APPLICATION**NOTE**

STA Measurements with Steel Furnace

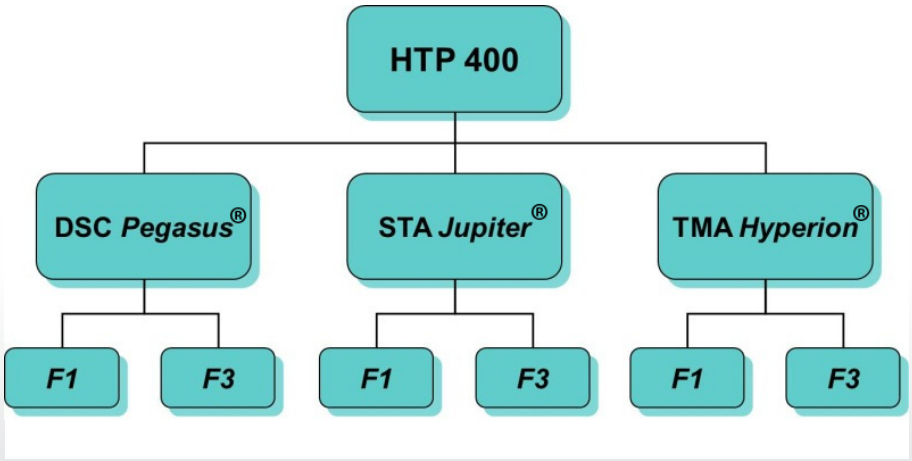
Dr. Ekkehard Füglein and Senol Gezgin

Introduction

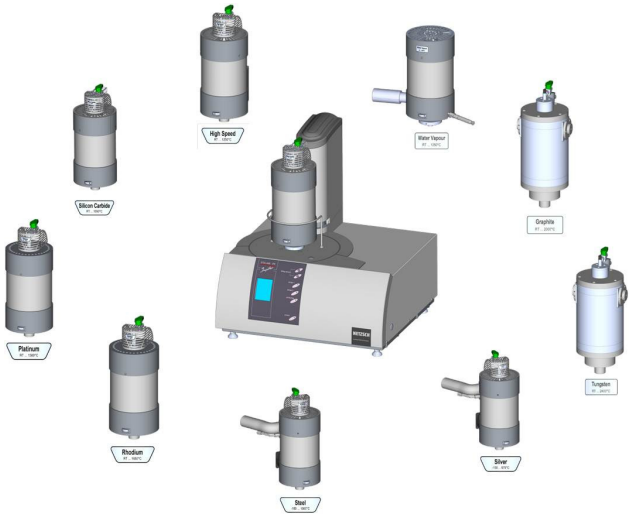
The Platform Concept at NETZSCH-Gerätebau GmbH

Our platform concept is currently comprised of three basic instruments (DSC, STA and TMA), each of which comes in two different models (***F1*** and ***F3***). All electronic components necessary for operating these instruments along with the gas supply unit are contained in a single, integrative housing. The furnaces and sample holders can be quickly and easily exchanged by the operator. This modular setup not only lends a uniform appearance to the instruments, but also capacitates maximum flexibility for adjusting to changing analytical situations and for facilitating the implementation of any consequent modifications necessary in instrument operating conditions. Figure 1 depicts the various instrument versions comprising the platform concept.

A steel furnace is available for all three instrument types. This allows coverage of a temperature range of -150°C to 1000°C at the sample. This application note will discuss measurement results typical in this temperature range for polymers (thermoplastics, elastomers) and crystalline organic substances, such as sugar.



1 High-temperature platform design (HTP) with the DSC, STA and TMA instrument models



2 STA 449 with nine different furnaces

*STA 449 **F1** Jupiter® with Steel Furance*

In addition to the instrument variations mentioned above, a number of add-ons can be supplied for simultaneous thermal analysis (STA), such as coupling methods, *Pulse*TA® or the water-vapor generator. There are presently nine furnace systems available for the STA 449, covering a temperature range of -150°C to 2400°C at the sample (figure 2).

NGB · Application Note 021 · E · 07/12 · Technical specifications are subject to change.

