APPLICATION**NOTE**

Thermal Analysis of a PTFE/Graphite Compound by Means of the New *Perseus* STA 449 FT-IR Coupling

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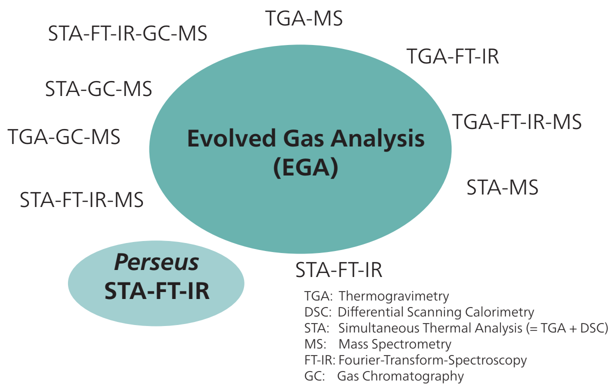
Introduction

Evolved gas analysis (EGA) coupled to thermal analyzers such as thermogravimetry (TGA) or simultaneous thermal analysis (STA) which refers to simultaneous TGA-DSC is well established since it greatly enhances the value of TGA or TGA-DSC results. The sensitive and selective Fourier Transfrom Infrafed (FT-IR) technique is in particular useful for the analysis of organic molecules but also for infrared-active permanent gases evolved during most

1 NETZSCH *Perseus* STA 449: The Bruker type "alpha" FT-IR spectrometer coupled directly to an STA 449 *Jupiter*® simultaneous thermal analyzer equipped with an optional automatic sample changer (ASC). The sample space of the furnace, the heated coupling interface as well as the gas cell of the FT-IR spectrometer are shown partially transparent in order to display the path of the evolved gases [1].



2 Various NETZSCH combinations of gas analysis techniques coupled to thermal analyzers

decomposition processes. Such permanent gases like CO_2 or SO_2 are gaseous at ambient conditions.

The coupling interface between thermal analyzers and FT-IR spectrometers is usually realized using heated adapters and a flexible, heated transfer line where the heating is required to avoid condensation of evolved gases on the way to the FT-IR instrument. Although integrated software solutions are available, thermal and gas analyzers are still physically separated. The path through the transfer line causes furthermore a delay between the release and the detection of the evolved gases and in some cases condensation or interaction effects.

For this work, the new direct *Perseus* coupling of an STA instrument and an FT-IR spectrometer without a transfer line was used [1]. A very small FT-IR spectrometer is directly mounted on top of the STA furnace leading to a compact and fully integrated STA-FT-IR coupling system called *Perseus* STA 449 (see figure 1). *Perseus* is a new member of the NETZSCH family of coupling systems as illustrated in figure 2.

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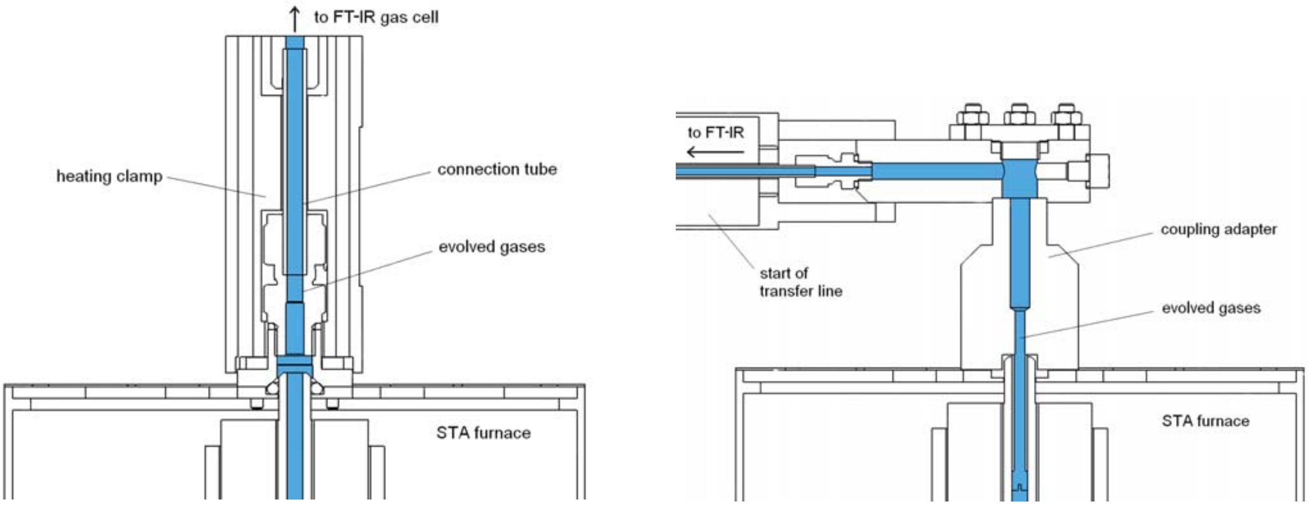

3 Interface of direct FT-IR coupling NETZSCH *Perseus* STA 449 (left) compared to coupling with a (flexible) transfer line (right) [1]. The path of the evolved gases is highlighted in blue.

