

NETZSCH

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TMA 402
-150...1000°C

Thermomechanical Analysis

TMA - rapid and reliable analysis in the process control, material research and development area.

When materials are heated or cooled their structure, state of aggregation and/or chemical composition may alter. These changes lead to continuous or sudden dimensional changes.

TMA is the generic term for thermoanalytical methods used for measuring mechanical characteristics of a sample, with a constant or oscillating load, and while the sample is subjected to a controlled temperature program.

The fields of application of the TMA 402 are diverse. Due to the high resolution of the inductive displacement transducer and the quality of the components used, measurements are even possible on thin layers without any problem.

The sample can be tested in a vacuum, static or dynamic protective or reactive gas atmosphere.

The change in length or volume of solids or liquids as a function of temperature and load can be measured easily and accurately with the TMA.

Measurements of viscoelastic changes can be made with oscillating load.

TMA is often used for the characterization of elastomers, thermoplastics and thermosets as well as composites, paints and coatings. The technique can also be used for the characterization of glasses, glazes, enamels and other materials like ceramics and metals.

Due to its excellent performance, the TMA 402 is becoming accepted as a standard instrument for the characterization of micro electronic parts.



TMA 402

TMA 402 for investigation in the field of plastics, rubbers, composites, metals, glass and ceramics.

Information on changes in structure, softening and sintering processes as well as on thermal expansion behavior is very important for the determination of the application of materials.

Thermomechanical Analysis provides quantitative information for:

- thermal changes in length or volume
- expansion coefficients
- softening temperatures
- phase transformations
- glass transition temperatures
- sintering steps

That is why TMA is indispensable in many application areas such as control of raw materials, quality assurance during manufacturing and material development and research.

Especially in the range of polymers, varnishes and fiber composites with polymer matrix the TMA is distinguished by a high sensitivity for viscoelastic transitions when working with oscillating sample load.

- Penetration and expansion measurements on partially crystalline polymers clearly show influences of the thermo-mechanical pretreatment, relaxation effects as well as influences on glass transition, crystallization and melting effects resulting from processing.

- Penetration measurements can be used for the optimization of the curing conditions of lacquers and coatings.

- A fast method for checking the quality of hardened or foamed materials is the determination of softening temperatures by penetration measurements.

- Information on the stretching process can be obtained from stretched films and fibers from sudden shrinkage starts as well as from the rate and magnitude of shrinkage.

Software

The TMA 402 runs under a newly developed 32-bit MS® Windows™ software package which includes everything you need to carry out a measurement and evaluate the proceeding data.

It is based on the experience of our Applications Laboratories and the ideas of countless customers over many years. Through the combination of easy-to-understand menus and automated routines, a tool has been created that is extremely user-friendly and, at the same time, allows complicated analysis.

Standard Software Features:

- Windows Software: fully compatible with other MS® Windows™ programs
- multitasking: simultaneous measurement and evaluation
- multi-moduling: operation of up to 4 different instruments with one computer
- combined analysis: comparison and/or evaluation of DTA/DSC, TG and TMA measurements in one plot (up to 32 measurements)
- labeling: input and free placement of text elements
- calculation of 1st and 2nd derivative
- selectable scaling
- graphic and data export
- selectable colors and line types
- storage and restoration of complete analysis
- context-sensitive help system
- temperature calibration
- zoom function

TMA Features:

- various correction options:
 - sample holder expansion can be taken into account with either a calibration measurement or a sample holder correction
 - offset correction
- characteristic temperatures: semi-automatic routines for determination of onset, peak and end temperatures
- glass transitions and softening points:
 - evaluations conform to DIN (German standards)
 - automatic softening point detection / switch off
- expansion coefficients: graphic or tabular presentation of technical and physical expansion coefficients
- Rate Controlled Sintering (RCS) software: optional software for measurements under RCS conditions in 3 different modes: start/stop, stepwise isothermal, dynamic heating rate

