ECO-LINE

Simultaneous Thermal Analyzer –
STA 449 **F3** *Jupiter*®

Analyzing & Testing

Flexible, Sophisticated & Technically Outstanding

Simultaneous Thermal Analysis generally refers to the simultaneous application of Thermogravimetry (TGA) and Differential Scanning Calorimetry (DSC) to one and the same sample in a single 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, sample throughput is improved as more information can be gathered from each test run.

DSC Possibilities

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

TGA Possibilities

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

Standard*	Description
ISO 11358	Plastics – Thermogravimetry (TG) of Polymers
ASTM E793	Standard Test Method for Enthalpies of Fusion and Crystallization by Differential Scanning Calorimetry
DIN 51004	Thermal Analysis; Determination of Melting Temperatures of Crystalline Materials by Differential Thermal Analysis
DIN 51006	Thermal analysis (TA); Thermogravimetry (TG); Principles
DIN 51007	Thermal Analysis; Differential Thermal Analysis; Principles

* Depending on instrument setup

The NETZSCH STA Eco-Line

70% LESS ENERGY AND COST.
NO EXTERNAL TEMPERATURE CONTROL NEEDED.

To obtain exact Thermogravimetric results with low drift behavior, most manufacturers have to resort to thermostatic control using a water cycle. Having to run the thermostat continuously requires a lot of energy and produces waste heat, which subsequently needs to be regulated by air conditioning.

NETZSCH has succeeded in removing the external thermostat. The weighing chamber's temperature is now regulated electronically, while maintaining excellent temperature stability. By removing the thermostat, the energy consumption of an STA 449 **F3** *Jupiter®* will decrease by 70% for an average user.*

* When using the instrument 3 times a day on 250 days a year

Further advantages of the STA Eco-line are:

- 30% less waste heat
- Saves space
- Less maintenance
- Best performance

World-Leading Flexibility for Your Applications

The STA 449 **F3** *Jupiter*® combines a high-performance Heat-Flux DSC with a submicrogram-resolution thermobalance, thereby offering an unmatched sample load and measurement range. The Simultaneous Thermal Analyzer can easily be adjusted to nearly any possible application by selecting the optimum furnace, installing the most appropriate sensor and using the proper accessories.

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.

Setting New Benchmarks through Experience & Innovation

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 in 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.

Defined Atmosphere Conditions – Vacuum-Tight Design

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.

– 50 YEARS –
Leading Manufacturer of
High-Performance
Thermal Analysis Systems

STA 449 **F3** *Jupiter*®

Trend-Setting Technology

* Vacuum depends on the selected evacuation system

))) Select the Appropriate

Ten 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.

Day-to-Day Work Done Safely

For standard STA measurements, the silicon carbide furnace (SiC) is the robust workhorse in your laboratory, operating from ambient temperature to 1600°C. For measurements under corrosive atmosphere, the SiC furnace can be equipped with a protected TGA-DTA sensor, guaranteeing instrument-safe conditions.

Measurements in the Lower Temperature Range

The silver and steel furnaces allow for measurements in the subambient temperature range by using devices for controlled cooling. Whereas the silver furnace is ideally suited for the determination of the specific heat capacity, the steel furnace offers a broad temperature range from -150°C to 1000°C.

Specific Heat Capacity at Higher Temperatures

The platinum and the rhodium furnaces in combination with dedicated DSC sensors are specifically suited for determination of the specific heat capacity in the higher temperature range.

Your Results Achieved at the Highest Speed

The high-speed furnace allows for the simulation of realistic heating processes with linear heating rates up to 1000 K/min. Additionally, the high heating rates are useful when implementing kinetic studies.

STA 449 F3 *Jupiter*

Furnace
for Your Application!

Highest Temperatures

The tungsten heating element allows for measurements under helium atmosphere from room temperature to 2400°C.

Measurements in Humid Atmospheres

The copper furnace is ideal for measurements under relative humidity between room temperature and 100°C. For this purpose, a humidity generator is available which offers a maximum dewpoint of 80°C corresponding to 47% molar concentration. In addition, the copper furnace can be used for conventional STA measurements including determination of the specific heat capacity up to 500°C.

The water-vapor furnace covers the broad temperature range from room temperature to 1250°C. The furnace can be connected to the aforementioned humidity generator, or to a vapor generator which produces steam by evaporating water. A molar concentration of up to 100% can be achieved.

