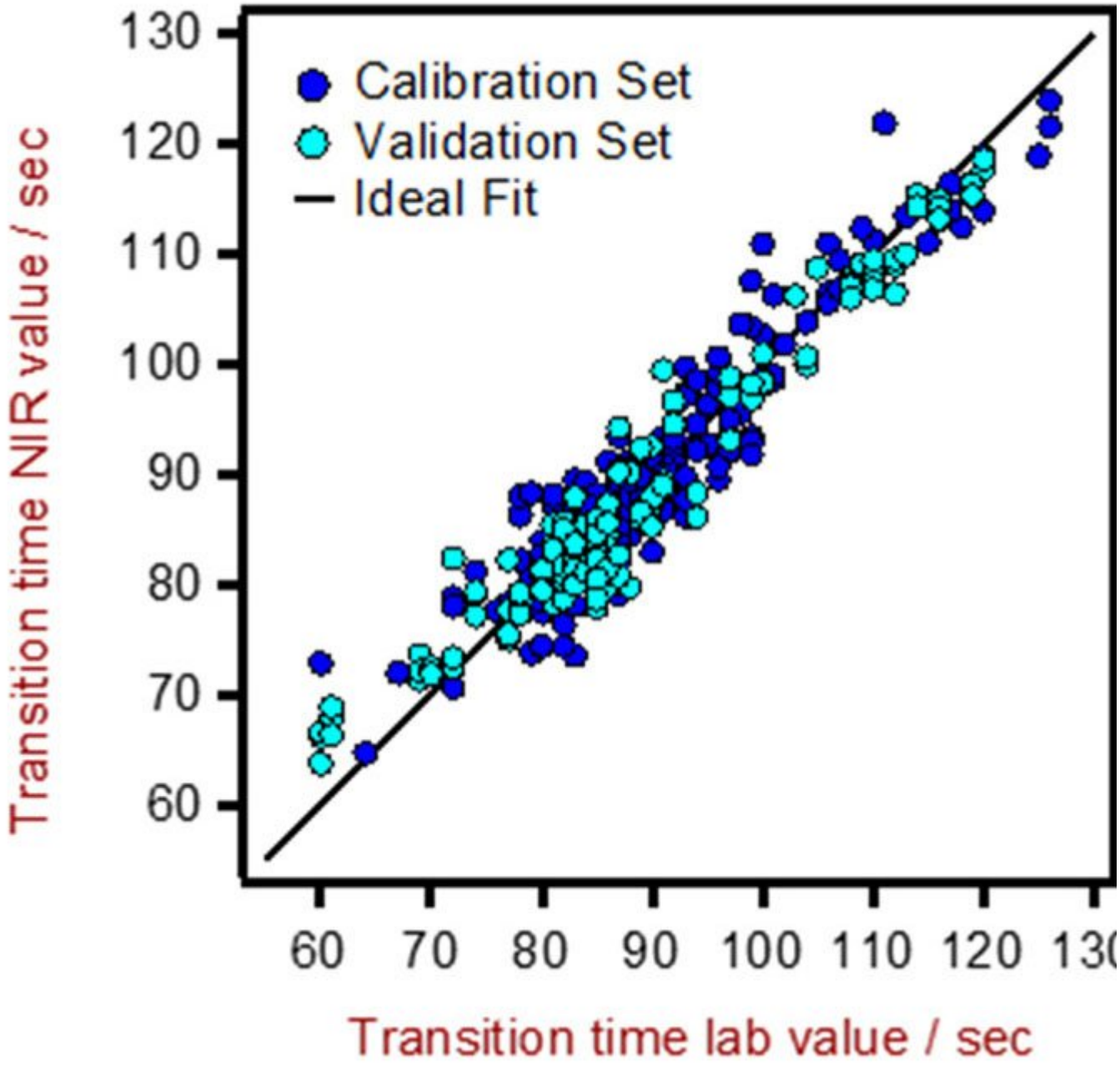

Figure 3. Correlation diagram for the prediction of transition times using a DS2500 Solid Analyzer.

Table 2. Figures of merit for the prediction of transition times using a DS2500 Solid Analyzer.

Figures of merit	Value
R^2	0.95
Standard error of calibration	3.64 s
Standard error of cross-validation	4.02 s

Conclusion

This application note demonstrates the feasibility of the DS2500 Solid Analyzer for the determination of transition times of polymer resins. Vis-NIR spectroscopy enables a fast determination without any sample preparation and therefore represents a suitable tool to check the transition kinetics of PCB laminates.

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