

NETZSCH

Thermomechanical Analysis



Leading Thermal Analysis. ■

TMA 202
-150 °C to 600 °C

Thermomechanical Analysis - TMA

TMA - The method

Temperature dependent dimensional changes in solids, liquids or pastes determine the suitability of that material for certain applications or provide information concerning the composition, structure and conditions of processing.

TMA determines these dimensional changes, whereby the sample can be subjected to an additional mechanical load (DIN 51005, ASTM E 831, D 696, D 3386). Thereby the thermal length change (with negligible mechanical load: dilatometry, DIN 51045) as well as the thermomechanical characteristics are determined, particularly with materials which demonstrate viscoelastic behavior.

What can the TMA 202 do ?

The coefficient of thermal ex-

pansion is an important thermophysical value for any material. In particular, semi-crystalline and amorphous materials demonstrate clear changes in expansion behavior under stress during transformation, relaxation or phase transitions. Linear **expansion**, softening and **penetration** are measured in solids at a constant load which can be set from 1 cN to 100 cN. The expansion and contraction of fibers and films are tested under **tension**. With the aid of expansion containers, the volumetric changes of pastes and liquids can be investigated.

TMA 202 - The concept

After opening the instrument, the user has free access to place the sample. Electronic control eliminates the need for the user to operate the sensi-

tive mechanism which transmits the change in the sample size to the transducer. Consistent preselection of the load is also carried out automatically via the electronic control, with clear digital display.

The TMA 202 for your application

• Polymer development and processing

The TMA 202 shows the dimensional stability of molded parts and their changes during post-crystallization and tension relaxation. Orientation effects, stretching conditions and contraction under stress are measured on fibers and films.

• Development and application of paints and coatings

Penetration tests with the TMA 202 aid in the characterization

of paint compositions and the increase of hardness due to curing or post-curing. They are also important for measurement of the glass transition and the degree of curing.

• Materials research and development

Control and optimization of the required expansion behavior and broadening the scope of application. Transformation behavior of inorganic glass (DIN 52324 and 52328) and amorphous metals. Expansion of fats and waxes during solid-liquid transition. Swelling effects as influenced by moisture and solvents, shrinkage due to drying.

• Development of composite materials

Optimization of the fiber reinforcement and fiber/matrix interaction (expansion behavior and adhesion failure).



TMA 202

TMA 202 - The Technique

Principle of operation

The TMA 202 makes all important adjustments for the user when starting the test:

- the pushrod is automatically moved to the sample
- the measurement system adjusts itself to various sample heights
- the load on the sample is preset in 1 cN increments

Every dimensional change in the sample is transmitted via the pushrod to the highly-sensitive inductive transducer (LVDT) and converted to a digital signal with a resolution of 1.25 nm in the TASC 414. This signal is further processed by the PC.

The inductive transducer can record displacement up to 5 mm, thus allowing the use of **diverse sample geometries and sample holders**.

The furnace can be swung

upwards and allows **unrestricted placement** of the sample on the measuring system. The sample holder and pushrod of fused silica can be easily replaced.

The thermostatic control eliminates the possibility of external effects on the measuring system and furnace.

Versatile Sample Holders

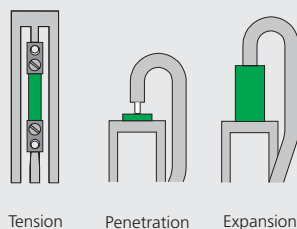
The versatility of the TMA 202 is emphasized by the variability of the sample holder. Thus, the best method for testing the sample can be selected for every application. In addition, it is easy to change the sample holder and the door is open for special customer requests. With the holder used for **expansion measurements**, the expansion and contraction behavior of a sample with parallel ends is recorded exactly. Volumetric expansion can be determined

with the appropriate containers for liquid samples. The load on the sample is usually low (dilatometry).