Technical Attributes

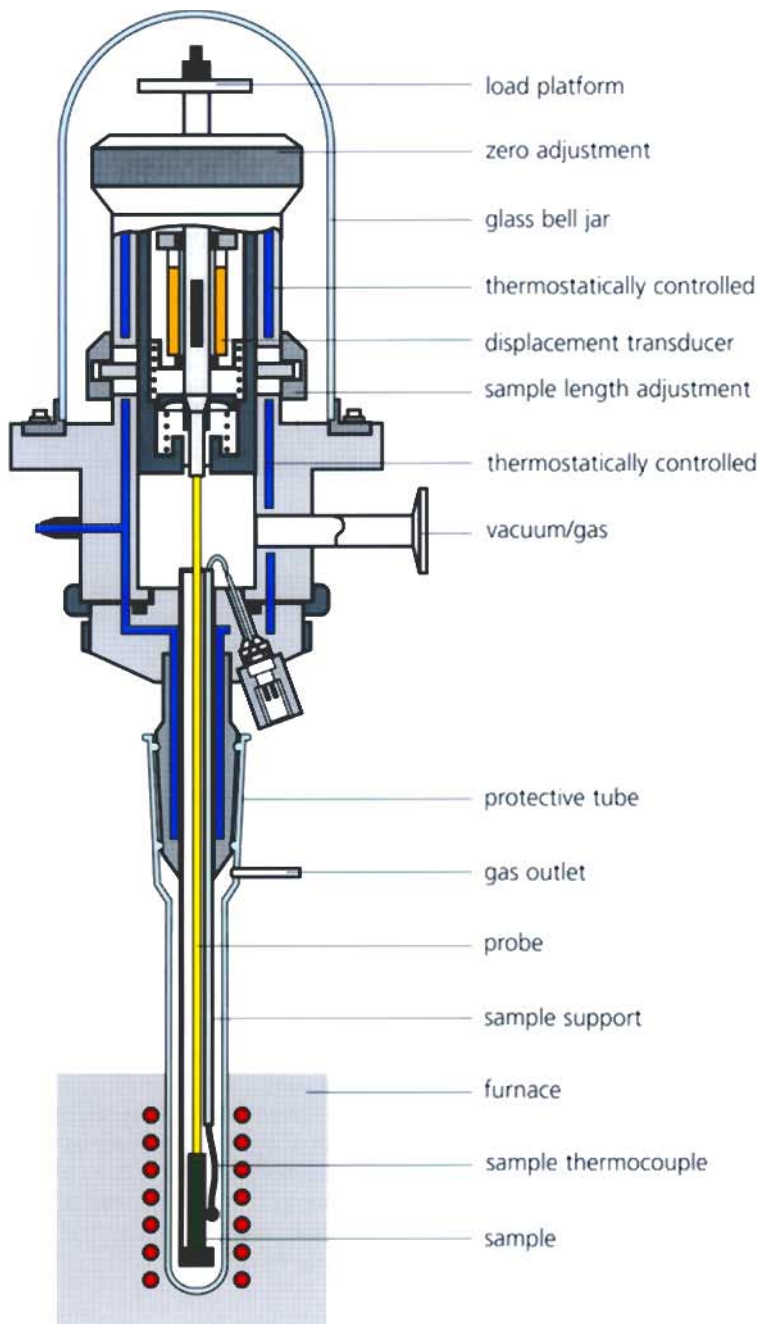
Instrument Components

The TMA 402 is mounted vertically and consists of a linear variable displacement transducer (LVDT), thermostatically controlled, user exchangeable sample carriers and furnaces. Protective gas or vacuum connections, electronic preamplifier for measuring values ΔL (expansion) and T (temperature) as well as thermostat for the reference thermocouples are located directly in the TMA.

Changes in length (or volume) of sample caused by expansion, shrinkage or bending, moves the quartz probe and the ferrite core of the LVDT and causes an electrical signal in its coil. This electrical signal is amplified and recorded as a change in length. The temperature of the sample is measured and recorded by a thermocouple.

Ease of use of the instrument is ensured by:

- the vertical mounting with selectable pushrod load of 0.1 to 200 g
- changeable furnaces for the temperature ranges of -150 to +500 °C and from ambient temperature to 1000 °C. The temperature gradient over 50 mm of sample length is less than ± 3 °C for the furnaces
- measuring the sample temperature close to the sample and recording the change in length directly as a function of the temperature
- easily exchangeable sample carriers and probes
- mechanical adjustment of the measuring head, so that the sample length can be chosen between 1 μm and 50 mm
- accessory for dynamic elasticity measurements



Sample Holders

Sample holder for expansion and penetration measurements. The probe for penetration measurements has an extended flat tip of 1 mm Ø.

