WHITE PAPER

NETZSCH

The NETZSCH 360° View

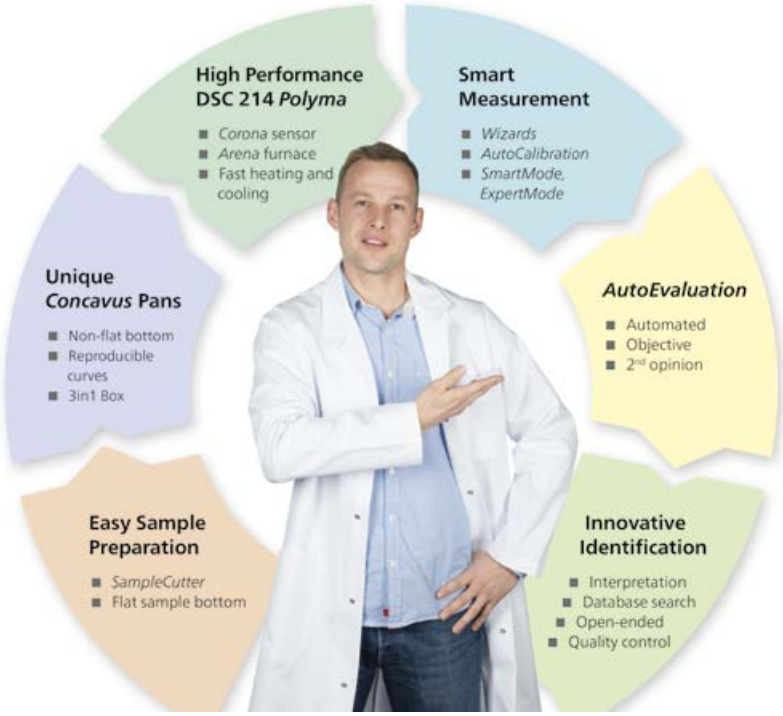
A Streamlined, User-Friendly Approach to the Thermal Analysis of Polymers by DSC

Dr. Andreas Spörrer

Introduction

In today's plastics industry, effectively integrated quality management is essential for zero-defect, state-of-the-art production technologies. Within quality management, detailed knowledge of the state of the materials utilized is necessary in every step of the processing chain since the quality of the polymers directly affects their processability and suitability for end products. Therefore, knowledge of the fundamental chemical and physical properties of polymers is paramount. Besides the polymer grade and filler content, the following properties are of main interest:

- Density
- Viscosity
- Mechanics
- Color
- Surface appearance
- Stabilizers
- Thermal properties



1 NETZSCH 360° view for the thermal characterization of polymers

The most important methods of thermal analysis is differential scanning calorimetry (DSC). The main focus of this article is on a holistic solution with the objective of simplifying the application of DSC for plastic converters such as compounders, extrusioneers, rotary molders, and injection molders. NETZSCH Analyzing & Testing has developed a streamlined approach to perform DSC analyses in such a way to significantly simplify the required individual steps to obtain reproducible and meaningful results.

Redefinition of the DSC Characterization Process

NETZSCH Analyzing & Testing has over 50 years of experience in materials characterization by thermal analysis and over 25 years of experience in the analysis of polymers. The NETZSCH 360° view for thermal characterization (figure 1) is a systematic, streamlined approach for performing high-quality characterization.