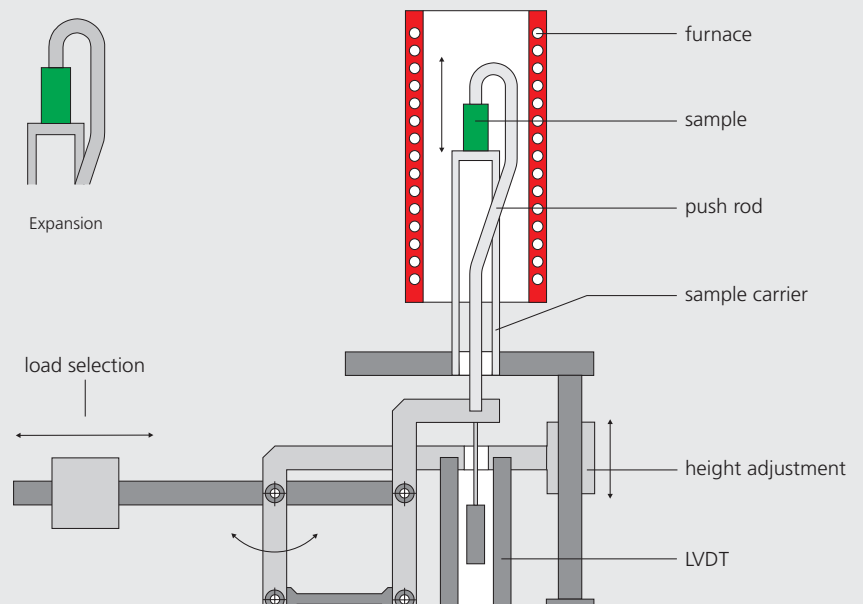
Viscoelastic transitions, the onset of softening and the composition of coatings are investigated at higher specific load in the **penetration mode**. The probe, e.g. with an extended flat tip of 1 mm diameter, penetrates the sample with every change of the elastic characteristics.

Films and fibers can be best tested in the **tensile stress mode**. Clamps on the sample holder and pushrod hold the thin materials securely and reliably transfer the tension to the sample cross section. The long measuring path of the inductive transducer allows suitable sample lengths in this tension mode.

Versatile Sample Holders



Principle of Operation



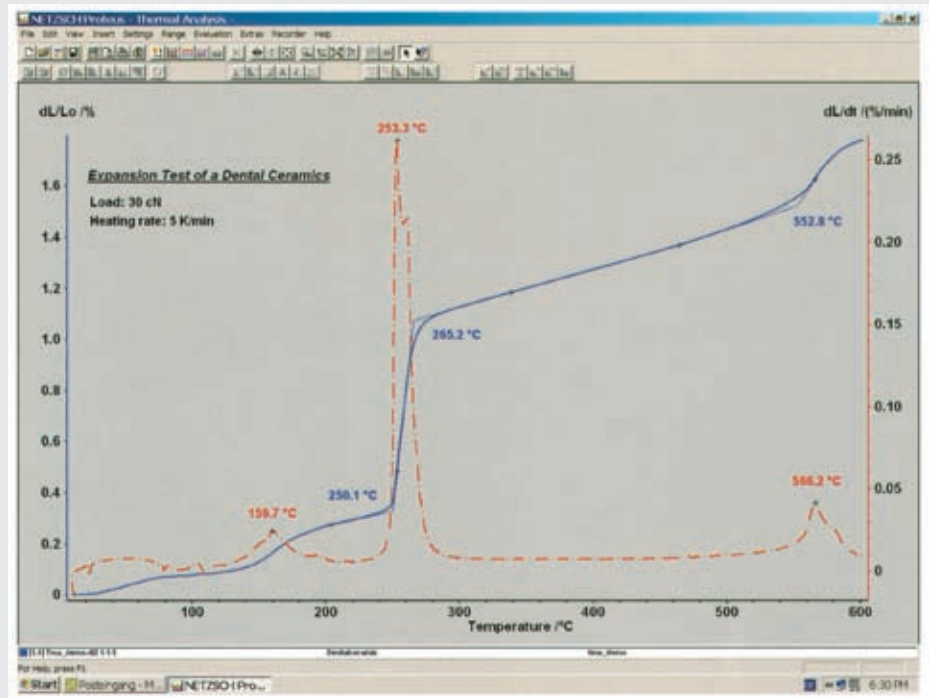
TMA 202 - The *Proteus*® Software

Easily understandable input masks are displayed to help you **program** the test run. The temperature program can contain up to 96 segments with heating, cooling and isothermal sections. Even on-line modifying of the temperature program of an already running measurement is possible. The TASC 414 (Thermal Analysis System Controller) controls the TMA 202 according to your input and transfers the measurement data via the IEEE interface to the PC.

