ONset NETZSCH CUSTOMER MAGAZINE

Edition 29 | January 2025

Unlocking Potential

Discover our New Products for Enhanced Flexibility in the Determination of Thermal Properties!

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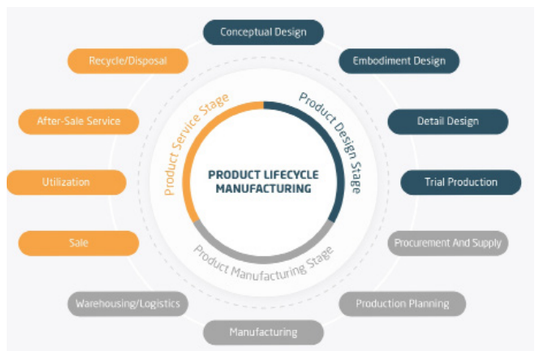

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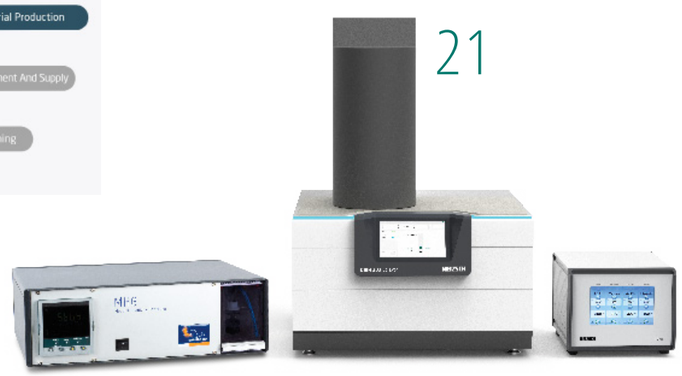

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Editorial

Dear Reader:

These are challenging times. There are crises in many areas and uncertainty is growing. However, you can always rely on one constant: NETZSCH is and will remain your reliable partner for thermal analysis, rheology and combustion testing – true to our motto of "Proven Excellence".

In this issue of our customer magazine, we are proud to present several new products and useful accessories. First and foremost is our high-temperature series which, following the launch of the STA 509 *Jupiter*® last summer, has now been expanded to also include the DSC 500 *Pegasus*®. The flexibility of these instruments makes them ideal for both routine and demanding applications. Just think about working in corrosive gas atmospheres or in glove boxes. Even hydrogen can be used as a gas atmosphere with our TÜV*-certified *H_2Secure* solution.

Another innovation involves light flash analyzers for the determination of thermal diffusivity (and hence also for thermal conductivity and the specific heat capacity of materials). The new LFA 717 *HyperFlash*® is available – depending upon the temperature range required – in two versions: the standard LFA 717 *HyperFlash*® (-100°C to 500°C) and the LFA 717 *HyperFlash*® *HT* (RT to 1250°C). These models both combine state-of-the-art hardware with unique, innovative software solutions – such as a calculation model for orthotropic materials, which exhibit different properties in three directions perpendicular to each other.

Two parameters that can also have a decisive influence on material behavior or the course of chemical reactions are humidity and pressure. Some plastics, for example, are sensitive to moisture, with the absorbed water acting as a plasticizer. By coupling the DMA 303 *Eplexor*® with the new Modular Humidity Generator (MHG), it is now possible to test the mechanical properties of plastics (among other materials) under defined humidity conditions.

Other material behavior – such as the rate of oxidation reactions, for example – is related to the partial oxygen pressure; the higher the oxygen content, the faster the reaction. In order to be able to evaluate even reactions such as these kinetically (pressure as ambient pressure or as the concentration of a gas component in a mixture), our Kinetics Neo software has recently been upgraded with the ability to create models that are a function not only of temperature but also of pressure. Read more about this on page 18.

In our *CUSTOMERS FOR CUSTOMERS* section, CeramTec, a leading manufacturer of advanced ceramics, describes how instruments for thermal analysis and for the determination of thermophysical properties are used.

Last but not least, Dr. Marc Egelhofer of LabV takes us into the exciting world of material intelligence platforms and explains how they can be used to support data-driven decisions at every stage of a product's life cycle.

I hope you enjoy reading this issue of **on**set.

Dr. Gabriele Kaiser
Standardization, Data Management & Life Sciences Consultant

*TÜV = German Technical Inspectrion Authority

STA 509 & DSC 500

High-Temperature Thermal Analysis with Our New STA and DSC Product Lines

Dr. Michael Schöneich, Product Line Manager

Fig. 1. STA 509 *Jupiter*®

The latest developments in NETZSCH's high-temperature product portfolio, the STA 509 *Jupiter*® and DSC 500 *Pegasus*® systems, represent a significant advancement in the field. These innovative solutions are designed to address the inherent challenges associated with high-temperature applications, offering users enhanced versatility and precision.

Introduction

Thermal analysis methods such as Thermogravimetry (TGA) and Differential Scanning Calorimetry (DSC) are essential tools in materials science, providing key insights into material properties and behavior. Thermogravimetric analysis focuses on detecting changes in a material's mass as a function of temperature or time, revealing processes like decomposition, oxidation, or evaporation. DSC, on the other hand, measures heat flow to uncover phase transitions, crystallization, and other thermal phenomena. Taken together, these methods provide a comprehensive understanding of the properties of a material under varying thermal conditions.

Nevertheless, the analysis of materials at elevated temperatures presents a distinctive set of challenges, including the management of material interactions, the control of atmospheric conditions, and the accurate capture of rapid kinetic reactions, all while maintaining precision in measurements. Addressing these challenges requires advanced instruments capable of handling extreme conditions with accuracy and reliability.

STA 509 *Jupiter*® – The Versatile Solution

The STA 509 *Jupiter*® integrates Thermogravimetry (TGA) and Differential Scanning Calorimetry (DSC) into a single measurement setup. This combination allows for simultaneous observation of mass changes and heat flow, which is particularly beneficial for studying complex thermal processes such as decomposition, oxidation, and phase transitions. The instrument's broad temperature range (-150°C up to 2400°C) and advanced interchangeable sensor design enable measurements, even in challenging experimental conditions (e.g., extreme temperatures, atmospheric conditions, or sample shapes and sizes).

The Benefit of Dedicated High-Temperature DSC

Precision and reproducibility are important considerations in the desgn of any analytical device, and are the main reasons for the existence of our NETZSCH DSC 500 *Pegasus*®. Unlike Simultaneous Thermal Analysis (STA) systems, which have a moveable sensor arrangement due to the balance incorporation, the DSC 500 features a fixed sensor setup that can be adjusted with micrometer precision (see Figure 2). This design minimizes variability between measurements, offering the highest level of reproducibility and precision. Such levels of performance are of crucial importance when executing complex analyses, such as the determination of specific heat capacity at elevated temperatures.

Fig. 2. Micrometer-precise adjustment of the sample carrier position

The Importance of Atmospheric Conditions

Achieving precise results in thermal analysis requires more than simply precision and reproducibility, particularly at elevated temperatures. It is also essential to exercise rigorous control over the surrounding atmosphere to maintain optimal conditions for the analysis. Any unintended reactions – such as oxidation – can compromise the integrity of the measurements. Our high-temperature product line is designed to prevent these issues by offering advanced features like vacuum tightness, gas flow control, special corrosive or hydrogen setups that control the atmosphere within the instrument, thus allowing for reliable results to be obtained even with sensitive materials.

One of these features is our Oxygen Trap System (OTS) (see Figure 3). A special oxygen getter here enables the elimination of even the minutest traces of oxygen from the atmosphere, thereby establishing ultra-pure conditions within the system. This renders the STA 509 and the DSC 500 as optimal choices for the analysis of samples such as highly reactive titanium alloys.

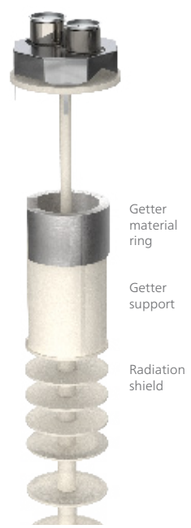

Fig. 3. Oxygen Trap System (OTS)

Built for Versatility

Our high-temperature STA and DSC systems are designed with adaptability at their core, ensuring they meet the diverse requirements of any application. Understanding that no two analyses are alike, these instruments are engineered to offer maximum flexibility, enabling researchers to tailor their setup precisely to their experimental needs.

