NETZSCH

Analyzing & Testing

Simultaneous Thermal Analysis

Method, Technique, Applications

Leading Thermal Analysis

Method

For 50 years, NETZSCH Analyzing & Testing has been a leading manufacturer of high-performance thermal analysis systems – flexible, sophisticated and technically outstanding. Our customers' wishes and requirements are our guidelines. This, combined with experience and innovation, allows us to consistently set new benchmarks and standards in the field of thermal analysis instrumentation. Our success is a result of the creativity and enthusiasm of our engineers and scientists and our close cooperation with you as our customer.

Simultaneous Thermal Analysis generally refers to the simultaneous application of Thermogravimetry (TGA) and Differential Scanning Calorimetry (DSC) to one and the same sample in one instrument. The advantages are obvious: The test conditions are perfectly identical for the TGA and DSC signals (same atmosphere, gas flow rate, vapor pressure on the sample, heating rate, thermal contact to the sample crucible and sensor, radiation effect, etc.). Furthermore, it improves sample throughput as more information is gathered from each test run. Since the early years of its existence, NETZSCH has given high priority to the development and continuing optimization of its Simultaneous Thermal Analyzers yielding the new STA 449 ***F3*** *Jupiter*®. It meets nearly all respective instrument and application standards for TGA and DSC systems including: ISO 11357, ISO 11358, ASTM E 967, ASTM E 968, ASTM E 793, ASTM D 3895, DIN 51004, DIN 51006, DIN 51007.

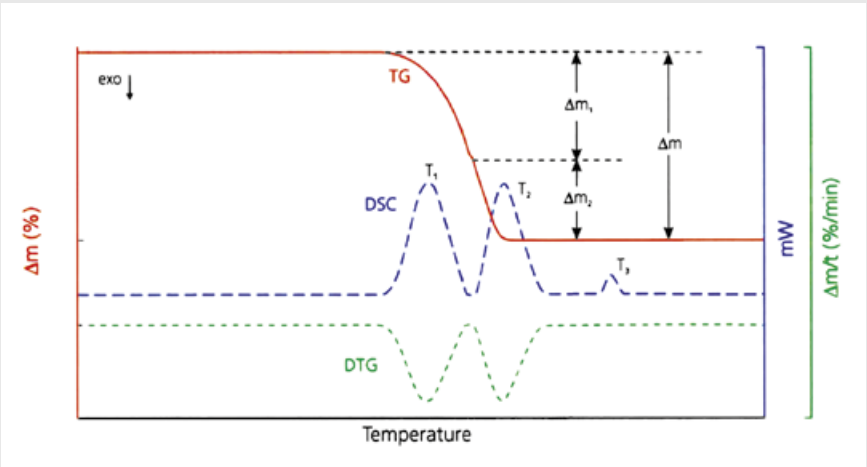
Measured signals in an STA

DSC analysis possibilities:

- Melting/crystallization behavior
- Solid-solid transitions
- Polymorphism
- Degree of crystallinity
- Glass transitions
- Cross-linking reactions
- Oxidative stability
- *Purity Determination*
- Specific heat
- *Thermokinetics*

TG analysis possibilities:

- Mass changes
- Temperature stability
- Oxidation/reduction behavior
- Decomposition
- Corrosion studies
- Compositional analysis
- *Thermokinetics*



The NETZSCH STA 449 ***F3*** *Jupiter*® is a robust, flexible, easy-to-operate instrument for determining caloric effects (transformation temperatures and enthalpies) and mass changes at the same time. The top-loading Simultaneous Thermal Analyzer can easily be adjusted to nearly all possible applications by selecting the optimum furnace, installing the ideal sensor and using the proper accessories. It combines a high-performance Heat-Flux DSC with a microgram-resolution thermobalance, thereby offering an unmatched sample load and measurement range.

The STA 449 ***F3*** *Jupiter*® comprises a robust system for high-quality TGA and DSC measurements. The thermobalance can measure samples up to 35 g (the measurement range is 35 grams, as well). The resolution of this low-drift balance system is 0.1 µg. The system can operate in a temperature range between -150°C and 2400°C using various interchangeable sensors and furnaces. Different possible pump systems as well as the optional mass flow control device allow measurements under welldefined atmospheres. A double furnace hoist and an automatic sample changer (ASC) are also optionally available and improve sample throughput to increase the efficiency of operation for this high-performance thermal analyzer.

The various TGA-DSC sensors offer true DSC performance over an unmatched temperature range (-150°C to 1750°C). TGA and TGA-DTA sensors can be used for the highest temperatures of up to 2400°C.

The robust system setup, user-friendly software and flexible design along with the wide range of different options make the system an ideal tool for quality control and research for material characterization in your laboratory.

For evolved gas analysis, the system can be coupled to QMS and FT-IR individually or to a combination of QMS and FT-IR – even if equipped with an automatic sample changer – and GC-MS or a combination of FT-IR and GC-MS.