Furnace type	Temperature range	Cooling system
Silver	-120°C to 675°C	liquid nitrogen*
Copper	-150°C to 500°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

* Alternative vortex cooling allows for start temperatures around 0°C.

The Right Sensor for Your Demands

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. For special applications such as tests under corrosive atmospheres, the protected sensors can be employed. The TGA-DSC and TGA-DSC-c_p sensors are used for most tests and allow quantitative DSC testing simultaneously to the TGA results. The c_p versions additionally allow determination of the specific heat capacity with high accuracy.

The *Quick-Connect* connection of the sensors to the instrument allows sensors to be changed in a matter of seconds. This allows the sytem to be easily be adapted to any of the varous potential applications.

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

* in oxid. atmosphere up to 500 °C

Standard Type S Sensors – Workhorse and Specialty

In the high-temperature range, type S sensors combine a broad temperature range from room temperature to 1650°C with high sensitivity. For measurements in the presence of corrosive gases, the TGA-DTA sensor with protected thermocouples provides safe conditions without adversely affecting the sensitivity.

High Sensitivity in the Lower Temperature Range

The type P sensors are standard in the lower temperature range; they are ideally suited for the steel furnace. All sensors equipped with thermocouple E or K are characterized by the highest level of sensitivity and resolution. They are particularly well suited for detecting small effects.

High and Highest Temperature Range

True DSC measurements up to 1750°C can be monitored by using the type B sensor. At the highest temperatures up to 2400°C, the type W sensor for TGA and TGA-DTA can be used under inert, reducing, and vacuum conditions.

HIGHEST PRECISION
Maximum Flexibility

Labels for diagram:
gas outlet valve
furnace thermocouple
heating element
sample carrier
protective tube
radiation shield
evacuating system
balance system
hoisting device
MFC — purge 1
MFC — purge 2
MFC — protective
gas supply unit
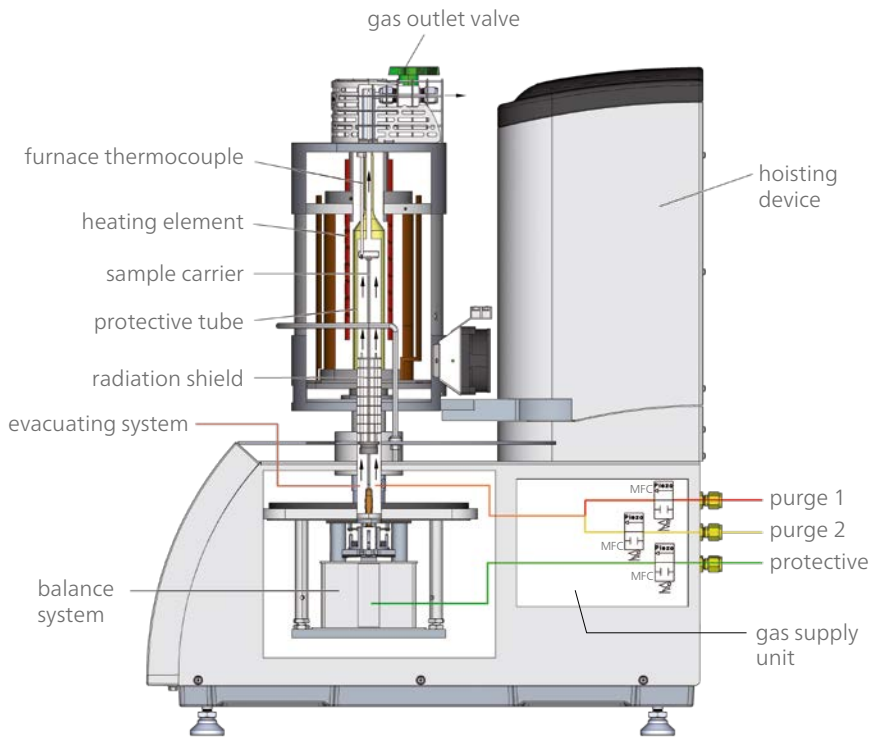
Gas Flow Control

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 accurate interpretation of the measured effects, e.g., to differentiate between oxidation and pyrolysis reactions.