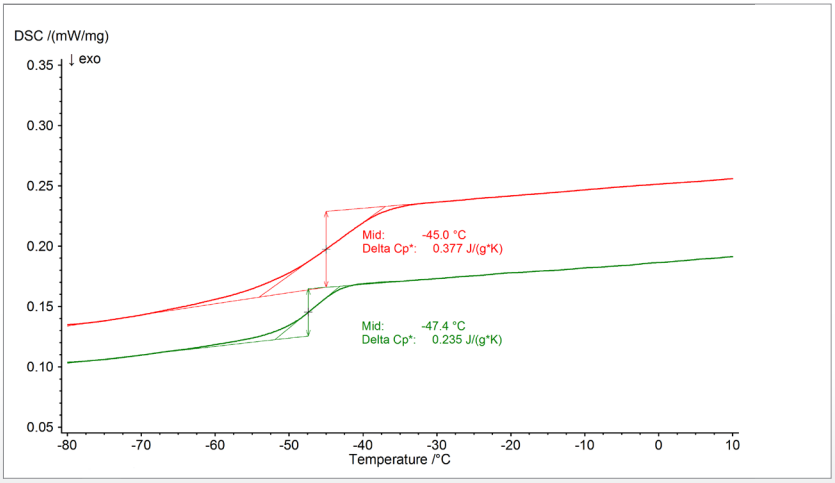
NETZSCH-Gerätebau GmbH
Wittelsbacherstraße 42 · 95100 Selb · Germany
Tel.: +49 9287/881-0 · Fax: +49 9287/881505
at@netzsch.com · www.netzsch.com

Tab 1 Measurement Conditions

	Elastomer	PET	Sorbitol
Measuring instrument	STA 449 ***F3*** *Jupiter®*	STA 449 ***F3*** *Jupiter®*	STA 449 ***F3*** *Jupiter®*
Furnace type	Steel furnace	Steel furnace	Steel furnace
Sample carrier	Octagonal (ASC)	Octagonal (ASC)	Octagonal (ASC)
Thermocouple	P	P	P
Sample Temperature Control (STC)	Off	Off	Off
Cooling parameters	GN_2, auto	GN_2, auto	GN_2, auto
Sample mass	13.493 mg; 12.292 mg	4.945 mg	6.724 mg
Crucible material	Platinum	Platinum	Platinum
Atmosphere	Helium	Helium	Helium
Gas flow rate	70 ml/min	70 ml/min	70 ml/min
Heating/cooling rate	10 K/min	10 K/min	10 K/min

Measurement Conditions

The measurement results for a polymer film made of polyethylene terphthalate (PET), two elastomer samples, and sorbitol – a C6 sugar – are presented in this application note. Standard conditions were employed for all investigations; these are summarized in table 1.



3 Determination of the glass transition temperatures of two elastomers in the temperature range between -80°C and 10°C

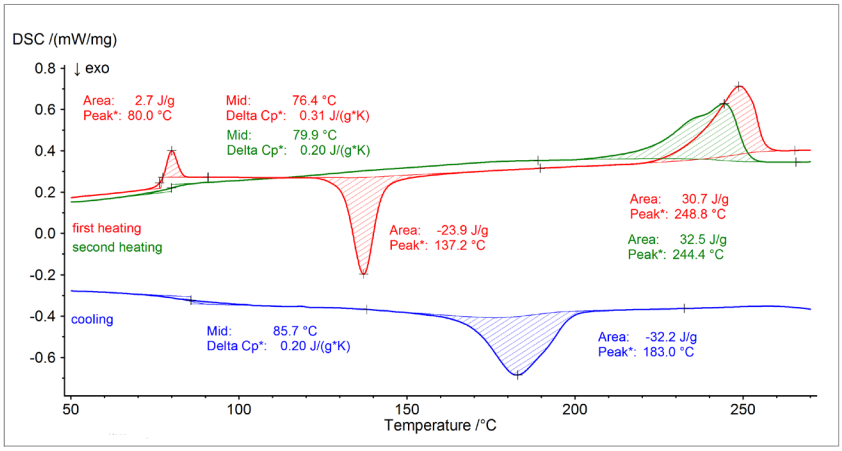
Measurement Results

In order to characterize elastomers, it is necessar to carry out the analyses in a range below room temperature. Since elastomers do not have any crystalline portions, no melting point or melting range exists for these substances. Elastomers are purely amorphous solids; i.e., ones which have solidified in an unstructured way. By means of DSC, however, important information about the material properties can be attained – for example, by determining the glass transition temperature. At this temperature, the mechanical properties of the sample change dramatically. At temperatures below the glass transition temperature (T_g), an amorphous material is brittle and fragile; above the glass transition temperature, on the other hand, it is elastic and flexible. This mechanical property change can be measured very easily with mechanical test methods such as DIL, TMA or DMA. Because the specific heat of a sample also changes during this mechanical property change, a caloric

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

method such as differential scanning calorimetry (DSC) can also be used to determine the glass transition temperature. In the DSC measurement results, the glass transition temperature can be observed as a step; the step height is a direct indication of the change in specific heat, in units of J/gK.