STA 449 ***F3*** *Jupiter*® with two furnaces and double furnaces hoist

STA 449 ***F3*** *Jupiter*® – Hardware

Top-loading – the standard for balance systems

The STA 449 ***F3*** *Jupiter*® is a top-loading system using a balance design that has been standard for a long time for other types of scales – in laboratories and even in the kitchen at home or in a supermarket, most balances have been top-loading for decades. The reasons are simple: These systems combine ideal performance with easy handling. Why should your thermobalance be any different?

Stability, low drift and high sample loads

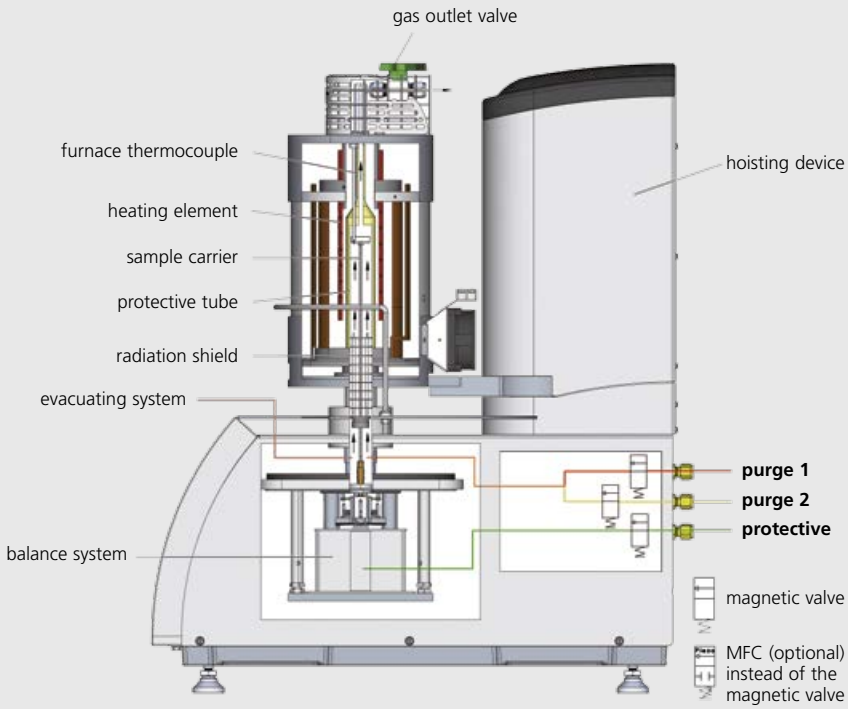
The balance system of the STA 449 ***F3*** *Jupiter*® offers high sample loads (up to 35 grams) and measurement range (35 grams) as well as high resolution (0.1 µg) and low drift (in the microgram range over hours). Another outstanding feature of the balance section of the STA is its high accuracy.

Vacuum-tight design – defined atmosphere conditions

The STA 449 ***F3*** *Jupiter*® is vacuum-tight by design. Several pump systems can be connected to the STA which allow evacuation down to 10^{-4} mbar and back-filling with well-defined atmospheres. The unique *OTS*® (oxygen trapping system) accessory can be used to reduce the oxygen partial pressure at the sample.

Various gas flow controllers

The gas flow is generally controlled by frits which are installed in the 3 gas flow channels (2 purge gases, 1 protective gas). Optionally available is a metal housed mass flow control system (MFC) for purge and protective gases offering optimum control of the atmosphere around the sample. Well defined gas flow conditions are crucial for an accurate interpretation of the measured effects, e.g. to differentiate between oxidation and pyrolysis reactions.



Furnace types		
Furnace	Temperature range	Cooling system
Silver	-120°C to 675°C	liquid nitrogen
Steel	-150°C to 1000°C	liquid nitrogen
Platinum	RT to 1500°C	forced air
Silicon carbide	RT to 1600°C	forced air
Rhodium	RT to 1650°C	forced air
Graphite	RT to 2000°C	tap or chilled water
Water vapor	RT to 1250°C	forced air
High-speed	RT to 1250°C	forced air
Tungsten	RT to 2400°C	tap or chilled water

Furnace and Furnace Hoists

Nine interchangeable furnaces are available to accommodate different application areas across the entire temperature range (-150°C to 2400°C). A double furnace hoist allows the simultaneous installation of two different furnaces for improved sample throughput or for low and high-temperature tests with the same instrument. The furnaces can easily be changed by the operator. Therefore, the system is adaptable to any future application range.

The high-speed furnace allows for high sample throughput for quality control as well as kinetic studies. Linear heating rates of up to 1000 K/min can be achieved at the sample.