Coupling to Evolved Gas Analysis

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.
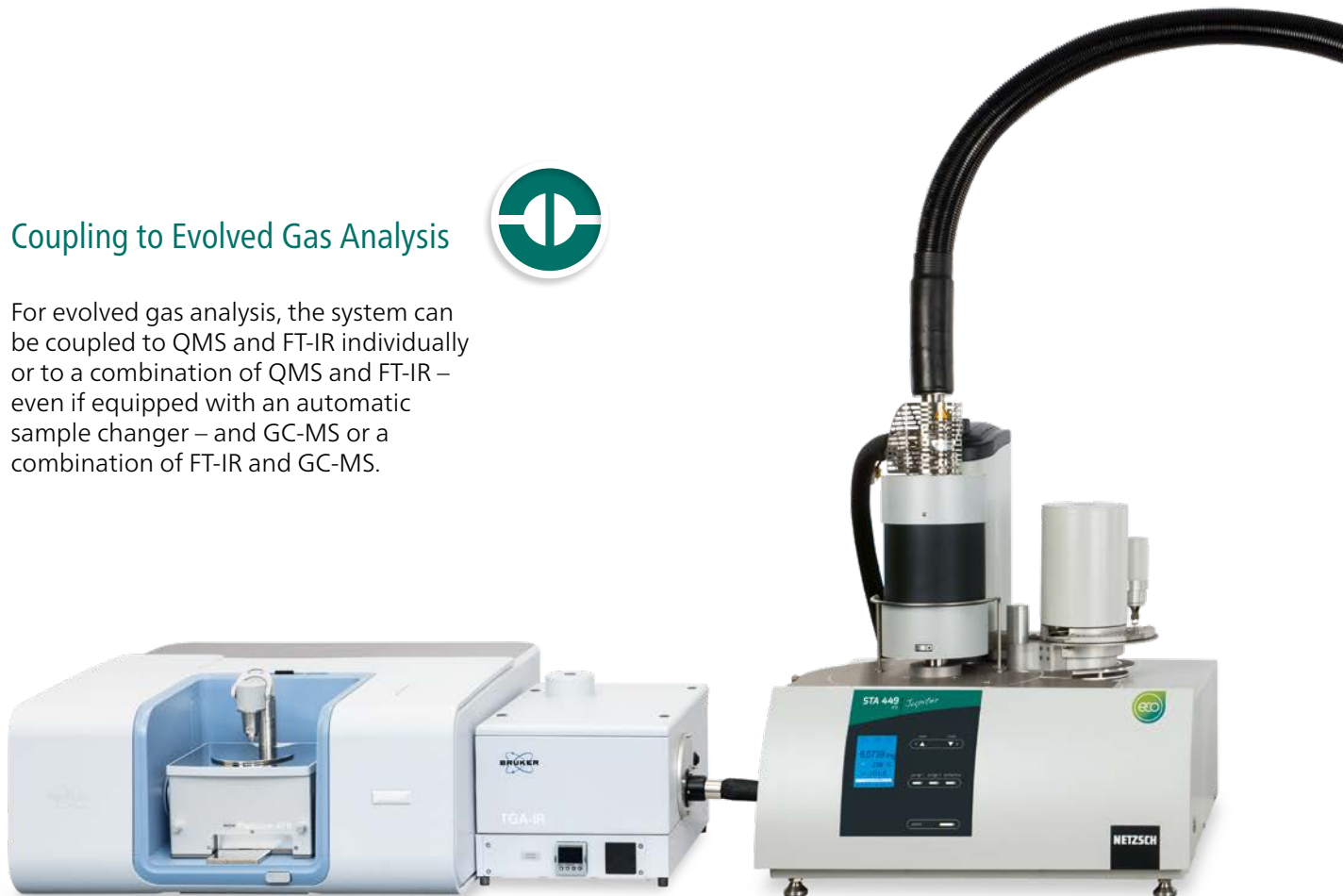
FT-IR Bruker Invenio ATR coupled to STA 449 *F3* *Jupiter*® with automatic sample changer and QMS 403 *Aëolos Quadro*

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.
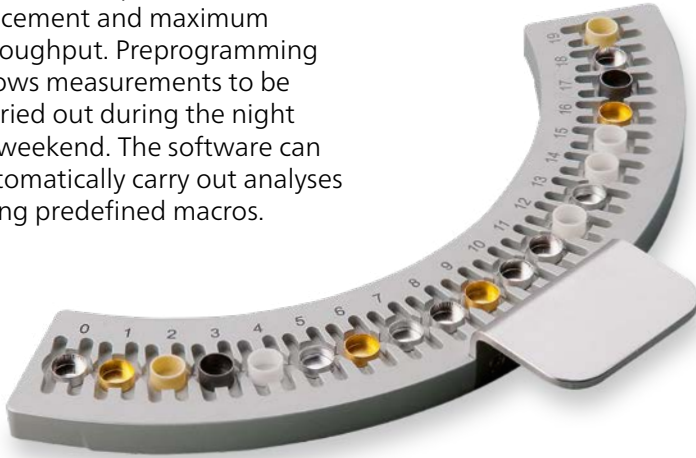
Accessories

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 atmospheres such as reducing.