Key to this versatility is the modularity of our high-temperature product line, which includes features such as dual-hoist functionality, allowing seamless transitions between measurement configurations.

With different furnace types available, users can select the ideal thermal environment for their specific study – whether it involves rapid heating, high-temperature stability, or specialized atmospheres. Furthermore, the systems offer over a dozen sensor options, ensuring optimal sensitivity and precision for a broad range of materials and thermal events (Figure 4).

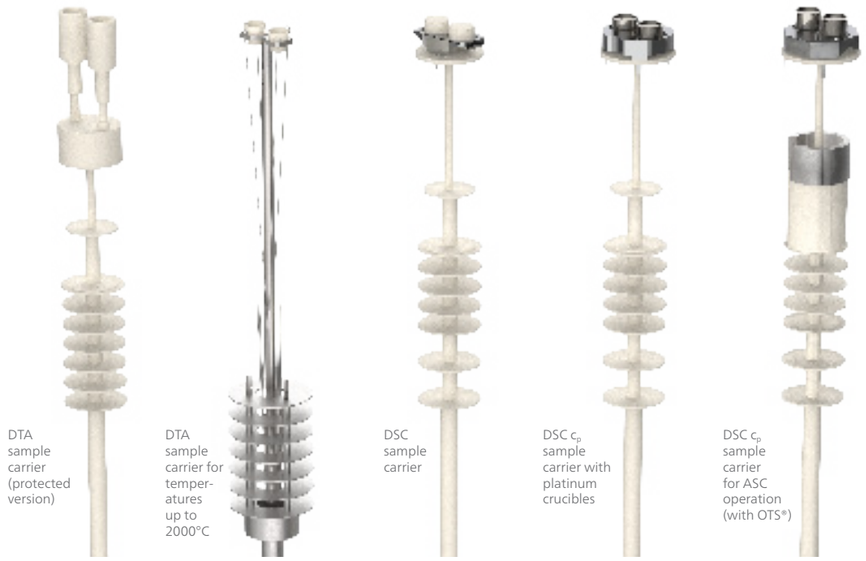

Fig. 4. STA sample holder variety

STA 509 & DSC 500

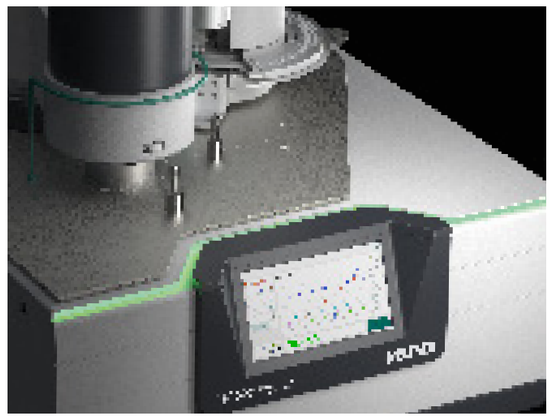

Fig. 5. Light Communication Unit

Change of User Interaction by New Design

The STA 509 *Jupiter*® and DSC 500 *Pegasus*® feature a modern approach to user interaction, centered around the innovative Light Communication Unit (Figure 5) and an intuitive touch display. The Light Communication Unit provides real-time visual feedback, enabling users to monitor the instrument's status immediately. Combined with the responsive touch display, which offers an intuitive and user-friendly interface for setup, control, and data visualization, these features transform the way users interact with the instruments.

Typical Applications

Melting and Crystallization Behavior of Alloys

Understanding the melting and solidification behavior of alloys is essential for ensuring the performance of alloy materials. Differential Scanning Calorimetry is a valuable tool for analyzing these thermal properties.

The DSC curve (Figure 6) shows the thermal characteristics of a nickel-based superalloy, known for its durability in extreme environments such as gas turbines and jet engines, with significant endohermic peaks in the heating step at about 1244°C, 1348°C, and 1362°C, indicating different phase transitions during the melting process. The cooling curve shows exothermic events at approximately 1170°C, 1237°C, 1297°C and 1345°C, reflecting the complex crystallization behavior of the alloy.

Analysis of Cement by Means of STA

Precise knowledge of cement composition is essential for optimizing its performance and durability. Simultaneous Thermal Analysis is a powerful tool that provides these detailed insights by a combined analysis of the energetic and gravimetric properties of cement.

Figure 7 illustrates both Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC) results for a cement sample, highlighting key thermal events.

The TGA curve (green) shows significant mass losses at distinct stages, indicating dehydration (7.5%), dehydroxylation (3.5%), decarbonation (5.9%) and desulfurization (17.2%). The DSC curve (blue) reflects the related energetic reactions with peak temperatures at values such as 153°C, 455°C, 783°C, 320 °C and 1386 °C. In addition, further structural transformations occurring at 575°C (α to β SiO_2) and 1217°C (β to α $CaSO_4$) are rendered visuable by endothermic effects.

STA 509 & DSC 500

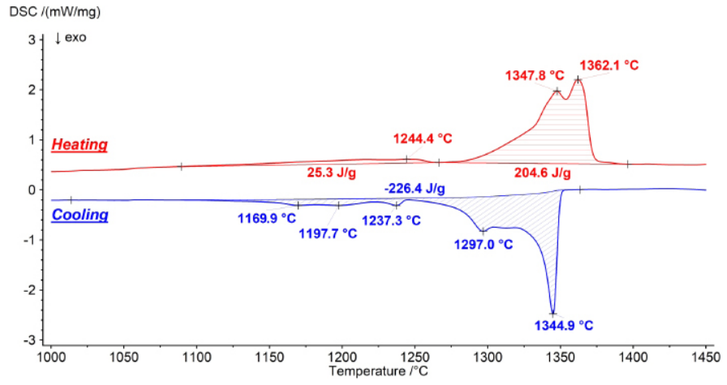

Fig. 6. DSC measurement on a nickel-based superalloy

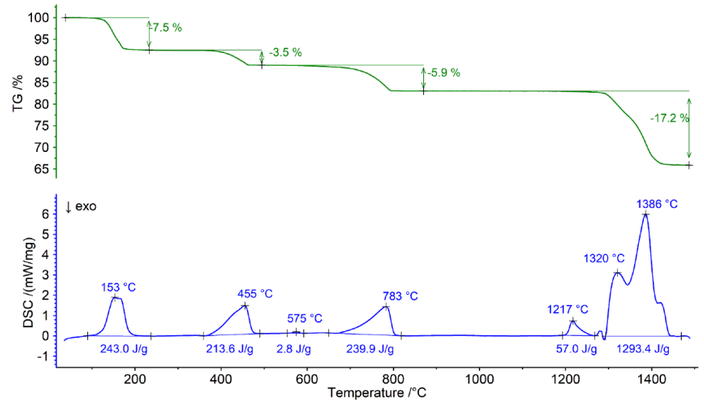

Fig. 7. STA measurement on cement; presented are the TGA curve in green and the DSC curve in blue.

Summary

In general, STA and DSC techniques are versatile and reliable tools for optimizing the understanding of materials used in high-temperature environments. This is particularly true of the NETZSCH STA 509 *Jupiter*® and DSC 500 *Pegasus*®, which offer critical insights into the thermal behavior, stability, and phase transformations of materials. This knowledge helps create safer, more durable, and more efficient materials for demanding applications in both industry and academia.

For more information, please visit https://netzs.ch/simultaneous-thermal-analysis

Ready for Special Tasks

The STA 509 *Jupiter*® and DSC 500 *Pegasus*® are available in a range of configurations, including specialized setups designed for demanding applications. These include operation in corrosive gas environments, water vapor atmospheres, glovebox setups, and even hydrogen applications with the integration of our TÜV*-certified hydrogen solution, H_2Secure. This versatility makes our high-temperature instruments some of the most adaptable and reliable solutions for STA and DSC analysis, ready to meet the unique challenges of advanced research and industrial needs.

*TUV = German Technical Inspection Authority

LFA 717
HyperFlash® (HT)

Thermal Design – Looking Beyond Cooling Boundaries

Dr. Stefan Diez and Nico Dilsch, R&D

Fig.1. The LFA 717 *HyperFlash® HT* (left) and LFA 717 *HyperFlash®* (right)

Introduction

Modern technology without powerful electronic components – almost unthinkable. Batteries, processors, LEDs, etc. are involved everywhere these days, from smartphones to cars to large technical systems. However, such electrical and electronic components produce heat during operation, which must be consistently dissipated into the environment. Not properly managed, excessive heat can accelerate ageing of these components, degrading their performance, or even lead to complete system failure.