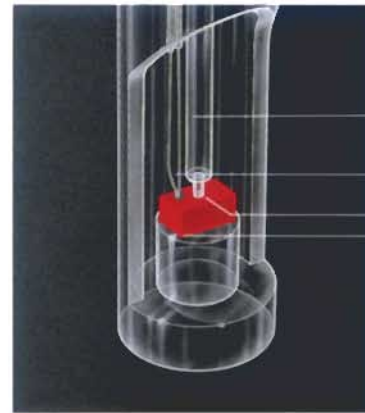
Sample Holder

Sample length:
Sample diameter:
Material:
Thermocouple:
Temperature range:
Optional probes:

The sample lies on a cylindrical body made of the same material as the probe and sample holder.

Standard

50 mm max.
9 mm max.
fused silica
type E or S
-150...500 °C/25...1000 °C
penetration,
dyn. elasticity (max. 250 °C)



Expansion and penetration measurement

Probe
Thermocouple
Penetration tip
Sample

Sample holder for measurements under tension. The sample to be tested (fibers, films) is held between two clamping devices.

Sample Holder

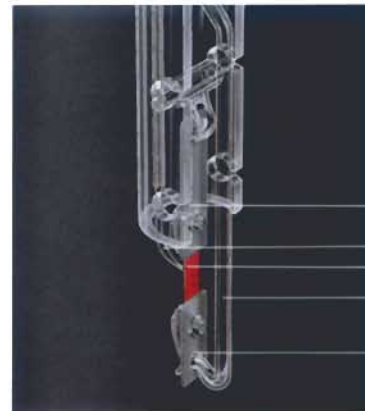
Clamping device:

Material:
Thermocouple:
Temperature range:

The measurement under tensile load is done via the tension probe, through which the load is also transmitted.

Tension Measurement

accessory for different sample geometries (fibers, film) fused silica
type E
-150...350 °C



Measurement under tension

Clamping device
Thermocouple
Sample
Tension probe
Clamping device

Sample holder for bending measurements. The sample is positioned on two edge supports and the bending is

Sample Holder

Sample length:
Sample diameter:
Support distance:
Material:
Thermocouple:
Temperature range:

measured with a knife-shaped probe through which the load is applied.

Bending Measurement I (II)

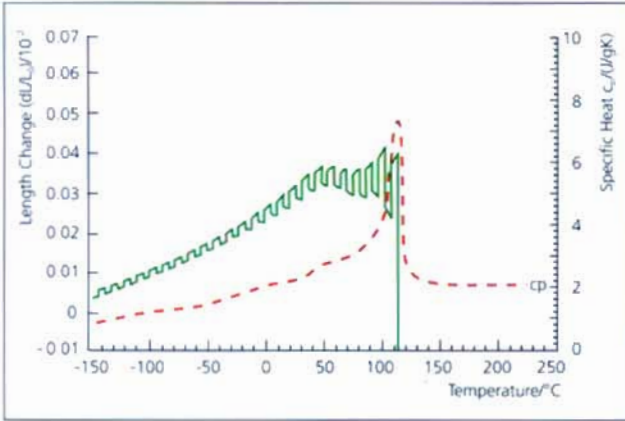
for low-temperature furnace I (II)
16 mm (30 mm)
5 mm max.
12 mm (20 mm)
fused silica
type E or S (Type E)
-150...500 °C (-150...500 °C)
25...1000 °C



Bending measurement

Sample holder
Loading probe
Thermocouple
Sample
Supports

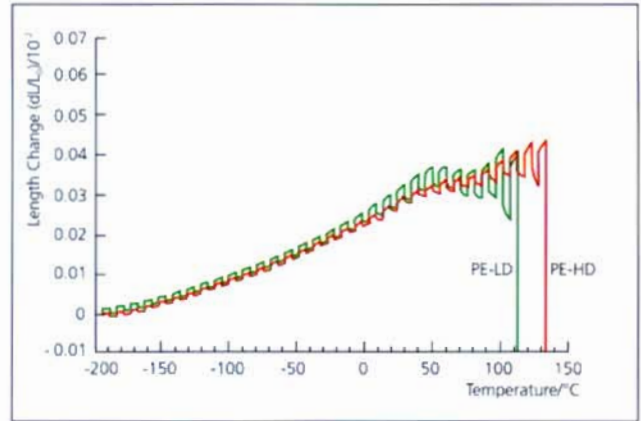
Applications



PE-LD

This plot shows the thermal expansion of PE-LD as a function of temperature while being exposed to a dynamic load in comparison to the curve for the specific heat. The transitions from the pure elastic to the viscoelastic state and the increasing