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.

Robust and Easy-to-Operate

Proteus® software is produced by an ISO-certified company and includes everything you need to evaluate the resulting data, and even perform complicated analyses.

STA 449 *F3* *Jupiter*® with *Proteus*® 8.0

OUR POWERFUL ANALYTICAL SOFTWARE

BeFlat – An Intelligent Way to Save Time

The TGA-*BeFlat* software feature keeps a record of the temperature as a function of the measuring influences – including the heating rate, the different purge gases and the gas flow rates – and can therefore provide the appropriate correction for the selected measurement conditions without having to carry out a blank value determination in the form of a correction measurement*.

AutoEvaluation – The World's Only Truly Self-Acting Evaluation

The unique *AutoEvaluation* function detects and evaluates thermal effects – i.e., peaks, glass transitions or mass changes – without user intervention. Intelligent algorithms are capable of handling DSC and TGA curves fully automatically. This generates completely objective test results.

Not only is this tool helpful for beginners, but experienced users can also use the results as a "second opinion". The operator has full control at all times. Values can be recalculated or further manual evaluations added.

*Identify*** – One Click Results

Identify marks a real turning point in the field of thermal analysis. This optional software package allows for the identification and classification of materials via database comparison with just one click.

In the case of DSC and TGA, the curve comparison is effect-based, which ensures fast and efficient processing. The result is a similarity hit list.

Besides one-on-one comparisons with individual curves or literature data, it is also possible to check whether a particular curve belongs to a certain class.

The database (more than 2000 entries, 1200 of which are already incuded in *Identify*) is open for adding users' own libraries and classes; it can be easily expanded with experiments and knowledge of their own.

* depending on the instrument combination
** optional

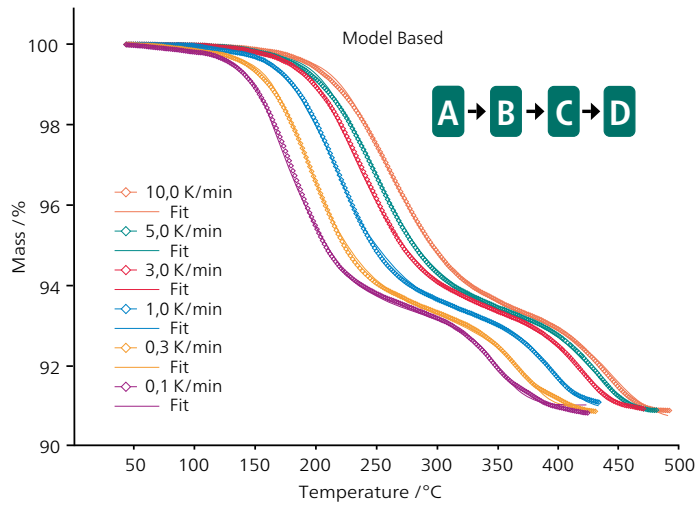# SIMULATIVE OPTIMIZATION OF DEBINDING

KINETICS NEO

Fig. 1: Experimental TGA data (symbols) are in good agreement with the results of the simulation (solid lines) according to a 3-step kinetic model for heating rates at 0.1, 0.3, 1, 5 and 10 K/min.

Optimization of the Burn-Out Process for a Polymer Binder

In sinter metallurgy, a polymeric binder is added to a metal powder to improve adhesion. During the sintering process, the binder is carefully removed to prevent micro cracks caused by the release of gases. Slow heating results in time loss during production, while fast heating results in quality loss due to intensive gas development during polymer decomposition.

With these conditions in mind, the objective here is to find the optimum temperature program for a tunnel kiln. The production process is simulated by six TGA measurements (Fig. 1) conducted at different heating rates and a kinetic model based on them. In this example, a mass loss rate of 0.05%/min yields optimal material quality. Under laboratory conditions, the temperature program in Fig. 2 achieves this. Fig. 3 shows the optimum temperature curve for the different zones in the tunnel kiln.