The environmental conditions influence the properties of these systems. Just think of the diminishing performance of batteries of electric cars in wintertime. Appropriate thermal management is therefore necessary. One important aspect for thermal design is knowledge of the heat transfer properties of the materials used.

The heat transfer property of a material is directly related to the terms of thermal conductivity and thermal diffusivity. While thermal conductivity describes the ability of a material to conduct heat, thermal diffusivity is a measure of how quickly a material reacts to a temperature change.

717: A Number Worth Remembering

Laser or light flash analysis is often the preferred method for measuring the thermal diffusivity of a specimen. The combination of the resulting data with density and specific heat capacity of the material enables precise determination of thermal conductivity.

The LFA 717 *HyperFlash®* models (Figure 1) are newly designed, cutting-edge instruments for the measurement of thermophysical properties of materials – especially of the thermal diffusivity, but also of the specific heat capacity and thermal conductivity – featuring an intuitive and state-of-the-art operating concept. Two models are available: LFA 717 *HyperFlash®* and LFA 717 *HyperFlash® HT*, which can be distinguished mainly by their temperature application ranges (-100°C to 500°C and RT to 1250°C). Access to a wide range of instrument accessories such as sample holders and reference materials allows the user to perform a variety of both routine quality testing and special applications on high-performance materials.

Challenges in Product Operation Temperatures

In high-tech areas such as battery technology, the investigation of a wide range of atmospheres and temperature environments is essential. The automotive industry, in particular, is vigorously researching the thermal behavior of batteries, especially at low temperatures, to achieve greater safety, efficiency and longevity. Other major areas include LED production (LED = light emitting diode) and the ongoing search for solutions involving cooling high-performance LEDs, such as optimum operating temperatures for improving light yield and service life. Similar cooling requirements

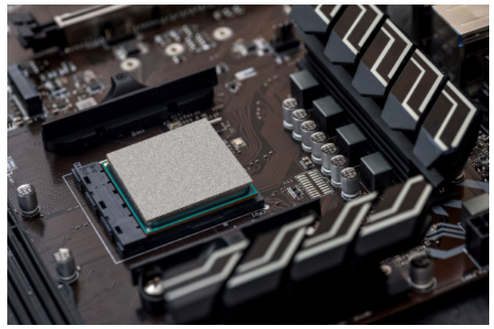

Fig. 2. Graphite interconnects are used between smart electronics, such as processors, and air coolers

are likewise desirable for solar panels, promoting panel efficiency in varying solar radiation conditions, and for a wide range of other smart electronic components.

The new LFA 717 *HyperFlash®* shines in all those special applications where high-performance cooling materials are required by being able to measure thermal conductivities of up to 3000 W/m·K and thermal diffusivities of up to 2000 mm^2/s. As an example, 0.1 mm thin flexible graphite sheets (Figure 2) are an outstanding material for cooling solutions and achieve conductivities up to 1800 W/m·K under ambient conditions. A notable downside of this material is that such high conductivities are only reached in the horizontal direction as graphite is naturally a layered carbon allotrope. To produce graphite foils of varying thicknesses, polymer-based adhesives are used to stack the individual graphite layers. As a result, the multilayered product of this process exhibits different conductivities in vertical and horizontal directions.

In order to successfully investigate the multidirectional thermophysical material properties of highly conductive graphite foils, carefully thought-out processes and strategies usually have to be followed when collecting and evaluating data. To facilitate the analysis of these orthotropic materials, a new calculation model had been introduced into the latest NETZSCH *Proteus®* software (version 9) for LFA 717 *HyperFlash®*: The In-Plane Model for orthotropic materials (IPO).

Special Case of Anisotropic Materials: The In-Plane Model for Orthotropic Materials (IPO)

The graphite sheet specimen should be considered a single-layer material that is impervious to light. Despite their sub-mm thickness, these materials are heterogeneous in terms of thermophysical properties and feature substantial anisotropy in perpendicular direction at different positions. Understanding the thermal diffusivity properties in both the horizontal and vertical direction is essential for evaluating the material properties for the aforementioned applications.

The IPO model assumes transverse isotropy, which means that a sample of constant temperature exhibits an isotropic thermal diffusivity in its horizontal xy-plane (α_{rad} = radial heat diffusion), and solely the diffusivity in z-direction (α_{ax} = axial heat diffusion) deviates uniformly. To isolate the axial and radial components from the data and analyze them, an LFA measurement must be carried out as illustrated in Figure 3. Two frequently applied setups, which are referred to as the inward or outward heat flow variation* of the LFA experiment, are available.

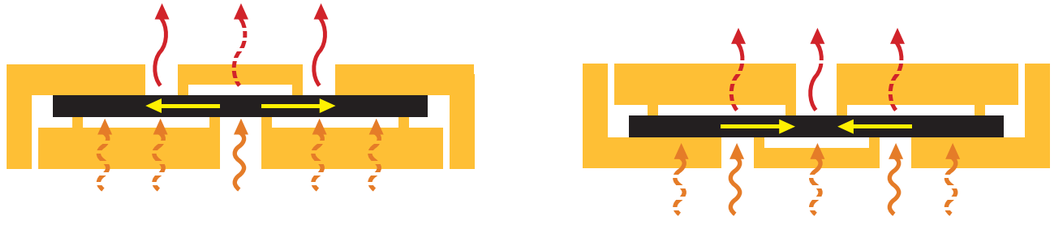

Fig. 3. Scheme of two typical measurement alignments; outward heat flow (left), inward heat flow (right).

* We recommend working with as little energy as necessary and carefully choosing the measurement geometry.

A graphite sample was investigated subsequently in two alignments, through-plane (perpendicular to the foil surface) and in-plane (parallel to the foil surface). The measurement results reveal that the in-plane diffusivity of the graphite foil (approx. 790 mm^2/s) is two orders of magnitude higher than its through-plane diffusivity (Figure 4).

The task was to employ a simulation to verify the observation as well as generalize it. For materials with significantly different thermal diffusivities in the axial and radial directions, these effects of this anisotropy can no longer be ignored. Highly oriented graphite foils were chosen for the study due to their extreme anisotropy, where the radial thermal diffusivity is 400 times higher than the axial diffusivity. This characteristic requires analysis methods that explicitly account for such directional differences. The parameters listed in Table 1 were used for the simulation.

Two distinct approaches were evaluated: the traditional isotropic model, which postulates uniform diffusivity in all directions, and the novel orthotropic model (IPO), which acknowledges the anisotropic characteristic nature of the material. The isotropic model yielded a radial diffusivity of 779.872 mm^2/s, a value that is 2.5% lower than the actual input value of 800.00 mm^2/s (Figure 5). This discrepancy highlights the inaccuracy of the isotropic model when applied on anisotropic specimens. In contrast, the orthotropic model exhibited superior accuracy for anisotropic materials, with an error of less than 0.2% in the calculation of the radial diffusivity.

Orthotropic Materials

Orthotropic materials have different properties in three mutually perpendicular directions. This means that their mechanical and thermal properties vary depending on the direction in which they are measured.

This directional dependence makes orthotropic materials a subset of anisotropic materials, which have properties that change with direction.

Table 1. Simulation parameters

Through-plane diffusivity (α_{ax}) = 2 mm^2/s	2.0 mm^2/s (z) This axial value is dominated by the polymeric adhesive, a rather weak thermal conductor, and additional thermal resistance within the layer structure.
Thermal diffusivity at ambient temperature in radial direction (α_{rad}) = 800 mm^2/s	800 mm^2/s (xy) The order of this diffusivity is typically several magnitudes higher. These high values are close to those of natural diamond and characterize extremely efficient thermal conductors.
Sample thickness	0.1 mm (= 100 micrometer)

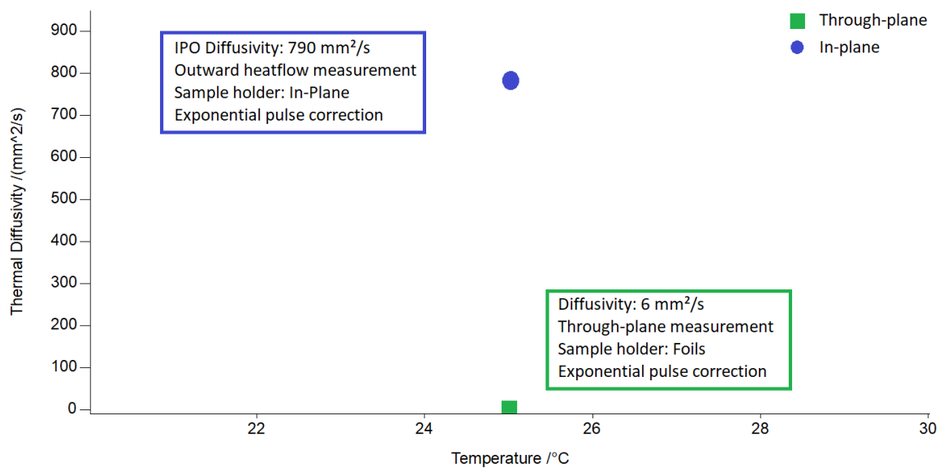

Fig. 4. The experimentally determined thermal diffusivities of a round-shaped graphite foil sample (diameter: 25 mm; thickness: 100 µm).