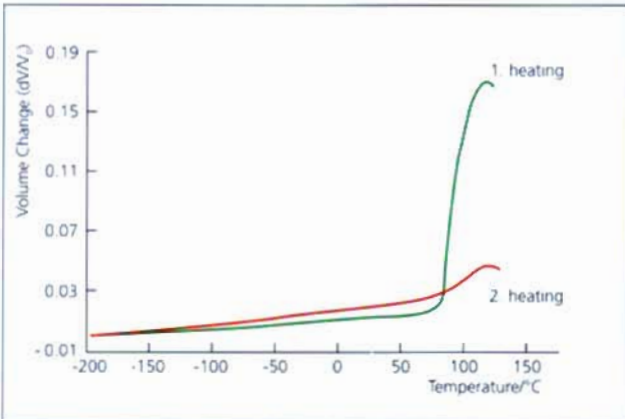
penetration during melting onset are clearly shown in the TMA curve. The good correlation between mechanical and energetic behavior is demonstrated by the comparison with the specific heat curve from a DSC measurement.



PE-LD and PE-HD

This curve comparison clearly depicts the difference in elasticity and melting temperature of PE-LD and PE-HD. This is evident by the

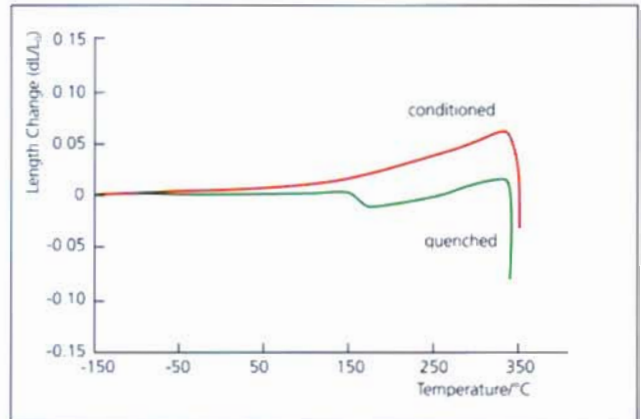
reduced amplitude of the square wave signal and the temperature of complete probe penetration for the PE-HD sample.



Amorphous Polystyrene

This figure depicts relative change in specific volume of amorphous polystyrene. Before the first heating the sample was aged below T_g, while the second heating was conducted on the same

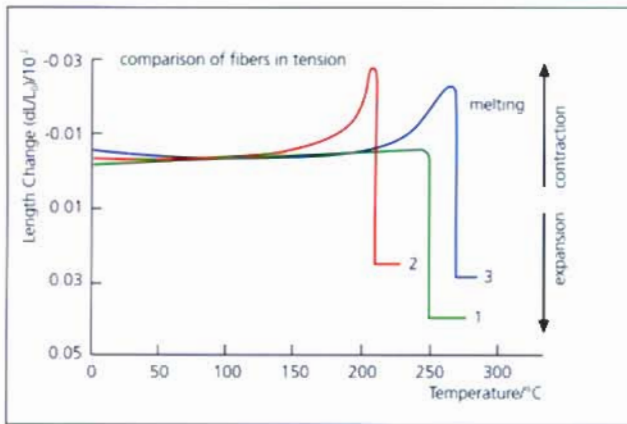
sample after controlled cooling. The volume relaxation for the first heating is clearly visible at the T_g, as well as the change in slope at the T_g for the second heating.



PEEK

The curve comparison of quenched and conditioned PEEK samples shows clearly how thermal history affects the thermomechanical behavior of polymers.

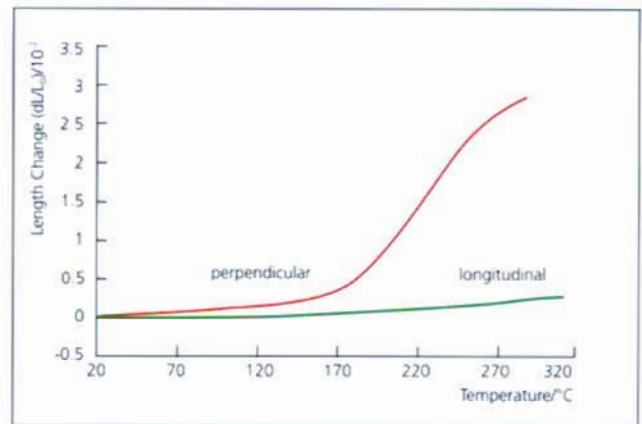
Note the shrinkage due to the cold crystallization in the quenched sample and its disappearance in the conditioned sample.