We also offer "Kinetics as a Service". For more information, please refer to www.kinetics.netzsch.com
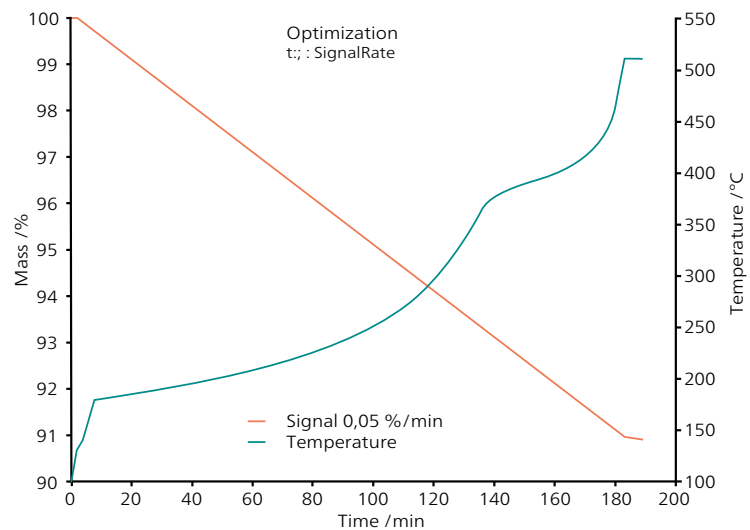

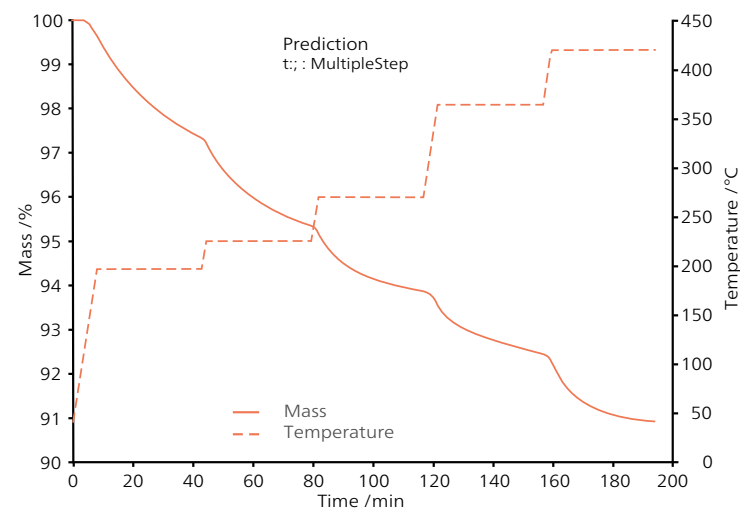Fig. 2: Optimized temperature program for the burn-out of the polymer binder under laboratory conditions

Fig. 3: Optimized zone temperatures for the burn-out of the polymer binder in the tunnel kiln during the production process