LFA 717
HyperFlash® (*HT*)

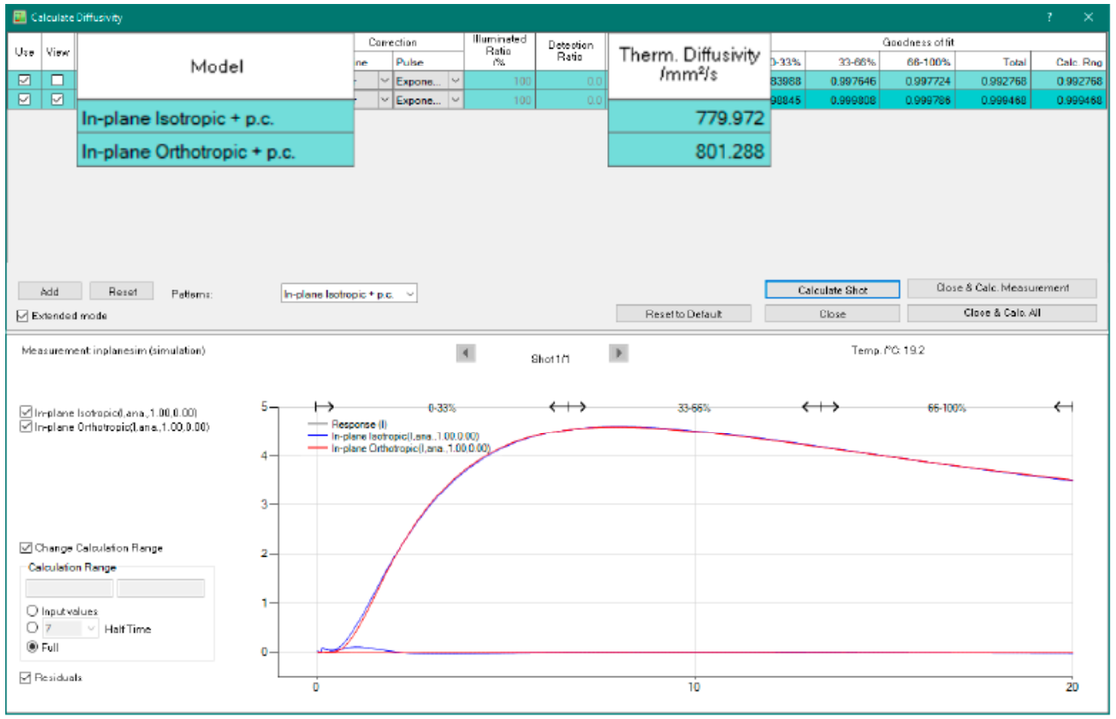

Fig. 5. Data simulation with known diffusivity parameters (provided in Table 1) fed into the analysis process of the latest LFA analysis software

Summary

The LFA 717 *HyperFlash®*, as a next-generation instrument for investigating thermal properties, seamlessly combines cutting-edge hardware with innovative software solutions. The aim is to offer unparalleled flexibility to customers; to dispel their preconceptions by dissolving the limitations that characterized earlier LFA models.

The results clearly demonstrate that even for thin specimens with large differences in thermal diffusivity between the axial and radial directions, the anisotropy should not be neglected. The assumption of isotropic properties leads to significant inaccuracies, while the orthotropic model provides a precise representation of the actual behavior of the material. This finding has significant practical implications, as accurate modeling of anisotropic materials help improve the efficiency of thermal management systems in high-tech applications.

More information can be found on our website netzs.ch/LFA717

High Performance Due to Precision: Quality Assurance in the Production of Technical Ceramics

CeramTec GmbH, Marktredwitz, Germany

Fig. 1. CeramTec develops and produces technical ceramics for customers in a wide range of industries.

About CeramTec GmbH

Technical ceramics offer many benefits. In order for them to be reliably used, the quality of the materials must be tested. CeramTec has relied on NETZSCH's expertise for many years – whether in development projects, in the manufacturing process or in series production, NETZSCH analyzers are in permanent use.

CeramTec's Innovation and Technology Section continuously researches and develops materials and manufacturing processes for new products (Figure 1). The Tape and Substrate Applications Department is focused on the development of new ceramic substrates and their optimization.

Aluminium Nitride High Performance

One successful example: The new AIN HP (Aluminum Nitride High Performance) offers significantly higher flexural strength than other substrate materials while maintaining its thermal properties. It is particularly suitable for continuous loading in power modules and is used in power generation and distribution, vehicle electrification and power converters in rail vehicle construction.

Insight into the Lab: Thermal Analysis of AIN

When it comes to measuring thermal properties, the team relies on NETZSCH products. Consistently positive experiences, the proximity of the locations and the excellent service have led to the use of more and more NETZSCH measurement technology. An overview of the technology used for thermal analysis is shown in Figure 2.

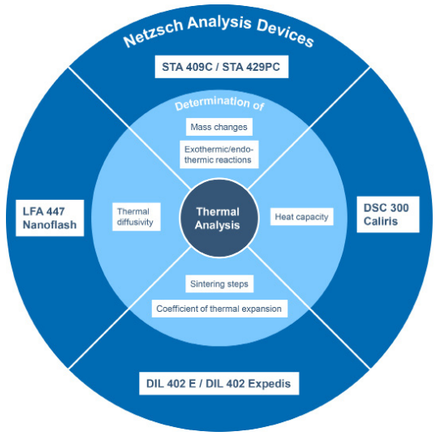

Fig. 2. Overview of NETZSCH analytical instruments and associated measurement tasks at CeramTec GmbH

LFA Investigations

Thermal diffusivity indicates how quickly a material reacts to a temperature change and is a material-dependent property. CeramTec tests the thermal diffusivity for ceramic substrates such as AIN (Figure 3) in the laboratory using a NETZSCH LFA 447 *NanoFlash*. To this end, the laboratory team prepares the test specimen to the format specified for the test device and coats it with graphite. The thermal conductivity can then be determined in a range from 20°C to 300 °C.

Figures 4a and 4b show a comparison of the temperature increase over time following the application of energy to an oxide ceramic (Figure 4a) and a nitride ceramics (Figure 4b). The temperature increase is higher for the nitride ceramics.

The thermal conductivity (λ) can be calculated from the thermal diffusivity (α) determined from the temperature increase as well as the specific heat capacity (c_p) and the density (ρ) of the material:

$$\lambda(T) = \rho(T) \cdot c_p(T) \cdot \alpha(T)$$

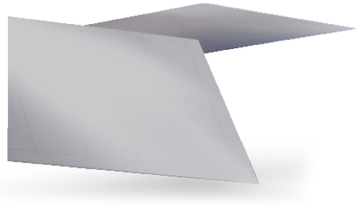

Fig. 3. Aluminum nitride

For aluminum nitride ceramics, this results in thermal conductivities to over 170 W/(m·K), depending on the material type. Depending on the application, the ceramic requires either low or high thermal conductivity. Especially in the case of power semiconductors, which generate high temperatures, the heat must be dissipated quickly and reliably.