The short interface to the STA furnace (see figure 3) as well as the gas cell of the FT-IR spectrometer is heated in order to minimize the risk of condensation. Furthermore, no liquid nitrogen is required since the FT-IR detector of DLaTGS type is operating at room temperature.

The basic instrument NETZSCH STA 449 ***F1/F3*** *Jupiter*® enables to measure high-resolution TGA and DSC or DTA simultaneously in a wide temperature range of -150°C to 2400°C depending on the furnace and sample carrier used.

Experimental

A PTFE/graphite compound with an initial sample mass of 11.54 mg was measured in Pt crucibles with pierced lids at a heating rate of 10 K/min. The gas atmosphere (flow rate 70 ml/min) was switched from pure argon to synthetic air at 870°C. A TGA-DSC sample carrier type S and a rhodium furnace were applied. The TGA-DSC results were baseline-corrected (empty-run signals were subtracted) and FT-IR acquisition was carried out at a resolution of 4 cm^{-1} and 16 scans were averaged for one FT-IR spectrum where one scan took about 1s.

Results and Discussion

The *Perseus* coupling is well suited for many applications [1]. As an example, the results on the above-mentioned PTFE/graphite compound – which can, for example, be applied as a lubricant – are shown [2]: figure 4 depicts the TGA-DSC results together with the Gram-Schmidt curve. The Gram-Schmidt curve portrays the intensity change of the entire IR absorption detected. At approx. 349°C (peak temperature), the DSC signal reveals an endothermic effect which is due to melting of the PTFE content. Between about 480°C and 620°C, a mass-loss step of 97.4% occurs together with an endothermic DSC effect and a peak in the Gram-Schmidt signal. In this range, pyrolytic decomposition of the PTFE content takes place. At 870°C, the gas atmosphere was switched from inert to oxidative, leading to exothermic burn-up of the graphite content of approx. 2.1%. The residual mass of about 0.6% is most probably due to a ceramic filler.

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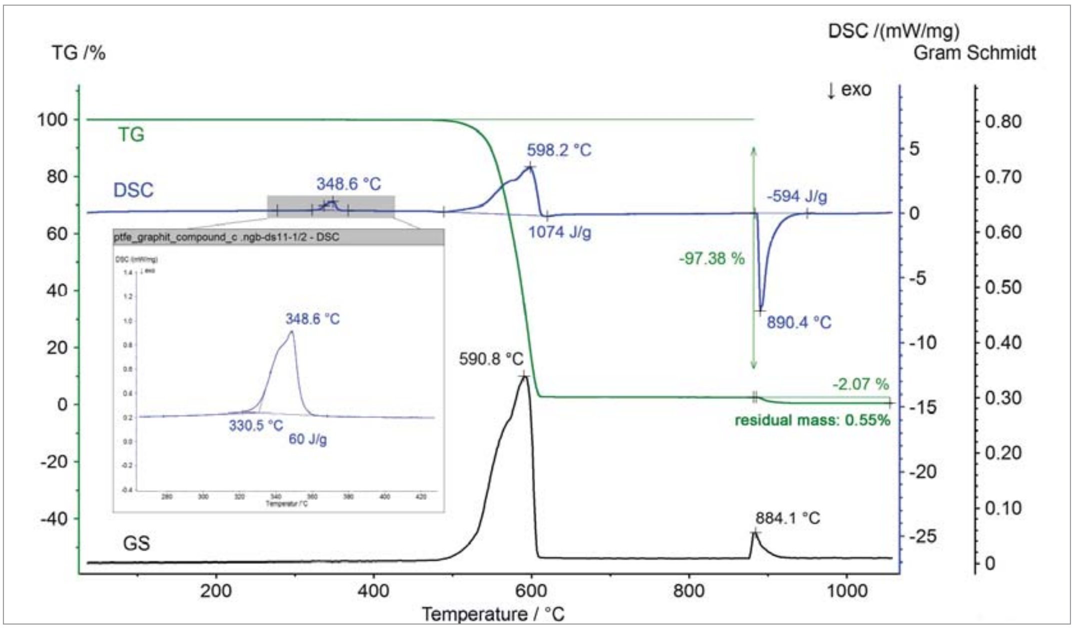
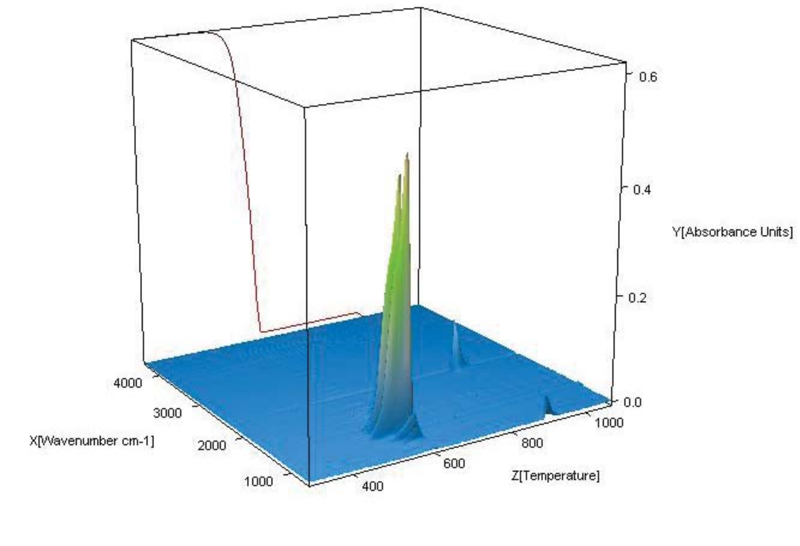