*Meaningful Material
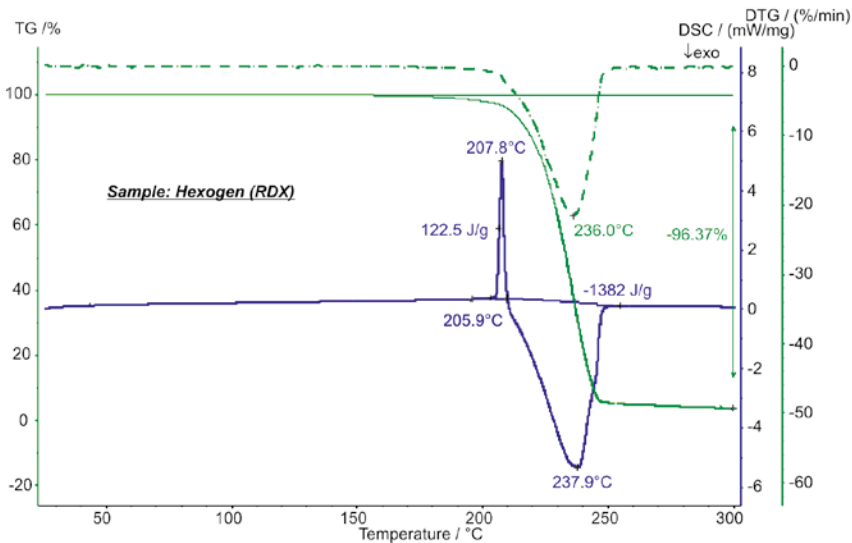Characterization*

LOW-TEMPERATURE

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.
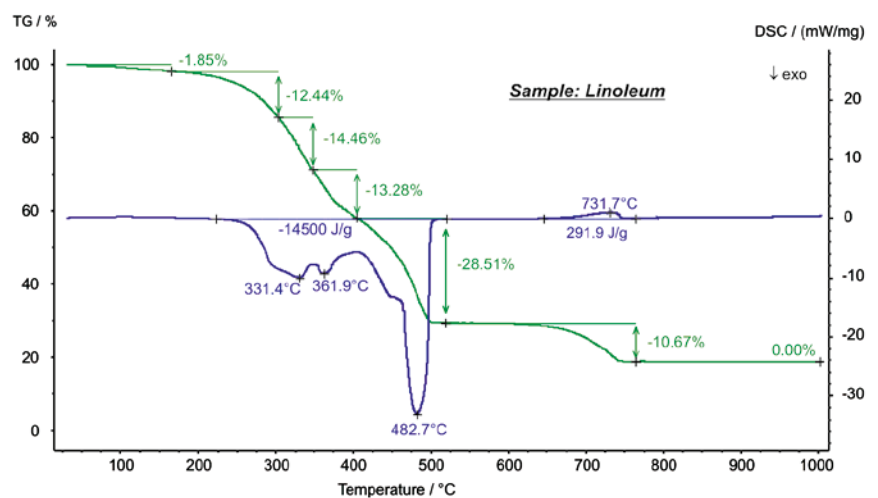
The measurement was carried out in the SiC furnace on a linoleum sample (5.52 mg) at a heating rate of 10 K/min in air atmosphere.

The hexogen measurement was made in the SiC furnace at a heating rate of 5 K/min in air atmosphere.

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.
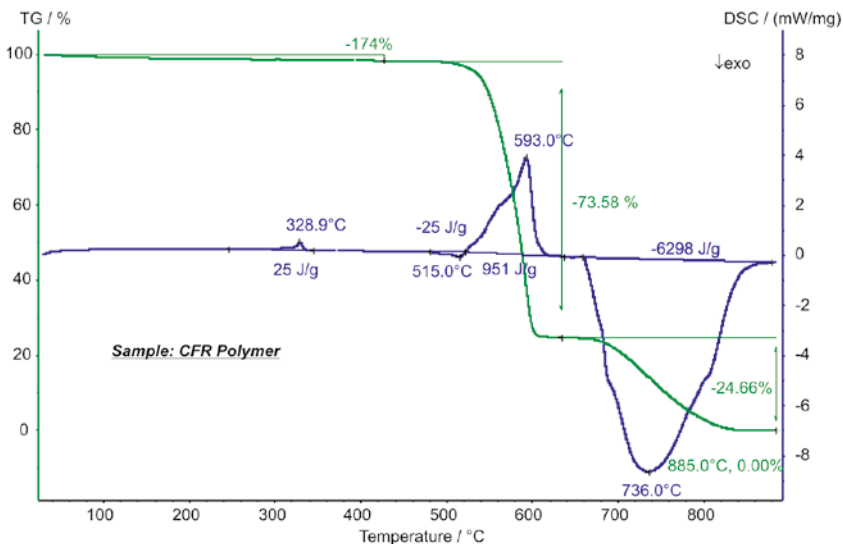
For the measurement on CFRP (7.62 mg) in the SiC furnace, the atmosphere was switched from nitrogen to oxygen at 650°C; heating rate: 10 K/min.

Analysis of Composite Materials

Carbon fiber-reinforced polymers (CFRP) are very popular composite materials which consist of a polymer matrix and embedded carbon fibers. They are 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.