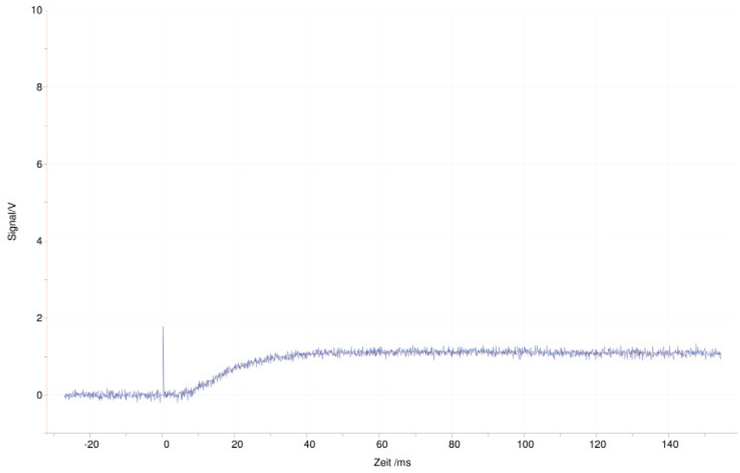

Fig. 4a. Temperature increase over time following the application of energy to an oxide ceramic.

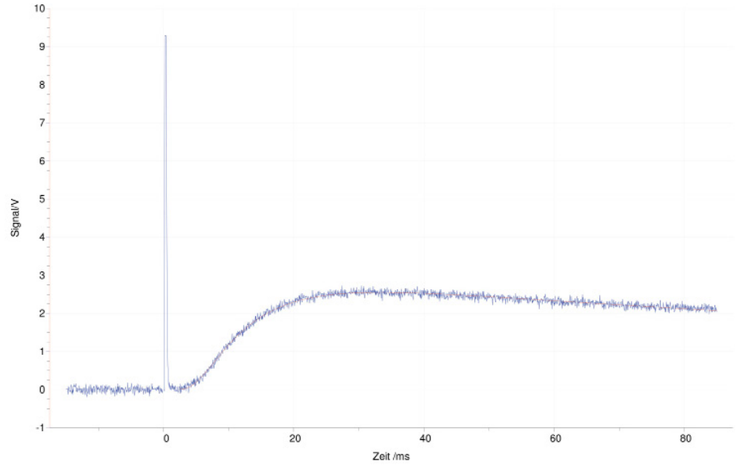

Fig. 4b. Temperature increase over time following the application of energy to a nitride ceramic.

CUSTOMERS FOR CUSTOMERS

Thermal Expansion Coefficient (CTE)

Thermal analysis also includes consideration of the coefficient of thermal expansion (CTE). The thermal expansion indicates how the geometric dimensions of a body change with temperature. This knowledge is important for calculating the thermal mismatch in material combinations, for example.

A precisely determined CTE is also important for metallization and packaging in order to know the tolerances of the outer dimensions of a substrate, for example. CeramTec determines the coefficient of thermal expansion of sintered materials in the laboratory using the NETZSCH DIL 402 E and DIL 402 *Expedis*® dilatometers (Figure 5). The thermal expansion of a ceramic body can be investigated in the temperature range up to 2000°C.

In addition, dilatometers offer the ability to carry out measurements under different atmospheres – such as air, nitrogen or argon – via gas control. This is important in order to be able to conduct measurements in the high-temperature range, for example. The *Proteus*® analysis software provides support in evaluating the measurement curve and determining the thermal expansion in different temperature segments.

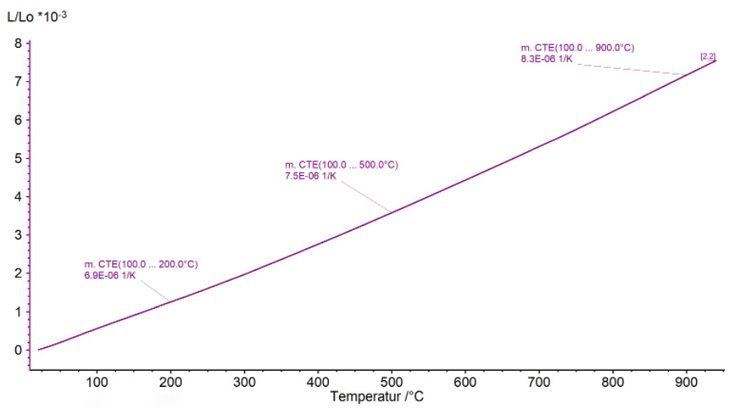

Fig. 5. Change in expansion of oxide ceramics as a function of temperature. The mean (average) coefficient of the linear thermal expansion corresponds to the slope of the linear connecting line between the temperature points given in brackets (6.9 to 8.3 [10^{-6}/K]), it is in agreement with the typical values for a ceramic.

STA and DSC Measurements

Simultaneous thermal analysis (differential scanning calorimetry in combination with thermogravimetry) is also part of thermal analysis. It is primarily employed for the investigation of exothermic and endothermic reactions as well as weight changes in raw materials (powders, binders and organic materials) under air and in green tapes under air or nitrogen. Various NETZSCH STA systems are used by CeramTec for these measurements.

The heat capacity describes how the measured temperature of a body changes in relation to the amount of heat added to it. CeramTec determines this for sintered materials using the NETZSCH DSC 300 *Caliris*®.

Another laboratory task in connection with the thermal parameters is monitoring the production process, since the temperature curve describes the temperature of the furnace and thus that of the sintering process. For example, the dilatometer can be used to determine sintering steps.

Ready for Top Performance

By the time a substrate leaves production at CeramTec, it will have been extensively tested: It is ready for use in high-tech electrical applications and for its material-specific advantages to be utilized. Laboratory analyses are essential not only for quality control, but also for the development of new innovative products. NETZSCH is an important partner to CeramTec in this regard.

Material Intelligence: The Key to More Efficient and Sustainable Products

Dr. Marc Egelhofer, Senior Marketing Manager, LabV®

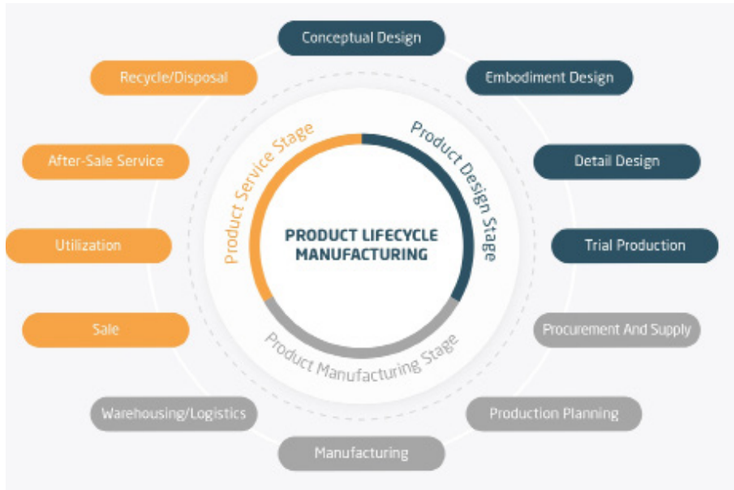

Fig. 1. Material data influences decisions throughout the entire product lifecycle – from the concept phase through production to continuing development (adapted from [1]).

Introduction

The world faces major global challenges, including climate change, resource scarcity, and the need for more sustainable management of natural resources. Innovative solutions are essential to address these issues, particularly in industrial development and manufacturing, where sustainable and future-ready solutions are needed. Material data plays a central role in enabling informed decisions and accelerating innovation processes. It holds the key to creating better products and more efficient operations.

The Role of Material Data in the Product Lifecycle

Material data influences the entire product lifecycle – from the initial concept to quality control (Figure 1). Traditional systems like Laboratory Information Management Systems (LIMS) often reach their limits here: They store data in isolation and lack full connectivity and advanced analytics, thus restricting the utilization of this data. LabV®, as a Material Intelligence Platform, offers a comprehensive solution: It integrates data from various sources, centralizes it, and makes it accessible and usable through intelligent, AI-driven analytics for both product development and quality assurance.

What Defines a Material Intelligence Platform?

A Material Intelligence Platform (MIP) goes far beyond the capabilities of conventional systems. It collects, integrates, and analyzes diverse material data using artificial intelligence (AI) and machine learning (ML). The result is a centralized, continuous platform that supports data-driven decisions at every stage of the product lifecycle. This leads to more precise material selection and targeted management of development and manufacturing processes, thereby shortening development times and optimizing resource utilization.

Material Data: More Than Just Numbers

Material data encompasses a wide range of information, such as raw material data, formulations, mixing ratios, measurements, and processing parameters from pilot plants or production. It provides a comprehensive view of a product's behavior and properties – from initial formulation through to production. LabV organizes and contextualizes this data, allowing companies to gain precise insights into their materials at any time. This ensures consistent material properties, which can be leveraged for product development and compliance with regulatory standards.