Highest Precision – Maximum Flexibility

Various sensors

The STA 449 ***F3*** *Jupiter®* can be equipped with different sensor types. TGA sensors with slip-on plates or large crucibles (up to 5 ml) allow tests on large sample volumes and masses. TGA-DTA sensors can be used for applications such as routine tests or measurements on aggressive sample substances. The TGA-DSC and TGA-DSC-c_p sensors are used for most tests and allow quantitative DSC testing simultaneous to the TGA results. The c_p versions additionally allow determination of the specific heat with high accuracy. For special applications such as tests under corrosive atmospheres, the protected sensors can be employed. The *Fast-Fix* connection of the sensors to the instrument allows sensors to be changed within seconds. The system can therefore easily be adapted among the various required applications.

Automatic sample changer

An automatic sample changer for up to 20 samples is optionally available. The sample changer guarantees optimal crucible placement and maximum throughput. Preprogramming allows measurements to be carried out during the night or weekend. The software can automatically carry out analyses using predefined macros.

BeFlat® and *Tau-R® Mode*

Optionally available software features such as *BeFlat®* and *Tau-R® Mode* allow a fully automatic baseline correction as well as correction for system time constants. All routines are fully software based. This can be optimized for your specific measurement conditions. Furthermore, the raw data signal can be accessed at any time.

Examples for STA 449 sample carrier and sensor types

Sensors

Sensor thermocouple	Temperature range	Sensor types	Atmospheres
Type E	-150°C to 700°C (500°C*)	TGA, TGA-DTA, TGA-DSC (c_p)	inert, red., vac., oxid.
Type K	-150°C to 800°C (500°C*)	TGA, TGA-DTA, TGA-DSC (c_p)	inert, red., vac., oxid.
Type S	RT to 1650°C	TGA, TGA-DTA, TGA-DSC (c_p)	inert, oxid., red., vac.
Type P	-150°C to 1000°C	TGA-DSC, TGA-DSC (c_p)	inert, oxid., red., vac.
Type B	RT to 1750°C	TGA, TGA-DTA, TGA-DSC	inert, oxid., red., vac.
Type W	RT to 2400°C	TGA, TGA-DTA	inert, red., vac.
Type S protected	RT to 1650°C	TGA, TGA-DTA	inert, oxid., red., vac., corr.

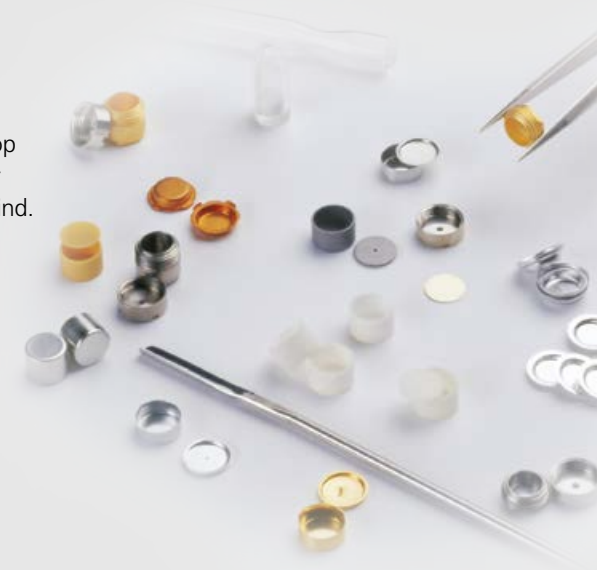
* under oxidizing conditions

Accessories

A wide range of crucibles (aluminum, silver, gold, copper, platinum, alumina, zirconia, graphite, stainless steel, etc.) is available for nearly all possible applications and materials.

For working in critical atmospheres, a "corrosive gas version" of the STA 449 ***F3*** *Jupiter*® can be supplied. This version is optimized for measurements under corrosive or reducing atmospheres. Gas flow control systems are prepared in a separate box and special sensors with protected thermocouple wires are available. For measurements on difficult samples or radioactive substances, the STA 449 ***F3*** *Jupiter*® can be prepared for installation in a glove box or hot cell. Electronics are removed from the measurement part and all cables and fittings are prepared for connection to an existing feedthrough.

If you have any other special application or test condition, ask us! Our engineers are prepared to develop special versions of instrumentation or software with your requirements in mind.



STA 449 ***F3*** *Jupiter*® with Automatic Sample Changer (ASC)

Proteus® Software for the STA 449 ***F3*** *Jupiter*®

The STA 449 ***F3*** *Jupiter*® runs under the versatile *Proteus*® software on a Windows® operating system. The *Proteus*® software includes everything you need to carry out a reliable measurement and evaluate the resulting data – or even carry out complicated analyses. The *Proteus*® software is licensed with the instrument and can also be installed on other computer systems.