4 Mass changes (TGA), heat-flow rate (DSC) and Gram-Schmidt signal (GS) of the PTFE/graphite compound as a function of temperature

The "3-D cube" presented in figure 5 shows the IR absorption as a function of wave number and temperature, together with the TGA curve. During the first mass-loss step, the well-known absorption bands of tetrafluoroethylene, C_2F_4, can primarily be identified in the range between 1100 cm^{-1} and 1400 cm^{-1} (as well as traces of HF in the range between 4000 cm^{-1} and 4200 cm^{-1}). The bands detected during the second mass-loss step, primarily in the range between 2200 cm^{-1} and 2400 cm^{-1}, can be attributed to CO_2 formed during combustion. Finally, figure 6 depicts the characteristic integration traces for C_2F_4 and CO_2 as a function of temperature showing again an excellent correlation between mass-loss steps and evolved gases.



5 IR absorption as a function of temperature and wave number together with the TGA curve

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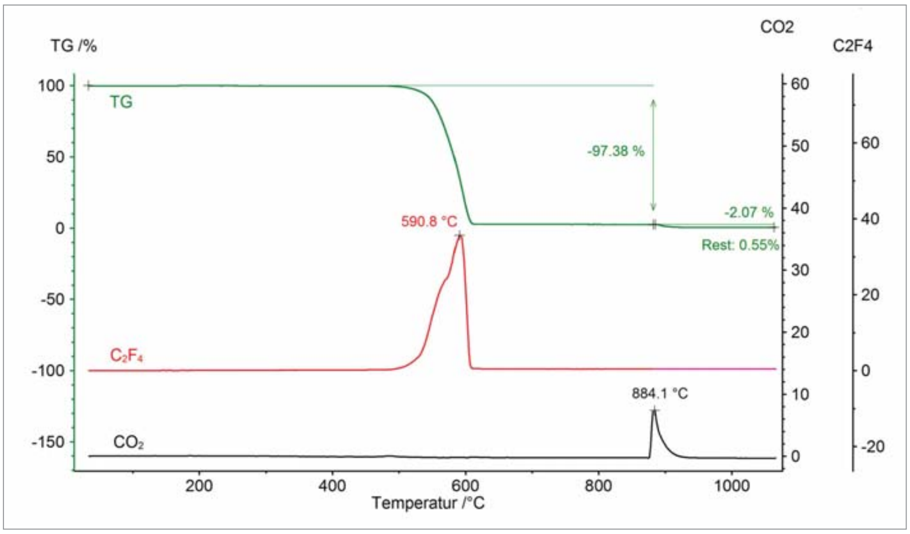

6 Mass changes (TGA) and FT-IR integration traces for C_2F_4 and CO_2 as a function of temperature

Conclusion

The application example presented demonstrates that *Perseus* allows for simultaneous recording of TGA and DSC and, at the same time, detection of the gases evolved by means of FT-IR. The entire STA-FT-IR results allow for quantification and identification of each sample component since initially unidentified gases can often be identified by means of a database search [1]. A very good correlation between the detected mass-loss steps and the gases evolved was shown which is a benefit of the direct coupling interface. All in all, the new Perseus STA 449 ***F1/F3*** is a high-performance, direct STA-FT-IR coupling without any transfer line that sets itself apart particularly by virtue of its compactness.

Literature

[1] A. Schindler, G. Neumann, A. Rager, E. Füglein, J. Blumm, T. Denner: J Thermal Anal Calorim, DOI 10.1007/s10973-013-3072-9

[2] A. Schindler: Onset[10], /www.netzsch-thermal-analysis.com/fileadmin/user_upload/netzsch-thermal-analysis/products/pdfs/NETZSCH-OnSet_10-E_1112w.pdf

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