LabV®

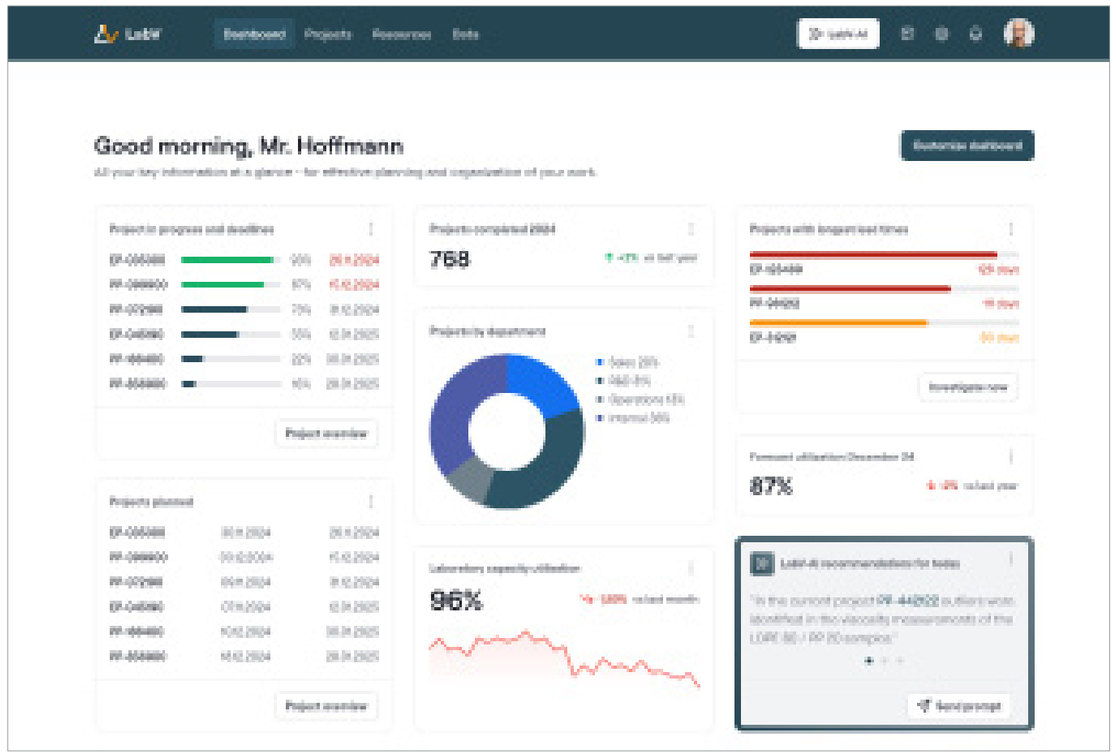

Fig. 2. .LabV® Dashboard – all essential data at a glance.

Figure 2 illustrates how intuitive dashboards within the LabV® Material Intelligence Platform make diverse data and complex information accessible at a glance. These representations facilitate analysis and allow insights to be quickly put into action. The AI-powered digital assistant also aids in the analysis and visualization of complex datasets, enabling users to quickly identify trends or spot abnormalities (Figure 3).

The Benefits of Material Intelligence

A Material Intelligence Platform combines AI and ML with a user-friendly interface and complete data connectivity, delivering significant benefits across the entire value chain:

1. Accelerated Product Development

An MIP enables fast and efficient development and adjustment of new products and formulations. For example, in the development of new polymer products, the platform allows immediate analysis and optimization of formulations, so adjustments can be made in the shortest possible time. This rapid access to relevant data and instant adjustment capability greatly accelerates the development process, helping companies respond quickly to market changes.

2. Optimized Quality Assurance

Quality control is crucial for product safety and brand reputation, especially in highly regulated industries such as the automotive sector. Here, LabV® acts as an early warning system, detecting quality deviations in real-time so they can be corrected early. Continuous monitoring and analysis of complete material property overviews help ensure product consistency, strengthen customer trust, and maintain quality over the long term.

Fig. 3. The AI-powered digital assistant enables data analysis and visualization of complex data sets.

3. Resource Efficiency and Sustainability

An MIP supports companies in achieving their sustainability goals by enabling more efficient use of raw materials and reducing waste. With LabV®, companies can quickly identify inefficiencies and make targeted optimizations, such as adjusting temperature or material flow during production. This conserves resources while also reducing costs.

4. Cross-Departmental Collaboration and Transparency

Material Intelligence enhances not only efficiency within individual departments but also cross-departmental collaboration. Research, development, production, and quality control teams share a common data base, creating transparency and supporting well-informed decision-making. By eliminating silos and harmonizing processes through LabV®, the seamless flow of information across departments is facilitated.

Conclusion

Material Intelligence and a powerful platform like LabV® are key to developing competitive and sustainable products. Through the central collection, integration, and analysis of material data, an MIP enables informed decision-making and accelerates innovation. In this way, Material Intelligence paves the way from data to better products.

Reference

[1] Wang, L., Liu, Z., Liu, A., & Tao, F. (2021). Artificial intelligence in product lifecycle management. The International Journal of Advanced Manufacturing Technology, 114, 771–796.

For further information, go to
https://netzs.ch/labV_en

Kinetics Neo Software

Kinetic Analysis of Pressure-Dependent Reactions in Solids

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Introduction

Every one of us is confronted with chemical reactions daily, and the rate of these processes depends on temperature. We enhance temperature during the baking of food to make it faster. We put our food into the refrigerator to slow down degradation processes. Temperature is also important in industrial processes for polymers or ceramic production.

Our Kinetics Neo software was created for the analysis of reaction rates depending on temperature conditions. It allows for creating kinetic models based on laboratory measurements, and then simulating the reaction progress at lower temperatures, for lifetime predictions or for the optimization of the temperature profile in industrial applications like polymers, food, pharmaceuticals or ceramic industries, to reduce costs and improve the quality of the final products.

However, many reactions depend not only on temperature, but also on additional parameters. The degradation of polymers depends on humidity and light exposure; the curing of photopolymers[1] depends on the intensity of UV light. The rate of many processes depends on the presence and concentration of the active component in the atmosphere. For example, enhancing the oxygen content increases the rate of oxidation reactions, and an increase in hydrogen concentration increases the rate of reduction reactions. The overall rate of reversible decomposition depends on the product concentration in the surrounding atmosphere, such as decomposition with water release in a humid atmosphere. Also, in an inert gas, some reactions exhibit pressure-dependent behavior.

Now available is the new version of Kinetics Neo, 3.0, in which it is possible to create a common kinetic model dependent on both temperature and pressure.

Influence of CO_2 on the Reversible Composition of $CaCO_3$

The decomposition of calcium carbonate in the presence of carbon dioxide is the reversible reaction:

$$CaCO_3 \rightleftarrows CaO + CO_2$$

in which two chemical reactions occur simultaneously.

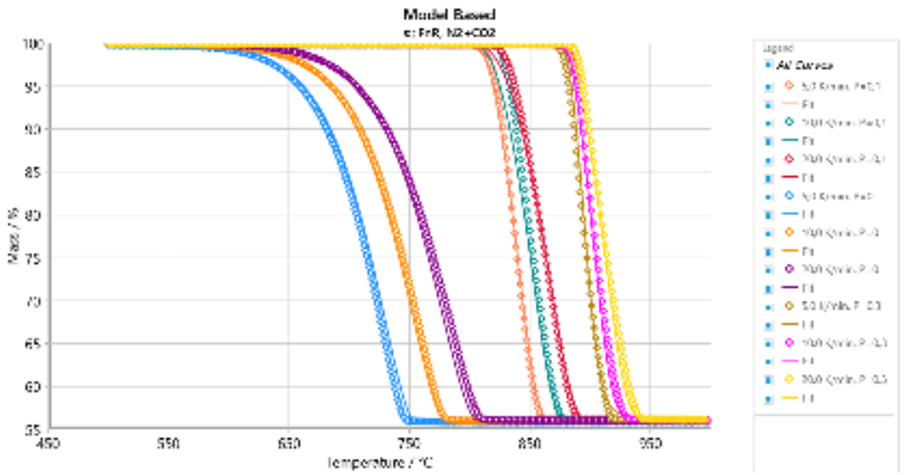


Fig. 1. Common kinetic model of $CaCO_3$ decomposition at heating rates of 5, 10 and 20 K/min, depending on the partial pressure of CO_2 of 0, 10 and 30% in the mixture of CO_2 and N_2. Perfect fit between the calculated curves (solid lines) and experimental data (symbols).