General Software Characteristics:

- For Windows® XP and Windows® 7 operating systems
- Multi-tasking: simultaneous measurement and evaluation
- Multi-moduling: operation of different instruments with one computer
- Combined analysis: comparison and/or evaluation of STA, DSC, TGA, DIL, TMA and DMA measurements in one plot
- Labeling: input and free placement of text elements
- Calculation of 1st and 2nd derivative
- Selectable scaling
- Graphic and data export
- Selectable colors and line types
- Storage and restoration of analyses
- Macro recorder (optional)
- Context-sensitive help system
- Temperature calibration
- Compatible with advanced software packages (*Peak Separation*, *Thermokinetics*)
- Software produced by iso-certified company

DSC Features

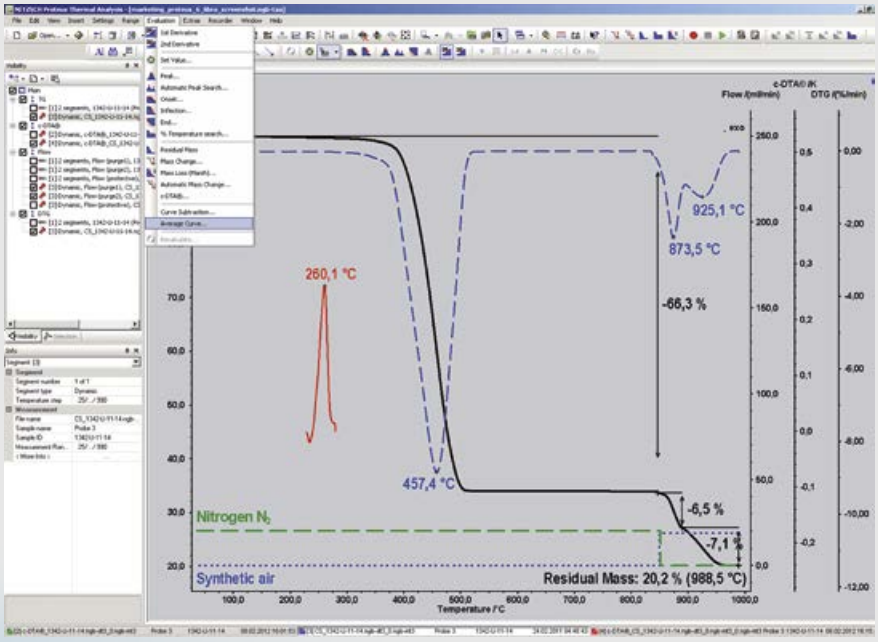
- Determination of onset, peak, inflection and end temperatures
- Automatic peak search
- Transformation enthalpies: analysis of peak areas (enthalpies) with selectable baseline and partial peak area analysis
- Comprehensive glass transition analysis
- Degree of crystallinity
- OIT (Oxidative-Induction Time) evaluation
- Specific heat determination (optional)
- *BeFlat*® for automatic baseline correction (optional)
- *Tau-R*® *Mode* (optional): evaluation of exo- and endo-thermal effects under consideration of system time constants and thermal resistance values
- *Purity Determination* (optional)

Advanced Software (options)

- *Peak Separation* Software: allows accurate separation and evaluation of overlapping transitions
- NETZSCH *Thermokinetics*: allows advanced characterization of reactions and kinetic parameters on the basis of multiple-step kinetic analysis on up to 16 curves, also provides predictions of the process

TGA Features

- Mass changes in % or mg
- Automatic evaluation of mass change steps
- Determination of the residual mass
- Extrapolated onset and endset
- Peak temperatures of the 1st and 2nd derivatives of the mass change curve
- Automatic baseline correction
- *c-DTA*® for the calculated DTA signal with evaluation of characteristic temperatures and peak area (optional for TGA measurements)
- *Super-Res*® for rate-controlled mass change (optional)



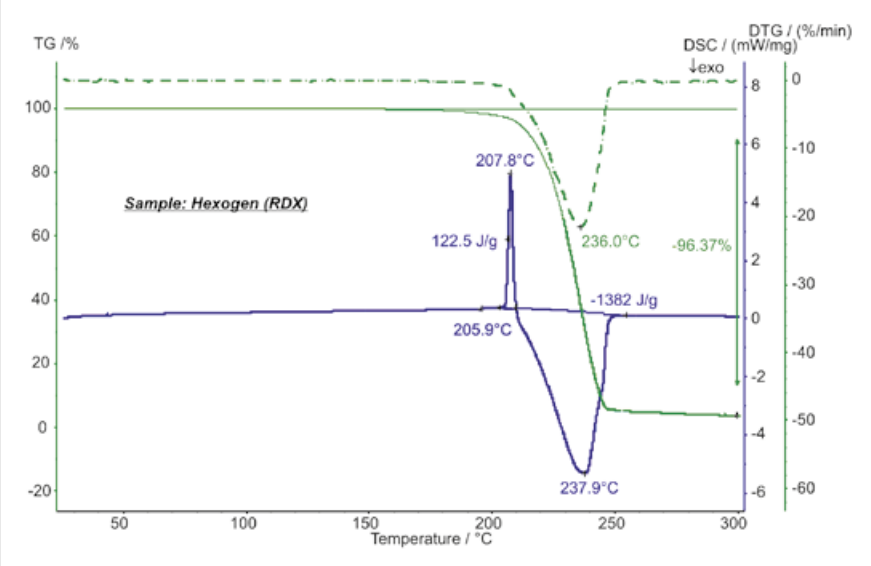
User interface during evaluation:
Graphical presentation of the TGA curve (black) together with the DTG curve (blue dashed line), the *c-DTA*® curve (red) and two curves representing the gas flow of nitrogen (green dashed line) and synthetic air (blue dotted line)

