WHITE PAPER

NETZSCH

Superiority of *Convacus* Pans over Standards Al Pans in Combination with the DSC 214 *Polyma*

Dr. Andreas Spörrer and Claire Straßer

Motivation

In order to perform DSC tests, two aluminum (Al) pans, a reference pan and a sample pan must be placed on the DSC sensor inside the DSC furnace. From a thermodynamic point of view, it would make sense that these pans should have form-fitting contact with the sensor to ensure optimal heat transfer between the pan and sensor. Until now, ideal contact had been sought by placing a flat pan bottom on the flat surface of the sensor.

Problem Description

In reality, truly flat bottoms on Al pans are not achievable for two reasons. Firstly, the metal surface of the Al pan exhibits technical roughness, which results in a surface with microscopically small points of contact between the pan bottom and sensor.

Secondly, the Al pans are manufactured by the deep-drawing of a thin Al sheet, meaning that the planarity of the pan bottom is subject to manufacturing tolerances. Accordingly, it is not possible to manufacture truly flat bottoms for Al pans, as the pan's bottom may be curved slightly inward or outward. Even within the same production, two pans may not have perfectly equal bottoms.

Hence, inconsistent Al pan bottom geometries are one reason for inconsistent thermal resistance between the pan and sensor, limiting the reproducibility of measurements. If the pan's bottom is curved outward, there is a point contact instead of a plane contact and the resulting heat flow is impaired. In contrast, a bottom which is curved slightly inward, thus concave, offers a ring-shaped and reproducible contact area with the flat sensor.

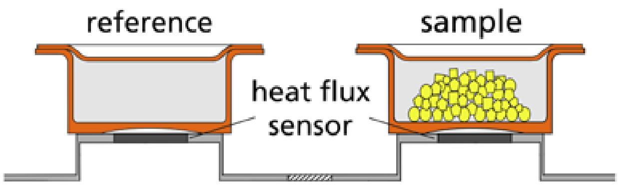
Solution

The issue of the reproducible manufacture of Al pans has been solved with the patent-pending *Concavus* pan by NETZSCH (figure 1), which offers a tiny, but intentionally created concavity of the outside of 10 µm while the internal area for the sample is flat. This design offers two main advantages:

- The thermal contact area between the pan and sensor is reproducible
- The position of the sample inside the pan is irrelevant

This leads to high reproducibility of the DSC measurements while maintaining high sensitivity.

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



1 Two *Concavus* pans on two ring-shaped sensors with ring-shaped contact zone; actual concavity of 10 µm is exaggerated in this figure while the internal area for the sample is flat.

Additionally, the principle behind the *Concavus* pan was carried forward in designing the sensor of the DSC 214 *Polyma*. The ring-shaped sensor consists of a ring-shaped arrangement of the thermocouple materials chromel and constantan, creating the thermal voltage in the ring, not in a point. This design enables the ring-shaped sensor to detect even the slightest temperature deviations between the pan and sensor, making it the perfect complement to the *Concavus* pan. With an Indium Response Ratio of >100 mW/K, this pan/sensor combination delivers increased reproducibility in combination with high resolution and high sensitivity.

NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · 95100 Selb
Tel.: +49 9287/881-0 · Fax: +49 9287/881505
at@netzsch.com · www.netzsch.com