[1] Photopolymer resin is a polymer that changes its properties when exposed to light, most commonly ultraviolet light.

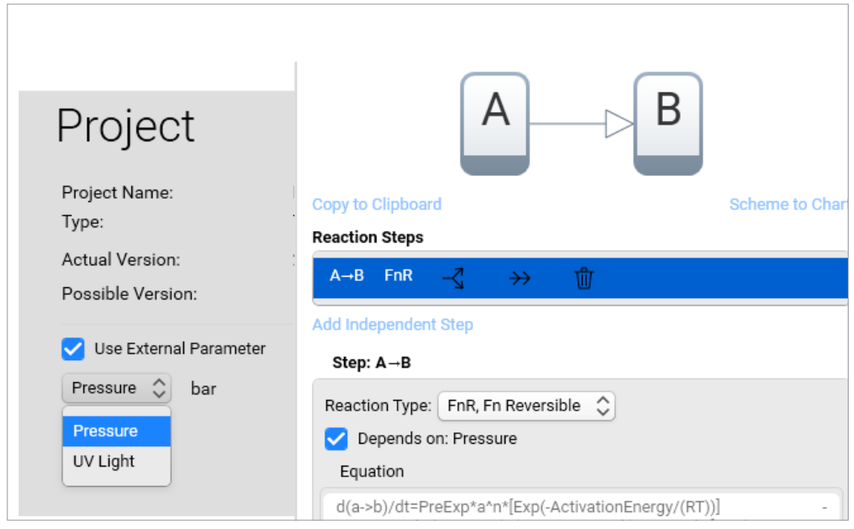


Fig. 2. It couldn't be easier to take the pressure dependency into account.

Here, the first one is the forward reaction with CO_2 as the product, and is therefore independent of the CO_2 concentration. The second reaction is the reverse reaction having CO_2 as the reactant and depending on its concentration.

The rate of the reverse reaction is higher for higher partial pressure of CO_2. The cumulative reaction rate is the difference between the forward reaction and reverse reaction, and it becomes slower for high concentrations of CO_2:

$$\frac{d\alpha}{dt} = A_1 \exp\left(\frac{-E_1}{RT}\right) f_1(\alpha) - P^{n_p} A_2 \exp\left(\frac{-E_2}{RT}\right) f_2(\alpha)$$

where P is the partial pressure of carbon dioxide, and n_p is the pressure parameter.

Figure 1 illustrates the common kinetic model for 9 experimental curves, measured at 5,10, and 20 K/min with a partial pressure for CO_2 of 0%, 10% and 30% in the two-component gas mixture of CO_2 and nitrogen with a total pressure of 1 bar.

An increasing CO_2 concentration leads to a shift of the mass loss curves to higher temperatures. In addition, a shift with the heating rate can be observed at a constant CO_2 concentration, although this is lower than before.

Pressure-Dependent Reactions in Inert Gas

Some solids decompose with the release of a gaseous product. If this gaseous product is non-reactive, then its presence or absence has no influence on the main decomposition rate. But sometimes, the gaseous product can react with another product, as happens in reversible decompositions. For reversible reactions, the increase in pressure of the inert gas leads to a decrease in the diffusion coefficient, and to an increase in the local concentration of the gaseous product, which cannot easily be removed from the reaction zone. Therefore, the high pressure of the inert gas increases the rate of the reverse reaction.

This example presents the three-step kinetic analysis of calcium oxalate monohydrate under enhanced pressures of nitrogen, where the first and the last steps

Kinetics Neo Software

are reversible reactions, and are therefore pressure-dependent. The second step is a non-reversible reaction, and independent of pressure.

$$CaC_2O_4 * H_2O \rightleftarrows CaC_2O_4 + H_2O$$

$$CaC_2O_4 \rightarrow CaCO_3 + CO$$

$$CaCO_3 \rightleftarrows CaO + CO_2$$

In closed systems, the final concentrations of all reactants are in equilibrium, where the rates of forward reaction and reverse reaction are equal. However, in thermal analysis like DSC or TGA, the system is open; all gaseous products will be removed by the purge gas, and no equilibrium occurs.

Experimental measurements are carried out in pure nitrogen under different pressures. Higher pressure makes the diffusion of H_2O and CO_2 products difficult, and these reaction steps become slower and are moved to higher temperatures. The higher the pressure of the inert gas, the slower the reversible reactions, and the higher the decomposition temperature.

Figure 3 shows the common kinetic model for 8 experimental curves, measured in nitrogen at 2, 5,10, and 20 K/min under normal pressure and additionally at 20 K/min under 5,10, 20 and 50 bar. This three-step model was built in Kinetics Neo software and has two pressure-dependent steps: the release of water in the first step and the release of carbon dioxide in the last step. Kinetic equations for these steps contain the pressure *P* and its order n_p. The second step is a non-reversible reaction and therefore has no dependence on the nitrogen pressure.

For the first and the third reaction, a shift in the TGA curves to higher temperatures with increasing heating rates and increasing nitrogen pressure can be observed. The second reaction remains, indeed, virtually unaffected by the pressure increase. The impact of the heating rate is much lower as well.

Summary

All these kinetic models were created in our new version, 3.0, of the Kinetic Neo software, in which it is possible to model the pressure influence. Each of these models can be used in Kinetics Neo for the prediction of the reaction rate at a given temperature profile and given partial pressure of the gaseous reactant, or at a given total pressure of the inert gas.

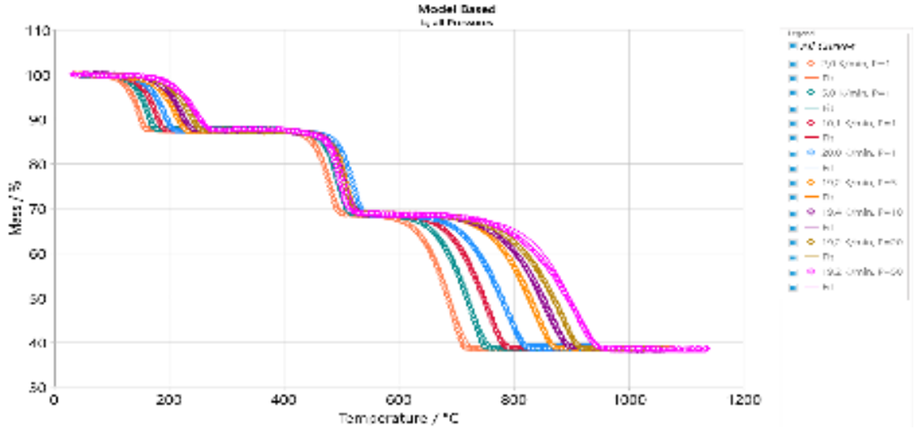


Fig. 3. Common kinetic model of the calcium oxalate monohydrate decomposition at 2, 5, 10 and 20 K/min in a nitrogen atmosphere of 1, 5, 10, 20 and 50 bar, depending on the total pressure. Comparison of the calculated curves (solid lines) with the experimental data (symbols).

DMA Measurements Under Defined Humidity – Expansion for the the New DMA 303 *Eplexor*®

Martin Rosenschon, Customer Training, Sascha Sebastian Riegler, Applications Laboratory, und Dr. Georg Storch, Head Experimental Workshop

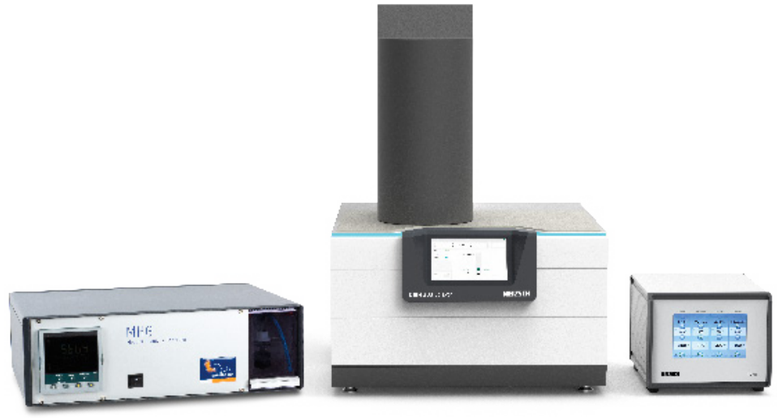


Fig. 1. DMA 303 *Eplexor*® with humidity generator (left) and TRG 004 temperature controller (right)

Introduction

Humidity and temperature are critical factors when analyzing the operating and application conditions of components and products. Depending on the material and its chemical composition, humidity may affect material properties to varying degrees. Relative humidity describes the water-vapor content of an atmosphere in relation to the maximum possible water-vapor content at a given temperature [1]. Relative humidity varies considerably depending on the region, geographical location and time of year.