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The NETZSCH 360° View

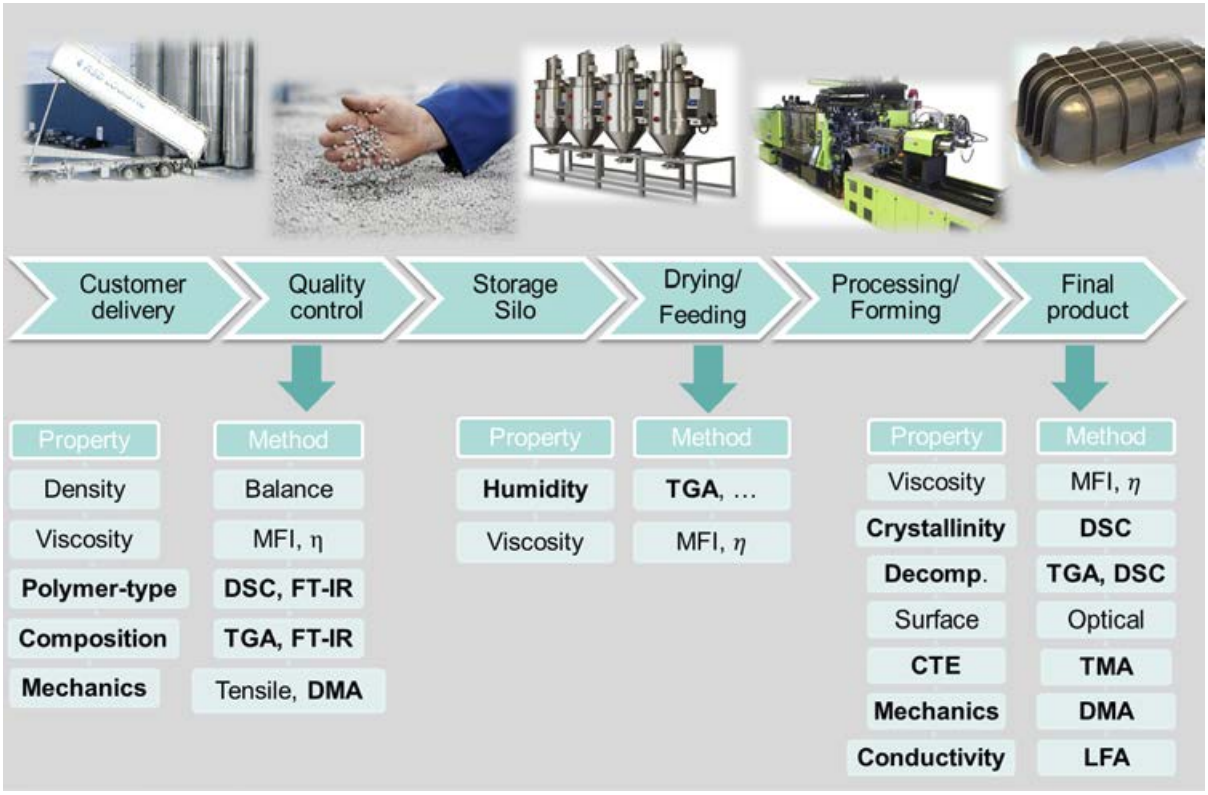
A Streamlined, User-Friendly Approach to the Thermal Analysis of Polymers by DSC

To support this approach, we have designed the new DSC 214 *Polyma* (figure 2) specifically for the thermal characterization of polymer materials in settings ranging from the academic research laboratory to industrial processing plant. The objective of NETZSCH 360° is a customer-oriented solution covering the entire polymer characterization chain (figure 3) – from easy sample preparation to a polymer-specific DSC device to smart evaluation of the results to lean documentation . It supports the customer by enabling characterization of the thermal properties of polymer granules, molded plastics, and rejected parts in an easy, fast, reliable and reproducible manner.

2 The new DSC 214 *Polyma*

NETZSCH 360° Helps Simplify Accessability to the DSC Technology

The NETZSCH 360° solution helps identify even the slightest differences between the different polymer samples by means of DSC which is accessible to everybody, independent of their knowledge about polymers.



3 Application possibilities of thermal analysis on polymers within the process chain at a plastic converter from incoming goods to the finished part

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Sample Preparation

For polymers of any geometry (granules, films, tubular profile or monolithic parts), the first step in their characterization is always the sample preparation. The composition of the sample should be representative of the much larger granule or part. A tiny sample of approximately 10 mg is sufficient for DSC analysis. The mass should be accurate to within ± 5% for better sample comparison. The portion of the sample in direct contact with the aluminum pan should be planar in order to ensure good contact. The *SampleCutter* (figure 4) was designed with these requirements in mind to assist in sample preparation.

