Specac
Application Note

In-situ high temperature, high pressure analysis of resin-based adhesives for prediction of final properties

High Temperature
High Pressure Cell

INSIDE: Discover a method for high temperature, high pressure analysis of viscous resins and pastes.

Introduction

FTIR can accurately determine the change in functional groups and the adsorption process of a material, allowing rapid screening of samples prior to further investment of R&D resources. To replicate a material's on-site or real-life condition, it is necessary to carry out in-situ testing because material properties change depending on temperature and pressure. It is vital to pick the right *in-situ* high temperature high pressure accessory for specific tests and samples.

Specac's High Temperature High Pressure Cell (HTHP) is a tri-modal accessory, especially suitable for the *in-situ* analysis of solid powders, viscous colloids, pastes, and adhesives by transmission mode, and dark-coloured materials by reflectance mode. The HTHP can reach a maximum temperature of 800 °C for sample analysis, and its separately heated windows reduce interference from condensation. The temperature ramp rate can be adjusted on the controller to quickly reach the desired temperature.

High optical throughput reaction chamber for IR analysis in multiple configurations: transmission, reflectance, and decomposition.

- Sealed sample chamber with controlled atmosphere.
- Temperature to 800 °C and pressure from vacuum to 68 bar (1000 psi).
- Converts to specular reflectance mode for opaque or reflective samples using external mirrors.
- Sample pans allow analysis of gases released from thermal decomposition of solids.
- Unmatched performance.
- Ideal for characterisation of coal, catalysts, resins/polymers, ceramics, and minerals.

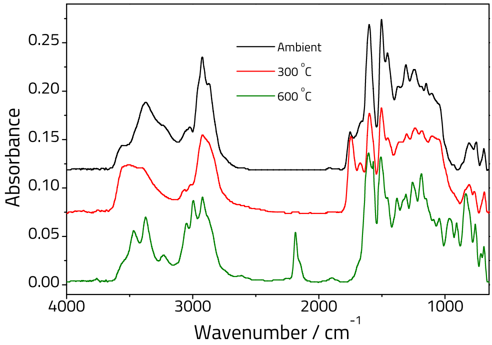


Figure 1: *Monitoring the change in FTIR characteristic peaks of a resin sample at varying temperatures.*

Experimental

Due to the viscous nature of colloids and resins, these samples cannot be pressed into power nor be analysed by DRIFTS. Therefore, one can use the 'smearing method' to smear a small amount of viscous sample on a compress KBr pellet for effective transmission analysis *in-situ*, at varying temperatures.

1. Record background spectrum of a compressed KBr pellet with 13 mm diameter at ambient temperature.

2. Use a sample needle to extract 1-2 g of resin-based adhesive sample.

3. Smear a small amount of the adhesive sample evenly onto the KBr pellet (or a new KBr pellet).

4. Record sample spectrum at ambient temperature.

5. Increase temperature to 300 °C, then 600 °C, at a ramp rate of 20 °C/minute.

Results and Discussion

An adhesive is usually a viscous fluid. Some need to be applied to a substrate at high temperatures, where its viscosity is significantly reduced to ease its wettability across the substrate surface. However, high temperatures may bring about the decomposition of key components such as the solvent, base resin, and antioxidants used in the manufacture of an adhesive, resulting in poor adhesion performance despite ease of application.

This decomposition can be monitored using Specac's HTHP, to produce FTIR spectra that reflect the change in the chemical composition of the key components as a function of temperature. Subsequently, the thermal resistance of the key components can be determined, and the adhesion performance of the final product can be predicted.

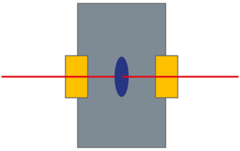
The IR absorption at 1746 cm^{-1} at 300 °C (Fig. 2, red spectrum) is attributed to the formation of 2-ethoxyethyl acetate due to solvent decomposition, while the -OH absorption at 3496 cm^{-1} is attributed to the evaporation of moisture from the sample. The reduction of antioxidants at 600 °C (Fig. 2, pink spectrum) is observed by the appearance of an IR peak at 2993 cm^{-1} and disappearance of IR peak at 1746 cm^{-1}.

These observations give an indication of the highest temperature that the solvent, base resin, and antioxidant components can withstand before decomposition, and in turn, their suitability for high temperature applications. The FTIR results also serve as an indicator of whether the final product can provide good adherence to a substrate, without the need to apply the adhesive to an actual substrate to measure its tensile strength, saving R&D resources. It can take up to a week to perform measurements of tensile strength on multiple samples of cured adhesives, compared to a few minutes when using FTIR to predict the end performance of an adhesive.

Conclusion

It is evident from the spectra above that the HTHP cell is superb at monitoring the change in a resin-based adhesive's chemical make-up at elevated temperatures. This is due to its high light throughput and absorbance during transmission analyses. The growth and decay of peaks assigned to functional groups in the solvent, base resin, and antioxidants in the adhesive sample play an important role in predicting results of *in-situ* testing.

HTHP modes of operation

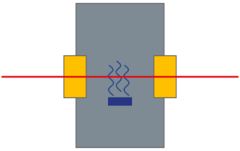


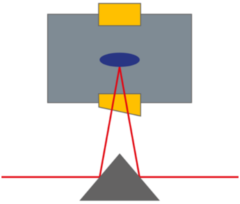
Transmission Mode

The sample is prepared as a pellet or thin film of 13 mm diameter and up to 6 mm thickness. It is clamped directly into the Incalloy sample heating stage.

Decomposition Mode

The sample is placed in an Incalloy pan which rests on the sample heating stage. Evolved gases rise upwards into the path of the beam. The pathlength in this configuration is 30 mm.





Specular Reflectance

A modified baseplate with external mirrors is used. The cell is turned through 90 degrees to rest on its side with one window facing down towards the external mirrors. The angle of incidence is 15° and a specially modified window assembly is provided to help reduce interference fringing.

AN21-07

United Kingdom
sales@specac.co.uk

China
frank.li@specac.com

United States
sales@specac.com

Singapore
kamhar.woo@specac.com

UK: +44 (0)1689 892 902 | US: +1 866 726 1126

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www.specac.com

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