Meaningful Material Characterization in the Low-Temperature Fields

Application Examples

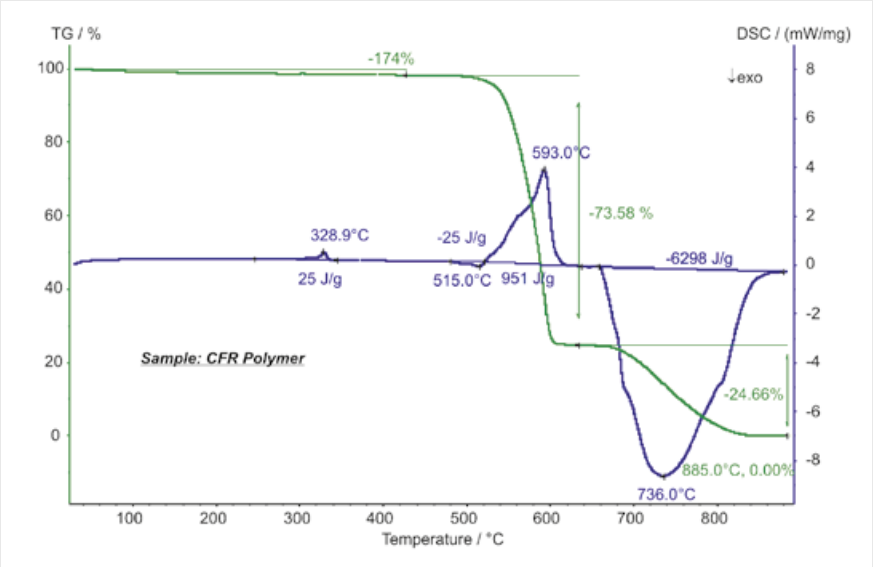
Characterization of Explosives

The highly explosive material hexogen (also called RDX, T4, etc.) starts to lose mass already at about 150°C, as can be seen from the TGA curve. The endothermic DSC peak at an onset temperature of 206°C with an enthalpy of 123 J/g is due to melting of the sample, followed by a strongly exothermic decomposition, releasing 1.38 kJ/g of energy. This experiment was carried out in a synthetic air atmosphere at a heating rate of 5 K/min using an initial sample mass of only 2.32 mg.