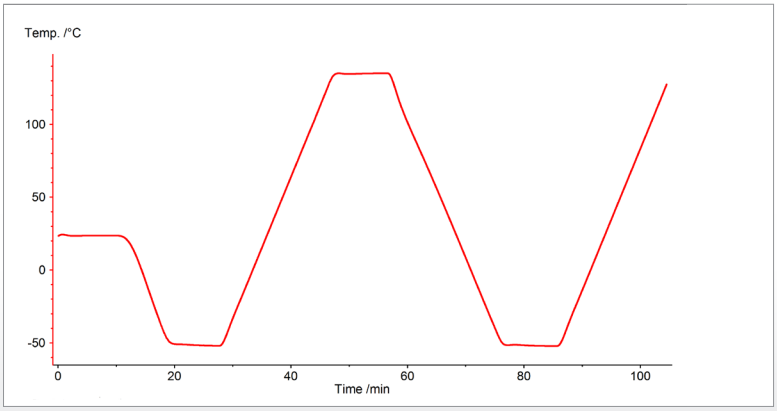
In the investigation of polyisoprene (NR, natural rubber), the glass transition is expected to occur at a temperature of approximately -50°C. This glass transition temperature, however, can vary depending on the rubber mixture and the selection of additives such as plasticizers, and can therefore be adjusted to the corresponding application requirements. Figure 3 shows the results of the determination of the glass transition temperature for two elastomer samples.



4 Measurement results for a polyethylene terephthalate (PET) film, 1st heating (red), 2nd heating (green) and cooling (blue)

For semi-crystalline materials, amorphous regions exist alongside crystalline ones (domains). The amorphous regions are characterized by means of the glass transition temperature as described above, whereas the crystalline regions are characterized by their melting behavior. Since mechanical and thermal treatment steps can alter the ratio of the amorphous to the crystalline region, DSC investigations usually involve the comparison of two heating segments. Between these two heating runs, the samples undergo linear cooling in the DSC instrument by means of a controlled cooling program in order to avoid subjecting the material to any new states of stress. Figure 4 depicts the comparison of these two heating segments (red: 1st heating, green: 2nd heating), along with the cooling segment (blue) that was carried out between the two heating runs.

It can be clearly seen that the transparent PET film was largely amorphous prior to the first heating and that it was characterized by a higher crystalline proportion following the controlled cooling which took place at a rate of 10 K/min.

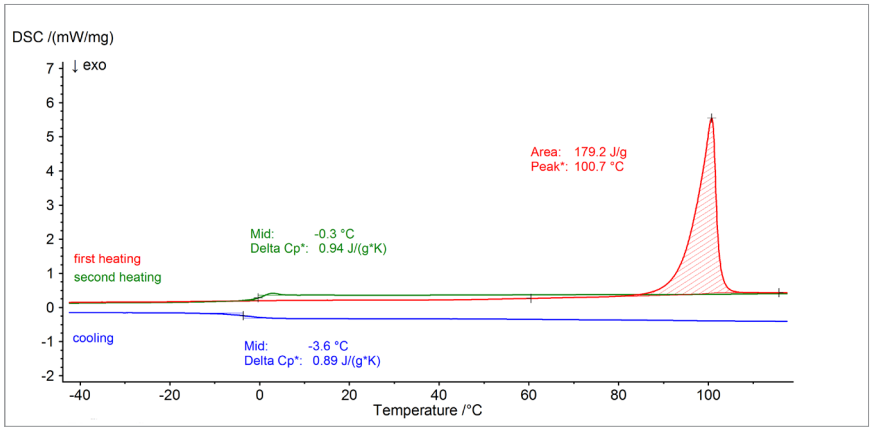


5 Temperature program for the investigation of sorbitol consisting of a heating-cooling-heating series of segments in the temperature range between -50°C and 150°C

A typical temperature-time-profile for such cyclic treatment of a sample is depicted in figure 5 applied for the investigation of sorbitol.

NGB · Application Note 021 · E · 07/12 · Technical specifications are subject to change.

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



6 Measurement results for a sorbitol sample; 1st heating (red), 2nd heating (green) and cooling (blue)

The measurement results for sorbitol are presented in figure 6. The substance was fully crystalline before the investigation, which is why no glass transition was observed during the first heating (red) in the range around 0°C. Sample melting was detected at a peak temperature of 101°C. During the cooling of the liquid sorbitol sample (blue) no crystallization was observed; instead, the sample solidified amorphously, as indicated by detection of the glass transition at -3.6°C (midpoint). During the second heating (green), the glass transition was detected again (midpoint: -0,3°C); by then, the sample was completely amorphous and thus exhibited no melting. The cyclic temperature treatment at heating and cooling rates of 10 K/min caused the sample to change from a completely crystalline to a completely amorphous state.

Summary

The measurement examples demonstrate that even an STA – which is designed primarily for the high-temperature range – is capable of analyzing samples for which a DSC 204 ***F1*** *Phoenix*® or a DSC 200 ***F3*** *Maia*® would normally be used, simply by virtue of changing the furnace.

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