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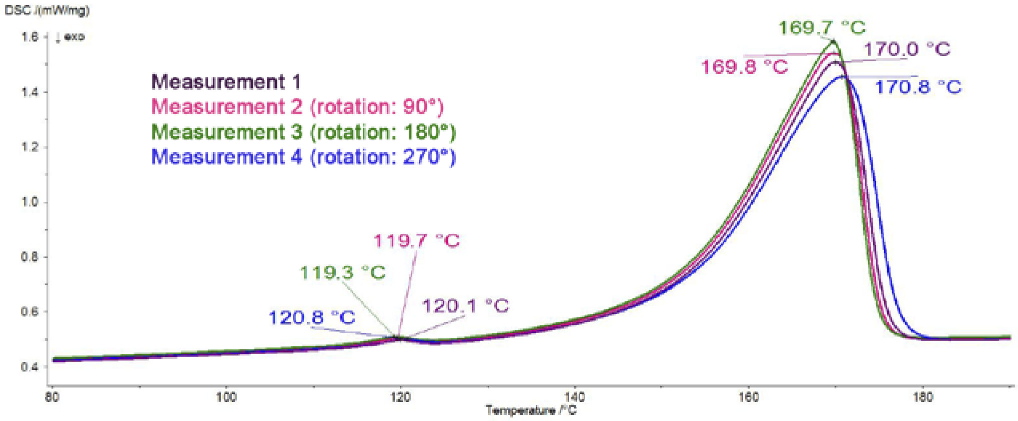
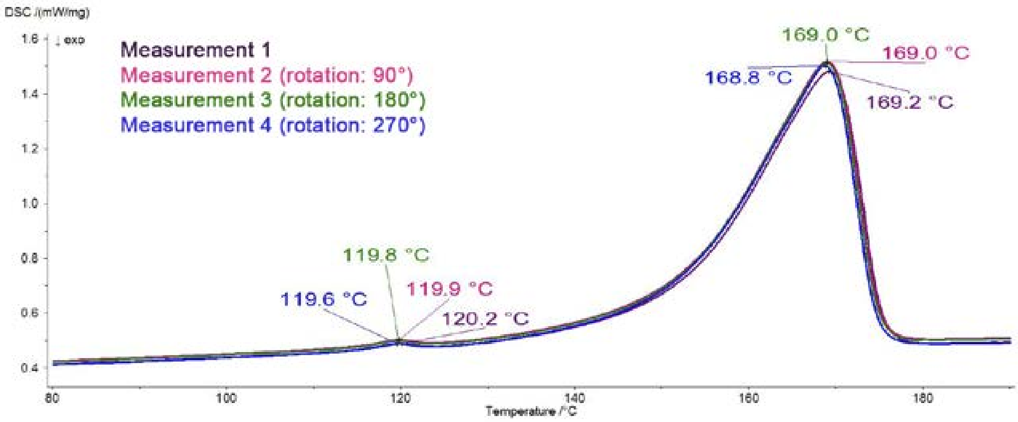
Reproducibility

The high reproducibility of the measurement signal can be seen in figure 2 and figure 3. Two polypropylene samples of the same material were prepared; the first one of 10.94 mg in a standard Al pan, the second one of 11.00 mg in a *Concavus* pan. Each of the two samples was measured four times with the same DSC 214 *Polyma*. Between each of the measurements, the sample was taken out of the furnace, rotated 90°, reinserted in the DSC furance and measured again. All measurements were carried out at a heating rate of 10 K/min between 50°C and 200°C. Two heating runs were performed; the second one was evaluated.

The improved reproducibility provided by the *Concavus* pan can be seen in its curves: they are nearly identical in shape contrary to the ones for measurements in standard Al pans. The standard deviation of the peak temperatures was also calculated: it's only 0.2°C for *Concavus* pans, but 0.5°C to 0.6°C for standard Al pans from both peaks. These measurements clearly demonstrate the superiority of the *Concavus* pan for repeatability purposes. Even rotation of the pans between the measurements has no significant influence on the resulting DSC curve.



2 Polypropylene measurements in a standard aluminum pan



3 Polypropylene measurements in a *Concavus* pan

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4 Packaging of the new *Concavus* pans and lids in the 3in1 Box (on the left) compared to standard aluminum pans and lids packaged bulk-style in containers (on the right)

Packaging

Aluminum pans for DSC analyses are usually purchased in bulk quantities and packaged loosely in containers within a single compartment. Experience has shown that the quality of Al pans packaged in this manner may be impaired by deformations caused by interaction between the pans. NETZSCH has designed the 3in1 Box, a sectioned container with 96 well organized high-quality pans; see figure 4. The 3in1 Box offers advantages such as:

- Storage without contamination
- Save removal of the pan
- Integration of labeling for documentation
- Post-analysis sample storage

The Authors

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 since 2013.

Claire Strasser has been employed as a chemist at NETZSCH-Gerätebau since 2005. Her main focus is on Differential Scanning Calorimetry (DSC).

Conclusion

In choosing the *Concavus* pan for day-to-day work, customers increase their obtainable reproducibility. NETZSCH recommends the *Concavus* pans for any heat-flow DSC instrument, as they offer the best cost-performance ratio on the market.

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