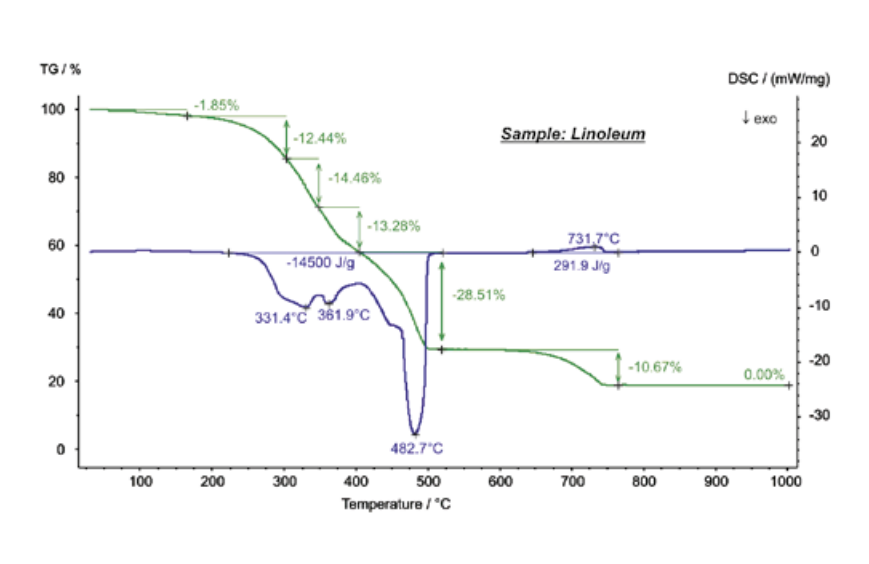
Analysis of Composite Materials

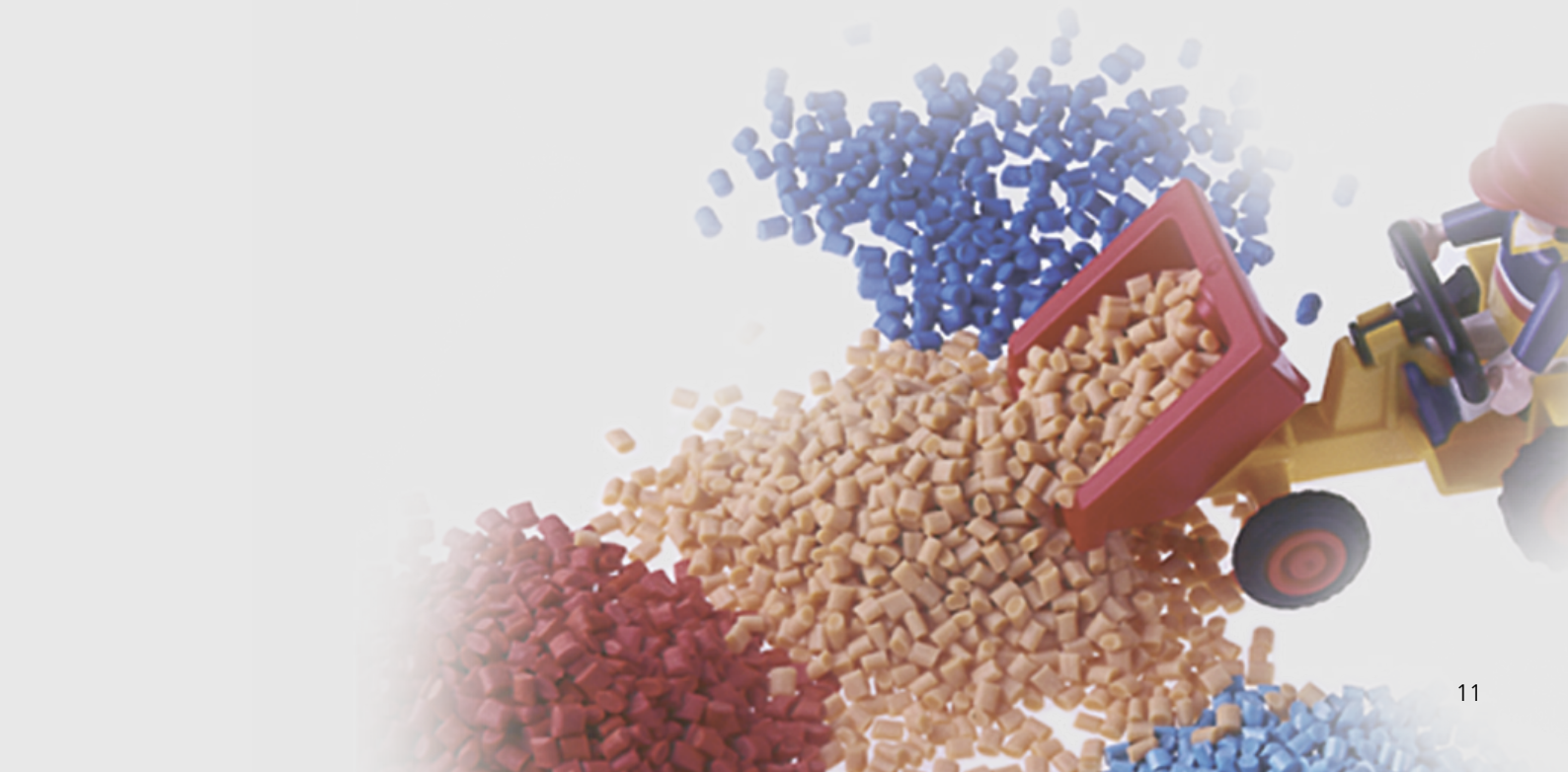
Carbon fiber-reinforced polymers (CFRP) are very popular composite materials which consist of a polymer matrix and embedded carbon fibers. CFRPs are very light, and yet they exhibit high stability and rigidity at the same time. They are thus very suitable for automotive, aircraft and space applications. This STA measurement shows an endothermic DSC peak with an enthalpy of 25 J/g at 329°C which is due to melting of the polymer matrix. Between approx. 480°C and 620°C, the pyrolytic decomposition of the polymer occurred. At 650°C, the purge gas was switched from N_2 to O_2, resulting in the strongly exothermic decomposition of the carbon fiber content (24.7%). The residual mass of 0.0% at the end of the experiment indicates that no further inorganic fillers or glass fibers were in the sample.