The level of humidity depends mainly on temperature [1], although pressure [2] and the amount of water vapor present also play a role. While relative humidity can be less than 20% in arid regions such as parts of Egypt, it can reach 90% or more in tropical climates [3].

Typical examples of moisture-sensitive materials are polyamides, which are considered to be highly hygroscopic and absorb water mainly at the amide groups in their amorphous regions. The absorbed water changes the intermolecular forces in the polyamide and thus acts as a plasticizer.

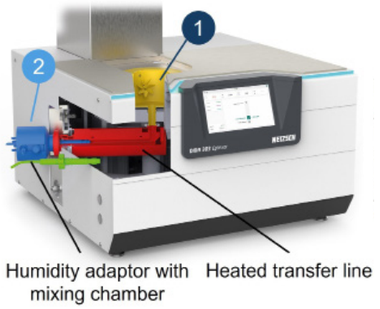
As shown for PA 6 [4], a relative humidity of 50% can reduce the storage modulus E', which describes the elasticity of a material, or in simple terms its stiffness, by more than 70% compared to the values in an almost dry environment. This highlights the importance of studying the effects of moisture on materials in order to ensure their functionality in the subsequent application.

New Accessories for the DMA 303 *Eplexor*® – MHG and TRG 004 Humidity Generators

The DMA 303 *Eplexor*® now allows for measurements at a defined relative humidity. To this end, the instrument is coupled with the Modular Humidity Generator (MHG) (Figure 1, left). The external TRG 004 heating unit (Figure 1, right) regulates the temperature of the gas humidifier and the transfer line.

The humid gas flow is generated in a mixing chamber that can be easily connected to the DMA by means of a quick-connect adapter (see Figure 2, left). Condensation-free transfer of the humid gas flow into the sample chamber is ensured by a permanently installed heated transfer line inside the DMA housing. As shown in Figure 2, right, relative humidity values can be realized between 0% and 90%, depending on the measurement temperature.

Expansion for the DMA 303 *Eplexor*®



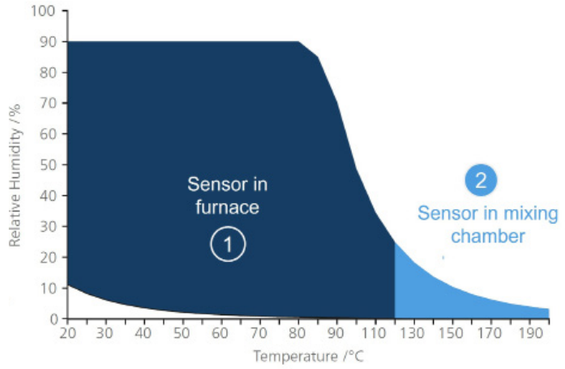
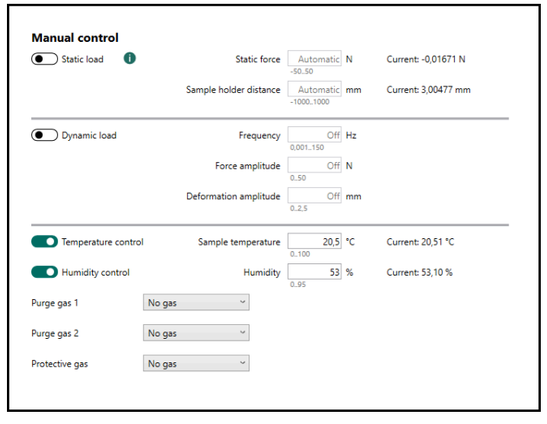


Fig. 2. Sectional view of the DMA303 *Eplexor*® with humidity adapter (left) and achievable relative humidity (right).

A humidity sensor, directly installed in the furnace, serves for precise monitoring and regulation of the relative humidity close to the sample up to temperatures of 120°C. This allows for controlling the humidity content of the gas flow directly after the mixing chamber and thus for analyzing the drying behavior of materials at high temperatures.

One innovation is the full integration of the control of the relative humidity in the measurement software of the DMA 303 *Eplexor*®, eliminating the need for any additional software for the humidity generator. Manual operation allows humidity values to be set and checked directly in the measurement cell (Figure 3, left). When creating measurement programs, the user now has the option of specifying defined humidity values along with a tolerance interval for each segment (see Figure 3, right).



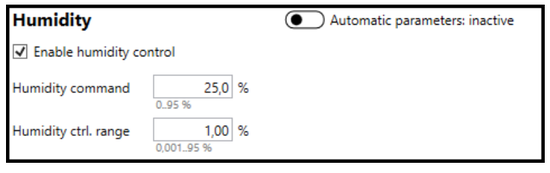


Fig. 3. Quick and easy access to the main settings of the NETZSCH *Proteus*® DMA measurement software. Left: View in "manual mode"; right: Setting the humidtiy in the measurement program.

Expansion for the DMA 303 *Eplexor*®

Viscoelastic Behavior of a PET Film Under Humidtiy

Figure 4 shows the measurement results for a 100-µm polyethylene terephthalate (PET) film dynamically tested in tensile mode at a temperature of 80°C and relative humidity values between 0% to 80%. The PET sample was successively exposed to relative humidities of 0%, 20%, 40%, 60% and 80% in isothermal segments of 120 minutes each. As can be seen, the DMA 303 *Eplexor*® regulates the relative humidity values quickly and accurately. The humidity values measured by the sensor are directly integrated into the measurement result file and can be displayed in the *Proteus*® analysis software without additional data import.

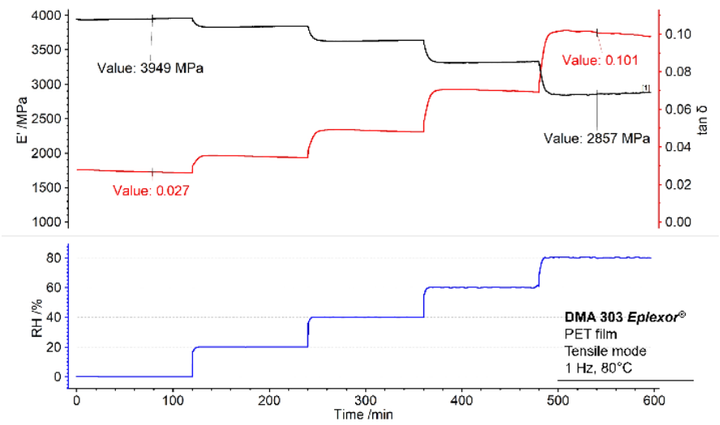


Fig. 4. Development of the storage modulus, E' (black), and loss factor, tan δ (red) of a PET film as a function of the relative humidtiy (blue) at 80°C.

PET is one of the most important thermoplastic polymer materials, accounting for 6% of all technically processed plastics [5]. Compared to the aforementioned polyamides, PET is typically less sensitive to moisture. However, the measurement temperature of 80°C and the high surface-to-volume ratio of the film promote water absorption.

In a dry atmosphere, PET film has a storage modulus E' of approximately 4.0 GPa. The storage modulus describes the elastic properties of the material. As the relative humidity increases, the material becomes softer: E' gradually decreases and reaches a value of about 2.9 GPa at 80%, which corresponds to a reduction of almost 30%. The loss factor, tan δ, which corresponds to the ratio between the viscous and elastic properties and describes the damping properties of the material, increases with increasing relative humidity. Starting from a value of 0.03 in a dry environment, tan δ increases to around 0.10 at a relative humidity of 80%. Accordingly, the PET film becomes increasingly viscous.

Summary

The new expansion of the DMA 303 *Eplexor*® allows for user-friendly analysis of the moisture sensitivity of materials and the influence on their viscoelastic properties. In combination with the humidity generator, relative humidity values up to 90% can be set precisely and reliably. A quick-release adapter makes installation simple and quick. Humidity levels are controlled and adjusted seamlessly via the integrated measurement software for maximum efficiency and ease of use.

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[2] S. A. Bell, S. J. Boyes: An Assessment of Experimental Data that Underpin Formulae for Water Vapour Enhancement Factor. National Physical Laboratory, UK, 2001.
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