3in1 Box – The Ideal Storing and Archiving System

Aluminum (Al) pans are typically used for the DSC analysis of polymers. While these pans are usually purchased in bulk quantities in containers having a single compartment, experience has shown that the quality of the Al pans packaged in this manner might be impaired by deformations caused by interactions between the pans. NETZSCH has designed the 3in1 box, a box with 96 well organized high-quality pans, see figure 5. The NETZSCH 3in1 box offers advantageous solutions such as:

- Storage without contamination
- Integration of labeling for documentation
- Post-analysis sample storage

Pan Geometry

In order to perform a DSC test, two Al pans, a reference pan and a sample pan, must be placed onto the round DSC sensor inside the DSC furnace. From a thermodynamic point of view, these pans must have a form-fitting contact to the sensor to ensure optimal heat transfer between the pan and the sensor. Until now, ideal contact was pursued via a flat pan bottom placed on the truly flat surface of the sensor. In actuality, truly flat Al pan bottoms are not accessible due to manufacturing tolerances. Hence, inconsistent Al pan bottom geometries is one reason for inconsistent thermal resistance between the pan and sensor, resulting in potential measuring errors and limiting the reproducibility of measurements. As a solution, we have designed the new *Concavus* pan (figure 6). As the name of *Concavus* indicates, the Al-pan has a non-flat, concave bottom geometry.

This design offers two main advantages:

- Reproducible contact between the pan and sensor
- Independence of the position of the sample inside the pan

The new *Concavus* pan is recommended for all commercial heat-flow DSC devices, as it significantly helps improve the measurement reproducibility of the DSC signal – from user to user and from sample to sample.

4 *SampleCutter*

6 *Concavus* crucibles

5 3in1 Box

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DSC 214 Polyma

Among the attractive features of the DSC 214 *Polyma* is its small footprint (35 cm x 51 cm). The newly designed DSC sensor and furnace offer optimized thermal resistance and high signal sensitivity and reproducibility (figure 7).

The ring-shaped sensor, named after its ring-shaped thermocouple design, enables detection of the slightest temperature deviations between the reference and the sample pan. With an Indium Response Ratio >100 mW/K, this sensor delivers high-resolution measurements in combination with significantly increased heating rates.

The reduced size and mass of the silver *Arena* DSC furnace, named for its oval geometry, fulfills several requirements:

- Homogeneous temperature distribution for the two pans and the sensor
- Low thermal inertia for high heating and cooling rates up to 500 K/min
- Excellent controllability of the temperature

Combining these three innovations – the *Concavus* pan, the ring-shaped sensor and the *Arena* furnace – results in a DSC measuring system with the following unique properties:

- Very high reproducibility of the individual measurements from sample to sample and user to user
- Exceptionally high heating and cooling rates, allowing for fast DSC measurements and high sample throughput
- High mechanical robustness, appropriate for daily use in any lab or industrial plant

7 Ideal combination of *Arena* furnace (a), ring-shaped sensor (b) and *Concavus* crucibles (c). The concave area is an exaggerated image for better illustration purposes; in reality, concavity is only approx. 10 µm.

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The New Proteus® 7.0 Software

Performing Measurements

Ultimately, the usability of any device depends on its user interface. Well-known for being user-friendly, the new, improved version of the NETZSCH *Proteus®* software (v. 7.0) developed specifically for the DSC 214 *Polyma* offers two operating modes, called *ExpertMode* and *SmartMode* (figure 8).

The objective of *SmartMode* is to make DSC analysis accessible to less experienced users to start a measurement with a minimum of input required. A new GUI (graphical user interface) enables the user to define a reliable measurement within a minute due to available wizards for frequent tasks. Start-up only requires definition of the gases and main parameters for the temperature program. In addition to the measurement program itself, these wizards can also include analytical tasks and report generation. This greatly simplifies both the acquisition of information from the polymer sample and subsequent analysis of the measurement.

Other automatic features of the measurement include *AutoCooling* and *AutoCalibration*.

With the *AutoCooling* function, the most suitable cooling device (i.e. liquid or gaseous nitrogen, intracooler or compressed air) and temperature program are chosen automatically by the software from pre-defined, optimized cooling programs, guaranteeing ideal temperature control and optimized energy consumption, especially for the liquid nitrogen option.

With *AutoCalibration*, prior to the measurement, the software automatically selects the best available calibration file for the measurement, relieving the customer of performing time-consuming instrument calibrations. By means of *AutoCalibration*, the software also reminds the user when a calibration is necessary and carefully guides the user through the calibration procedure.

Analysis of the Data

Analysis of raw DSC data normally requires trained personnel and expert knowledge to identify thermal transitions in the polymer such as glass transitions, crystal relaxations, and crystal melting. Overlapping events are sometimes encountered, which complicates the analysis.