Burn-out of Linoleum

The building material linoleum was invented in 1863 and is most often used as floor covering. It is very robust and has an insulating effect even at small thicknesses. This STA measurement in air reflects the natural contents of linoleum: after the evaporation of humidity below 150°C, the stepwise, strongly exothermic burn-out of linseed oil, natural resins, cork flour, wood flour and the substrate jute followed between approx. 200°C and 500°C. The entire heat released during the oxidation was 14.5 kJ/g. Between 600°C and 750°C, the endothermic decomposition of the filler $CaCO_3$ (chalk) is observed. Above 750°C, the residual mass remains constant.



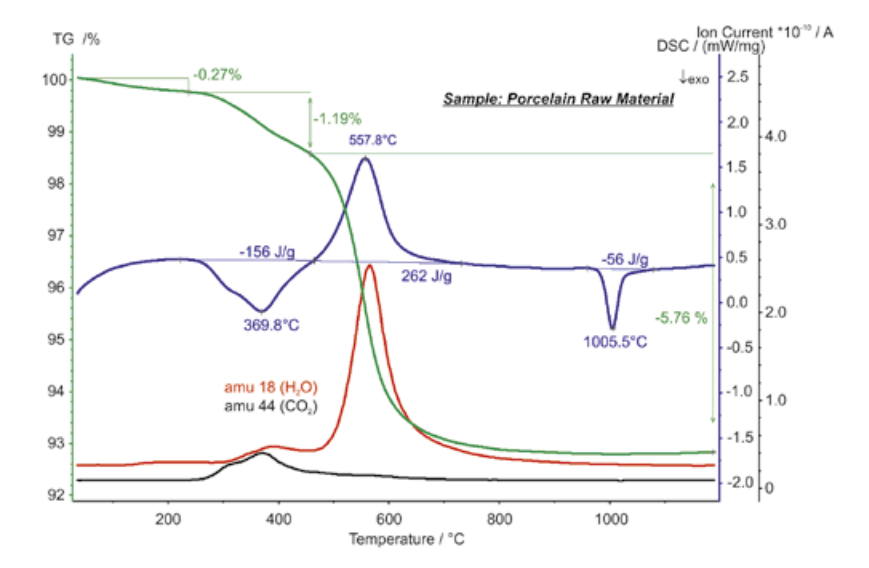


Meaningful Material Characterization in the High-Temperature Fields

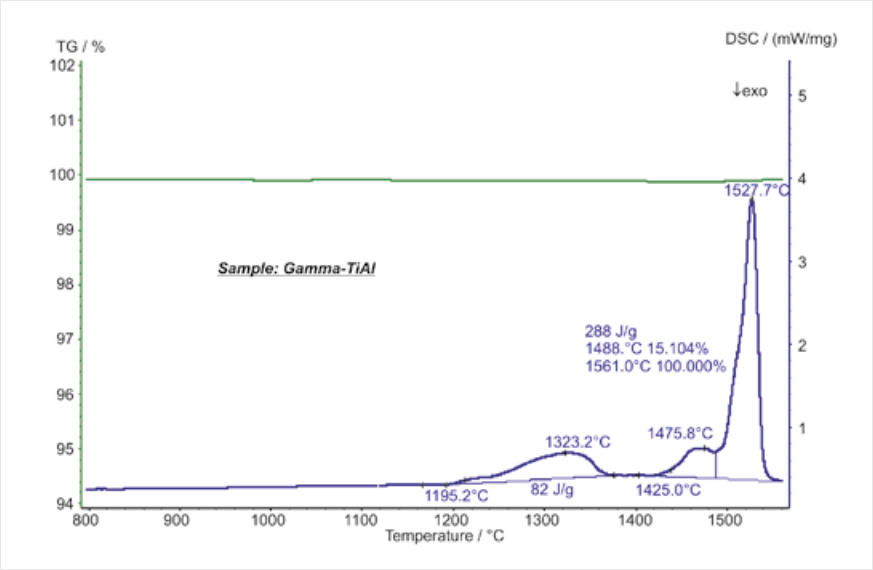
Application Examples

Characterization of Porcelain Raw Material

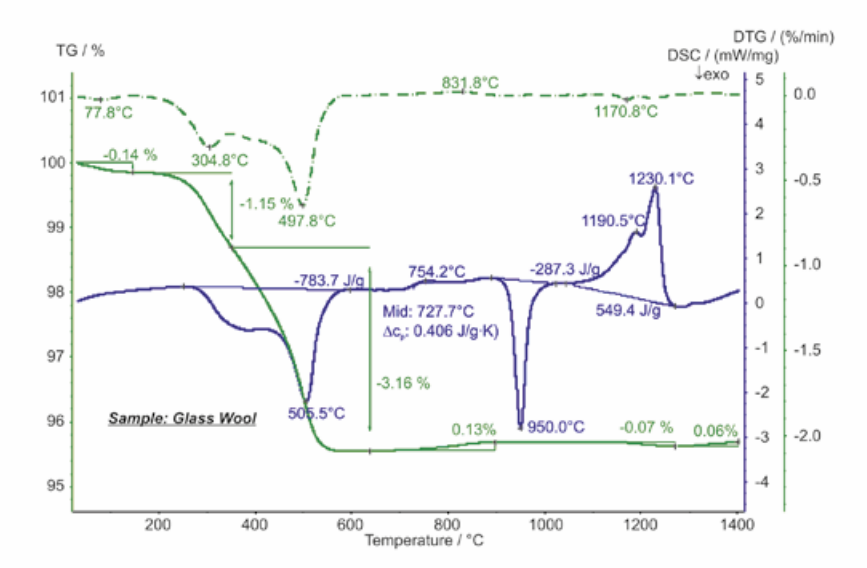
This STA-MS measurement on porcelain raw material shows three mass-loss steps. Below approx. 250°C, the evaporation of humidity occurred. At temperatures between 250°C and 450°C, the burn-out of organic binder was observed, during which 156 J/g of energy was released. The dehydration of kaolin occurred above 450°C and required 262 J/g. The mass spectrometer signals for mass numbers 18 and 44 reflect the corresponding release of H_2O and CO_2. The exothermic DSC peak at 1006°C with an enthalpy of -56 J/g is due to the mullite formation.



Phase Transitions of γ-TiAl



The refractory alloy γ-TiAl distinguishes itself through high temperature and corrosion resistance with a low specific weight. It is used, for instance, in turbo chargers, gas turbines, and engines as well as in aircraft and space applications. The DSC signal shows an endothermic effect (1323°C peak temperature) beginning at an extrapolated onset temperature of 1195°C; this is due to the structural $\alpha_2 \rightarrow \alpha$ transformation. At 1476°C (DSC peak temperature), the $\alpha \rightarrow \beta$ transformation occurred. The endothermic DSC peak at 1528°C is due to melting of the sample (onset at approx. 1490°C, liquidus temperature at about 1528°C). No significant mass changes were detected during the experiment.



Building Material: Glass Wool

Glass wool is often used for the insulation of houses and heating pipes. The measurement shows three mass-loss steps below approx. 600°C, which are due to the evaporation of humidity and the burn-out of organic binder. The latter can be seen from the strongly exothermic DSC signal in this temperature range. The step in the DSC signal at 728°C is due to the glass transition (increase in the specific heat of 0.41 J/[g·K]). The exothermic DSC peak at 950°C with an enthalpy of -287 J/g is due to crystallization; the endothermic effects between approx. 1050°C and 1250°C with an entire enthalpy of 549 J/g are due to melting. The slight mass changes above 700°C are most probably due to oxidation and evaporation of impurities.

Expertise in Service

Our Expertise – Service