Polyamide and Polyethyleneterephthalate Fibers

Before melting, fibers no. 2 (PA 612) and no. 3 (PET) show a considerable shrinkage due to stress relaxation. This stress is induced during

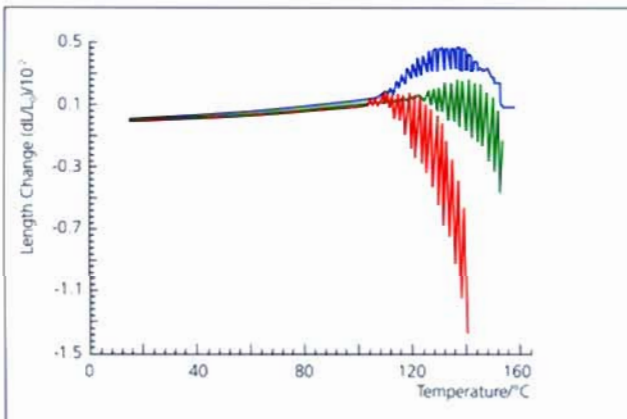
the spinning process of the fibers. Fiber no. 1 (PA 66) is comparably free of stress. Heating rate: 10 K/min load: 10 g, static



Fiber-reinforced Polymer Composite

The difference in the thermally induced expansion of a fiber-reinforced polymer composite is shown here with measurements in the perpendicular and longitudinal direction.

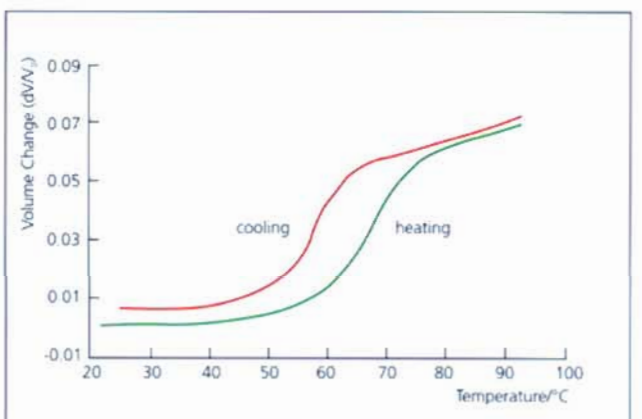
This composite is used in the manufacture of brake linings. The reason for the increased expansion of the perpendicular sample is reduced strength between fiber layers.



PMMA

Three different colored PMMA samples show considerable differences in the viscoelastic range above 100 °C at oscillating load

(10 g) and a heating rate of 5 K/min. These differences are caused by varied copolymerization with other monomers.



Wax

A special sample carrier is necessary for measuring the expansion during melting and the contraction during solidification of waxes. The change in volume of a wax sample of low viscosity being used for investment

casting, is reversible except for an hysteresis during cooling. The relative change in length as a value for the sample volume was measured at a heating rate of 2 K/min and load of 0.3 g.

Technical Data

Measuring Head

Linear range:
 ± 1.25 mm
Measuring ranges:
250, 2500 μ m
Sensitivity:
1 Digit / 0.6125 nm
Load:
0.1...200 cN

Dynamic Loading Mechanism

(optional)
Load range:
0...30 cN
Frequency:
0.03...5 Hz
Signal form:
square, triangular wave

Sample Atmosphere

Static / Dynamic
air, O₂, N₂, inert gases
Vacuum:
= 1 Pa

Low-Temperature Furnace I

Temperature range:
-150...500°C
Heating element:
sheathed wire
Inner diameter:
20 mm
Heating rates:
0.1...50 K/min
Thermocouple:
type E
Dewar vessel for cooling agent

Low-Temperature Furnace II

-150...500°C
Heating element:
sheathed wire
Inner diameter:
34 mm
Heating rates:
1...50 K/min
Thermocouple:
type E
Dewar vessel for cooling agent

High-Temperature Furnace

Temperature range:
25...1000°C
Heating element:
Kanthal A1 wire (3 zones)
Inner diameter:
20 mm
Heating rates:
0.1...50 K/min
Thermocouple:
type K
Water cooling:
1 l/min

Electrical Supply

Low-temperature furnace:
42 V, 6 A
Three zone furnace:
3 x 21 V, 10 A
Voltage: 220 V, 50 Hz
Other voltages
and frequencies on request

Accessories

Cooling gas supply and control unit
for controlled cooling programm;
thermostat for maintaining constant
measuring head temperature.