8 New GUI for *SmartMode* with pre-defined easy methods

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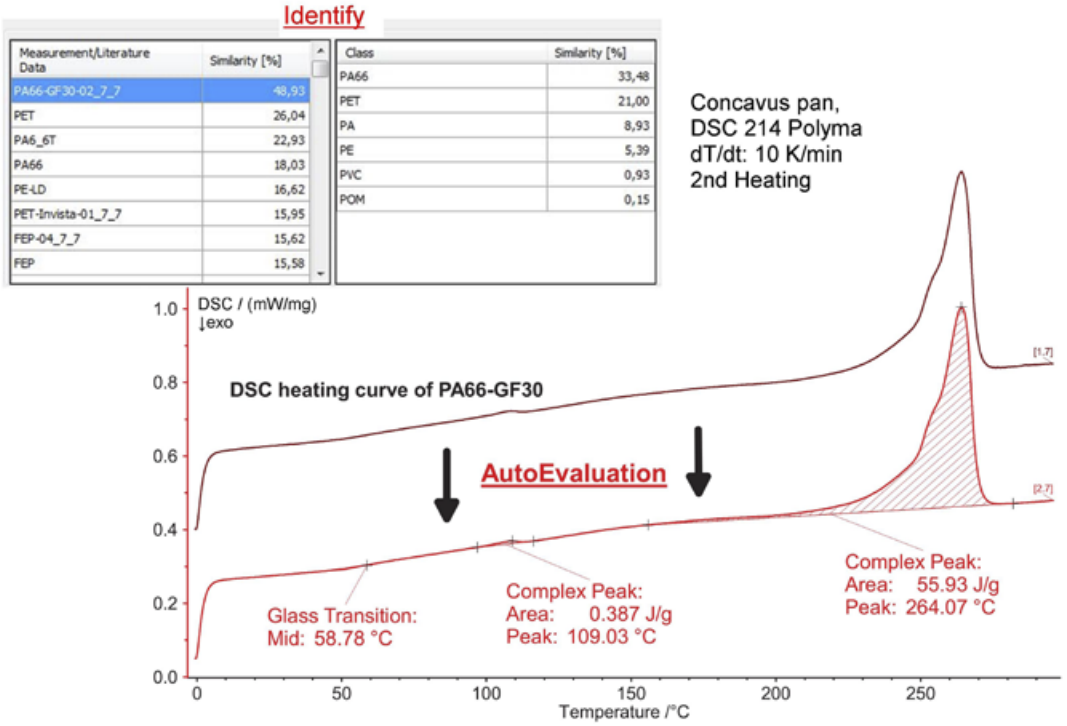

9 Typical curve of a polymer derived from a DSC measurement analyzed with *AutoEvaluation* and the proposal by *Identify*

With the *AutoEvaluation* and *Identify* routines of *Proteus*® 7.0, automatic analysis of DSC curves and identification of the polymer type can be accomplished for the first time. The software automatically analyzes the transitions and evaluates melting or crystallization peaks and glass transition, see figure 9.

Identify uses a software algorithm to compare the properties of a polymeric sample obtained from the analysis of a DSC curve (glass transition, endo- and exothermal peaks) with a database created by NETZSCH (also see figure 9) in order to identify an unknown polymer sample automatically.

The *Identify* database can be easily customized by the user due to the ability to archive measured data. This allows for easy access to previous measurement data at any time. Moreover, it is possible to create own classes for quality control. In this way, it offers a unique tool for material development, incoming goods inspection and quality assurance of molded parts.

Summary and Outlook

The NETZSCH 360° view for DSC technology is a comprehensive system solution specifically tailored to the polymer processing industry. Its supports the entire sequence of thermal analysis activities – from sample preparation to DSC testing, user-independent evaluation and documentation of the test results. The new DSC 214 *Polyma* along with the accompanying automatic sample analysis and identification features of the *Proteus*® 7.0 software greatly simplifies the characterization of polymers by means of DSC.

This provides the user with an all-inclusive system solution considering all aspects to guarantee high-quality DSC measurements on neat polymers and finished plastic parts. Never have DSC measurements been so easy while maintaining the well-known NETZSCH quality.

The Author

Dr. Andreas Spörrer studied materials engineering and obtained his doctorate degree in plastics technology. He has been working at NETZSCH Analyzing & Testing as Manager of the Polymer Business Segment

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