All over the world, the name NETZSCH stands for comprehensive support and expert, reliable service, before and after sale. Our qualified personnel from the technical service and application departments are always available for consultation.

In special training programs tailored for you and your employees, you will learn to tap the full potential of your instrument.

To maintain and protect your investment, you will be accompanied by our experienced service team over the entire life span of your instrument.

Summary of Our Services

- Installation and commissioning
- Hotline service
- Preventive maintenance
- Calibration service
- IQ / OQ / PQ
- On-site repairs with emergency service for NETZSCH components
- Moving / exchange service
- Technical information service
- Spare parts assistance

Our Expertise – Applications Laboratories

The NETZSCH Thermal Analysis applications laboratories are a proficient partner for nearly any Thermal Analysis issue. Our involvement in your projects begins with proper sample preparation and continues through meticulous examination and interpretation of the measurement results. Our diverse methods and over 30 different state-of-the-art measuring stations will provide ready-made solutions for all your thermal needs.

Within the realm of thermal analysis and the measurement of thermo-physical properties, we offer you a comprehensive line of the most diverse analysis techniques for materials characterization (solids, powders and liquids).

Measurements can be carried out on samples of the most varied of geometries and configurations. You will receive high-precision measurement results and valuable interpretations from us in the shortest possible time. This will enable you to precisely characterize new materials and components before actual deployment, minimize risks of failure, and gain decisive advantages over your competitors.

For production problems, we can work with you to analyze concerns and develop solutions. The minimal investment in our testing and services will reward you with reduced down time and reject rates, helping you optimize your processes across the board.

NETZSCH

The NETZSCH Group is a mid-sized, family-owned German company engaging in the manufacture of machinery and instrumentation with worldwide production, sales, and service branches.

The three Business Units – Analyzing & Testing, Grinding & Dispersing and Pumps & Systems – provide tailored solutions for highest-level needs. Over 3,000 employees at 163 sales and production centers in 28 countries across the globe guarantee that expert service is never far from our customers.

When it comes to Thermal Analysis, Calorimetry (adiabatic & reaction) and the determination of Thermophysical Properties, NETZSCH has it covered. Our 50 years of applications experience, broad state-of-the-art product line and comprehensive service offerings ensure that our solutions will not only meet your every requirement but also exceed your every expectation.

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