Meaningful Material Characterization
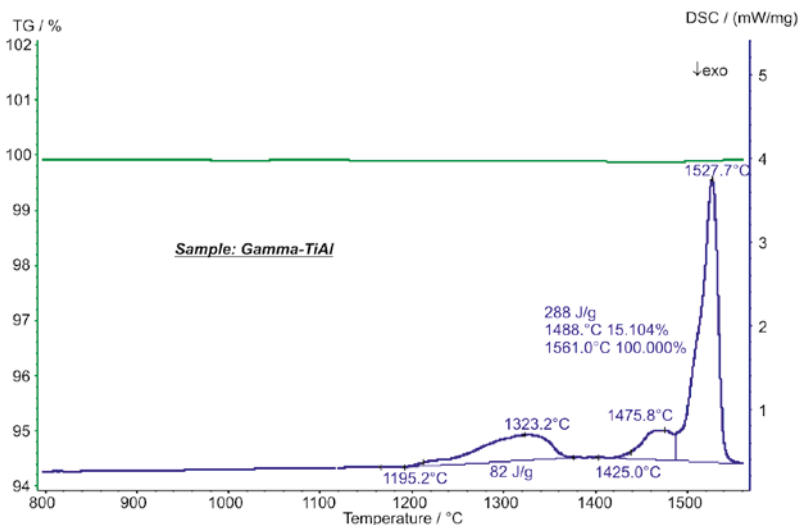# HIGH-TEMPERATURE

Characterization of Porcelain Raw Material
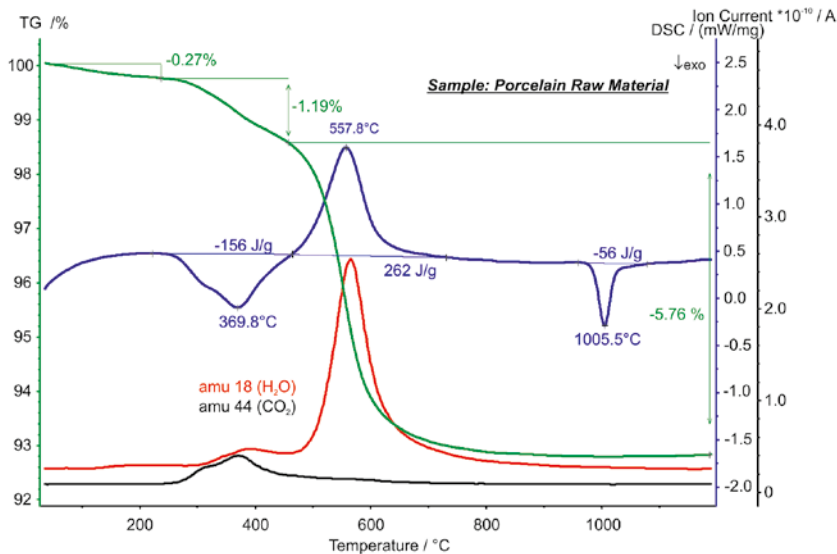
The porcelaine material (37.14 mg) was measured in the SiC furnace in air atmosphere at a heating rate of 20 K/min.

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 phase transitions of this metal alloy (32.08 mg) was recorded in the rhodium furnace at a heating rate of 20 K/min to 1600°C in argon atmosphere.

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 and gas turbines. 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 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.
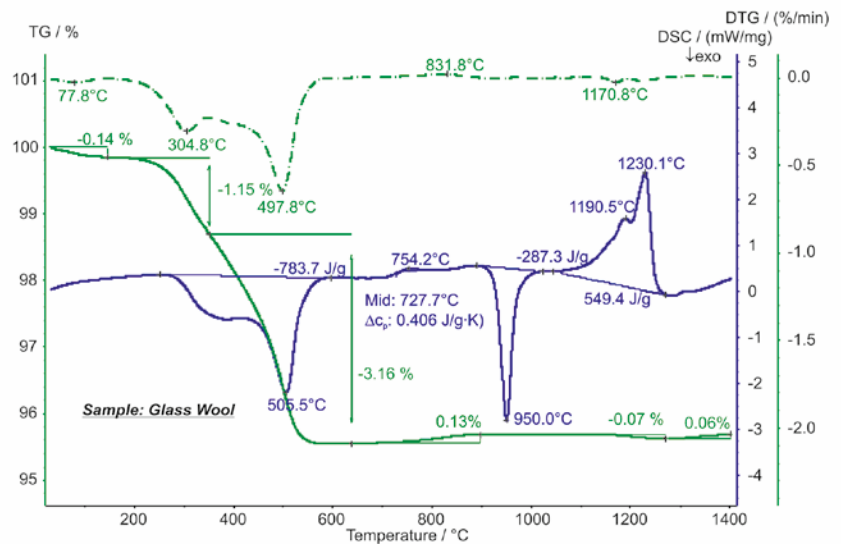
The decomposition of the building material (49.71 mg) was measured in the SiC furnace at a heating rate of 20 K/min in air atmosphere.