Of course, **multitasking operation** (simultaneous measurement and evaluation) as well as **multi-moduling operation** within the 200 or 400 series (simultaneous operation of several measuring modules) are possible.

All the convenience of the NETZSCH *Proteus*® 32bit Software for Windows® is available to you for **evaluation** in the multitasking mode:

- Unlimited segment selection, zoomed or entire display of up to 32 curves on the screen or output device
- Free selection of the repres-



entation and units for the y axes, e.g. ΔL , $\Delta L/L_0$, derivation, coefficient of expansion

- Picture in picture (PIP) for fading in cut-outs into the whole plot and vice versa (FLIP)
- Correction/calibration of the expansion measurement
- Coefficient of expansion (physical or technical)

- Automated evaluation of characteristic temperatures
- Snapshot: on-line evaluation of the measurement in progress
- Data (Excel® or ASCII) and graphic (Enhanced Meta File) export for further processing and reporting
- Comparative graphics and evaluation with curves from other instruments (e.g. DSC, TG)

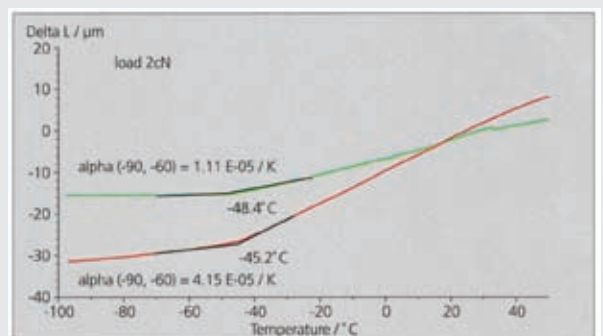
- Electronic manual as integrated context-sensitive HELP system
- Option for automatic read-in of sample length via external digital length caliper
- Option for full-automatic analysis with Macro-recorder software module
- Option for calculated DTA curve (c-DTA®)

Applications

Chloroprene CR

The lowest applicable temperature for rubber materials is determined by means of the glass transition. At temperatures below this range, the rubber becomes glass hard. The expansion curve for vulcanized molded parts of rubber changes its slope at the glass transition point if a small sample load is selected

for the TMA measurement. Thus, as shown here, the **glass transition temperature** can be determined exactly as a standard for the cold resistance. As with the dilatometer, with a small sample load, the **thermal expansion coefficient** can also be easily determined in the TMA (heating rate: 10 K/min).

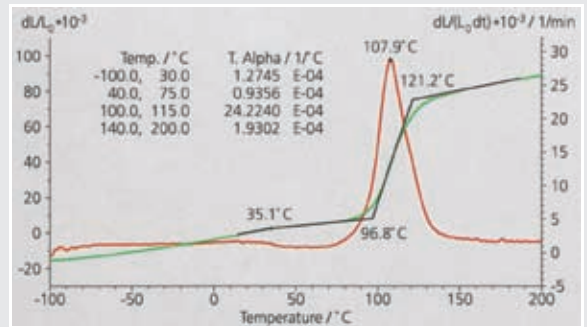


TMA 202 - Applications

Epoxy Resin EP

This figure depicts the thermal length expansion behavior of an EP-based bonding film, characterized by the expansion curve (green) and its derivative with respect to time (red). Softening of the 30 μm -thick sheet, measured in the **tension** mode, begins

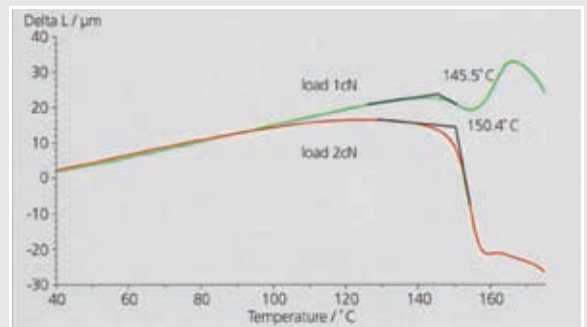
at 97 °C (load: 2 cN; heating rate: 10 K/min). The expansion behavior of the bonding film must be matched to that of the substrate. The technical expansion coefficients (α -values) determined are used for this purpose.