Technical Specifications

STA 449 **F3** *Jupiter*®	
Design	Top-loading
Temperature range	-150°C to 2400°C
Furnace	Variety of furnaces incl. high-speed, water-vapor, low to highest temperature, e.g., silver, platinum, tungsten, etc.
Motorized furnace hoist	Double hoist for two furnaces or one furnace + automatic sample changer
Heating rate	▪ 0.001 to 50 K/min (furnace-dependent) ▪ High-speed furnace: up to 1000 K/min
Sensors	TGA, TGA-DTA, TGA-DSC, TGA-DSCc_p, special sensors for hanging samples. Sensors can be changed out easily in a matter of moments
Vacuum-tight	10^{-4} mbar[1]
AutoVac	Option for software-controlled automatic evacuation
Evacuation system	Options for one and two furnaces; manual or software-controlled operation
Atmospheres	Inert, oxidizing, static, dynamic, vacuum
Oxygen trap system (*OTS*®)	Optional
Automatic sample changer (ASC)	20 crucible positions (optional)
Gas flow control	Integrated frits (optional 3 mass flow controllers)
Temperature resolution	0.001 K
Balance resolution	0.1 µg (over the entire weighing range)
Balance drift	< 5 µg/hour
Maximum sample load	35000 mg (incl. crucible), corresponds to TGA measuring range
Sample volume (max.)	▪ TGA: up to 5 ml ▪ DSC: 0.19 ml ▪ DTA: 0.9 ml
DSC enthalpy accuracy	± 2% (for most materials)
Evolved gas analysis	QMS, GC-MS and/or FT-IR couplings, *PulseTA*® (options)
Optional instrument specialties	▪ Glove box version ▪ Corrosion-resistant version

1 Actual achievable vacuum depends on the selected evacuation system

STA 449 **F3** *Jupiter*®	
Operating systems	Windows operating systems
General software features	▪ Multi-tasking: simultaneous measurement and evaluation ▪ Multi-moduling: operation of different instruments from one computer ▪ Combined analysis: comparison and/or evaluation of STA, DSC, TGA, DIL, TMA and DMA measurements in one plot ▪ Selectable scaling ▪ Graphic and data export ▪ Calculation of 1st and 2nd derivative including peak temperatures ▪ Storage and restoration of analyses ▪ Context-sensitive help system ▪ Report generator ▪ *AutoEvaluation*
DSC-specific features	▪ Determination of onset, peak, inflection and end temperatures, incl. automatic peak search ▪ Analysis of exothermal and endothermal peak areas (enthalpies) with selectable baseline and partial peak area analysis ▪ DSC-integral curve ▪ Comprehensive glass transition analysis ▪ Degree of crystallinity ▪ OIT (Oxidative-Induction Time) ▪ DSC-*BeFlat* ▪ Specific heat capacity ▪ *Tau-R*®
TGA-specific features	▪ Mass changes in % or mg ▪ Automatic evaluation of mass-change steps including determination of residual mass ▪ Extrapolated onset and endset ▪ Automatic TGA-*BeFlat* baseline correction for automatic correction of measuring influences ▪ c-*DTA* for calculation of the DTA signal with evaluation of characteristic temperatures and peak area, optional for TGA measurements
Optional software	Features ▪ *Super-Res* for rate-controlled mass change ▪ Specific heat capacity ▪ DSC-*BeFlat* ▪ *Tau-R*® ▪ c-*DTA* Packages ▪ Purity ▪ *Peak Separation* ▪ Kinetics Neo ▪ *Identify*

Software Features

The NETZSCH Group is an owner-managed, international technology company with headquarters in Germany. The Business Units Analyzing & Testing, Grinding & Dispersing and Pumps & Systems represent customized solutions at the highest level. More than 3,700 employees in 36 countries and a worldwide sales and service network ensure customer proximity and competent service.

Our performance standards are high. We promise our customers Proven Excellence – exceptional performance in everything we do, proven time and again since 1873.

When it comes to Thermal Analysis, Calorimetry (adiabatic & reaction), the determination of Thermophysical Properties, Rheology and Fire Testing, 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.

Proven Excellence.

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