Polycarbonate PC

The glass transition is an important limiting factor for the various areas of application for thermoplastics. With the different loads on the polycarbonate molded parts investigated here, the TMA shows that **softening** occurs long before the glass tran-

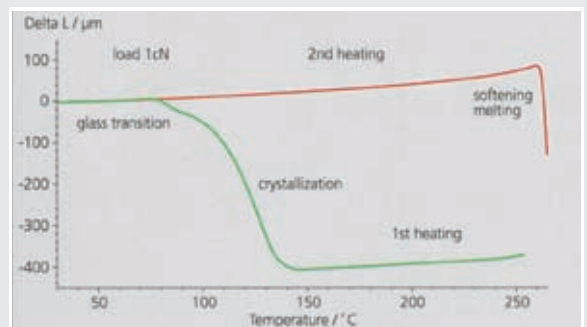
sition temperature has been reached. The pushrod penetrates the sample at varying depths in the transition range, depending on the load. The heating rate in each case was 5 K/min.



Polyethyleneterephthalate PET

The **dimensional stability** of molded parts is very important for the plastics processor and the user as well. If a finished part is not stable after processing because the cycling time was too short, the dimensional changes that occur during post-crystallization can distort the molded part, rendering it useless. This is true especially for semi-crystalline thermoplas-

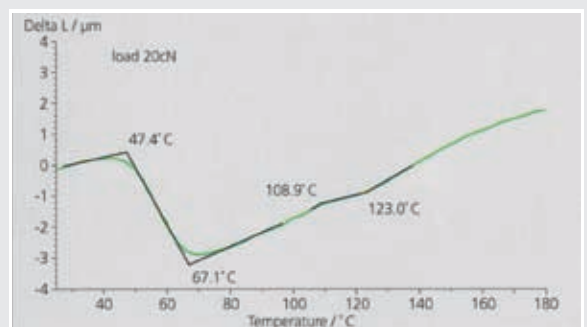
tics that crystallize relatively slowly, like the PET investigated here. During the second heating of the PET, after cooling slowly, no drastic dimensional change is recorded until the onset of melting. The second heating was displayed with the starting point shifted to that of the first heating. A heating rate of 5 K/min was selected in each case.



Two-layered Coating

In the penetration mode with a probe of 1 mm diameter, information can be obtained about the **softening behavior**, even with thin layers. Here the sample was a two-component, PU-based coating on a steel plate protected by a cathodic immersion coat. At a load of 20 cN and a heating rate of 5 K/min, the probe

first penetrates the PU paint and indicates the softening of the cathodic immersion coat during the second, smaller penetration step. The penetration depth and the temperature at which softening begins are important values for the characterization of the **degree of curing** in thermosetting paint systems.



TMA 202 - Technical Data and Worldwide Service

Temperature range:
-150 °C to 600 °C

Heating rates: 0.01 K/min to
50 K/min

Linear measuring range: ± 2.5 mm

Sensitivity: 1 digit/1.25 nm

Load:
0.01 N to 1 N
(1 cN to 100 cN)
in 1 cN increments

Sample holder: fused silica

Pushrod: fused silica

Modes of operation:

- expansion
- penetration
- tension

Sample sizes:
up to 20 mm height
up to 10 mm diameter

Temperature homogeneity:
 ± 2 K
(with max. sample size)

Atmosphere:
inert, oxidizing
static, dynamic

Cooling:
liquid nitrogen, cold gas
cooling jacket integrated
in furnace

Cooling rates:
10 K/min to -140 °C
Cooling time: 600 °C to
-140 °C < 20 min

TASC 414 system controller
for temperature programming
(up to 96 segments) and
control (PID and STC) as well
as data acquisition,
IEEE computer interface.

Digital resolution:
4,000,000 digits

NETZSCH *Proteus*[®]
Software (32 bit)
Windows[®]

Technical data subject to change

Leading Thermal Analysis .

NETZSCH

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