# ermal Analysis Excellence



# Thermomechanical Analysis

# for All Requirements



# **Outstanding Measurement Performance** Thanks to Swiss Precision Mechanics

Thermomechanical analysis (TMA) is used to measure dimensional changes of a material as a function of temperature. Thermal expansion and effects such as softening, crystallization and solid-solid transitions determine the properties of a material and provide important information about its potential application range. Viscoelastic behavior and curing can be studied by varying the applied force (DLTMA mode).

Features and benefits of the METTLER TOLEDO TMA/SDTA 2+:

- Wide temperature range from -150 to 1600 °C
- SDTA for the simultaneous measurement of thermal effects
- **OneClick™** provides efficient sample measurement
- Nanometer resolution allows very small dimensional changes to be measured
- Dynamic load TMA (DLTMA mode) measures weak transitions and elasticity
- Wide measurement range for small and large samples
- Modular design allows future expansion to meet new requirements
- Hyphenated techniques for evolved gas analysis (EGA) using MS, GC/MS, Micro GC/MS, or FTIR





The TMA/SDTA 2+ incorporates Swiss precision mechanics and is available in four versions with furnace systems optimized for measurements between -150 and 1600 °C.

www.mt.com/ta-tma

# **SDTA Sensor** Outstanding Temperature Accuracy

The TMA/SDTA 2+ is the only instrument on the market that measures the sample temperature very close to the sample in all operating modes, enabling temperature adjustment to be carried out using reference substances (e.g. the melting points of pure metals).

# SDTA sensor



The SDTA signal is the difference between the measured sample temperature and the reference temperature calculated using a model (US Patent 6146013). This means that besides the length change, the simultaneously measured SDTA signal is also available as a measurement quantity. In many cases, this can facilitate the correct interpretation of a measurement curve.

#### DLTMA



The DLTMA mode allows you to study the elastic behavior of samples. In the so-called dynamic load TMA, or DLTMA, mode, the force applied to the sample alternates periodically. This mode is very sensitive to changes in Young's modulus caused by the thermal effects of the sample, such as the glass transition, curing and melting.

#### OneClick™



The patented OneClick function allows you to start predefined measuring methods safely and easily from the terminal at the touch of a button.



The METTLER TOLEDO TMA models have two thermocouples: One measuring the furnace temperature that controls the programed temperature of the experiment, and another that is located next to the sample and measures the temperature of the sample.

#### www.mt.com/ta-calibration

# **Rapid Results** Innovative Solutions

The sampling area is freely accessible for installing the sample holder and measuring probe. These operations can be performed quickly and easily. An indentation ensures that the sample holder can only be installed in one position. The measuring probe is securely attached to the length sensor (LVDT) by means of a magnet and can easily be changed. Different sample holders and measuring probes are available for each measuring mode. This allows you to choose the best configuration for each application.

#### High-precision measuring probe



Swiss quality is also clearly present in the measuring probes and sample holders. We supply the following types of quartz glass probes and sample holders:

- Sample holder for the measurement range 0 to 10 mm
- Sample holder for the measurement range 10 to 20 mm

Wide range of sample holders



A diverse range of sample holders allows for measuring a wide range of materials from different applications areas.

TMA sorption



The TMA Sorption system is designed to analyze materials under a defined temperature and relative humidity (RH). An unique interface allows a humidity generator to be added.



The terminal of the TMA/SDTA 2+ improves ease of use. The SmartSens functionality allows basic operations to be performed without touching the instrument. OneClick function enables you to start predefined measuring methods. All force and length calibration routines are controlled via the terminal.

# **Configurations** Fits Every Need, Even if They Change

The TMA/SDTA 2+ is available is four versions with different temperature ranges. There is an instrument that will fit every possible need. Cooling options include an Intracooler or liquid nitrogen, providing the possibility to test samples at subzero temperatures. The high temperature version can reach 1600 °C, which is perfect for measurements of metals and ceramics. Conversion from one TMA version to another is always possible.





An IntraCooler version that operates from -80 to 600 °C. The IntraCooler is the most effective cooling option on the market that operates without the use of liquid nitrogen.

#### LN/600



A liquid nitrogen cooling option for the low temperature range from -150 to 600 °C.

#### LF/1100 and HT/1600



A standard temperature version for measurements from room temperature to 1100 °C. A high-temperature version for measurements from room temperature to 1600 °C.



The TMA/SDTA 2+ HT/1600 incorporates Swiss precision ceramics and is optimized for measurements between room temperature and 1600 °C.

# Sample Holders Simple, Ingenious and Timesaving

The TMA/SDTA 2+ offers various accessories that enable you to measure samples in different deformation modes. The most suitable mode for your particular application depends on the nature and properties of the sample.



This is the mode most commonly used in thermomechanical analysis. The expansion coefficient is determined as a function of temperature. A typical feature of this mode is that the probe exerts only a very small force on the sample.

#### **Compression mode**

In this mode, the sample is subjected to a large force.



This mode is ideal for studying the elasticity of stiff samples such as fiber-reinforced polymers. Mainly used for DLTMA measurements. Penetration mode



The purpose of a measurement in the penetration mode is to determine the softening point of a sample. This is usually performed using the ballpoint probe.

#### **Tension mode**



The fiber or film accessory is used to perform measurements in tension. This allows you to determine changes in length due to shrinkage or expansion.

#### Swelling



Many substances swell when they come into contact with liquids. The resulting change in volume or length can be measured using the swelling accessory.

#### Volume expansion



Liquids expand just like solids. An accessory enables you to measure volume changes of liquids.



#### Easy sample insertion

The sampling area is freely accessible for installing the sample holder and measuring probe. These operations can be performed quickly and easily. An indentation ensures that the sample holder can only be installed in one position.

www.mt.com/ta-sampleprep

# **Reliable. First-Class Performance** Over the Entire Measurement Range

Types of sample holder	IC/600	LN/600	LF/1100	HT/1600	
0 to 10 mm sample holder, quartz glass	K-type / included with standard equipment		R-type / included with standard equipment	R-type / optional	
10 to 20 mm sample holder, quartz glass	K-type / optional		R-type / optional		
Fiber attachment accessory – set with 1 hook, quartz glass	K-type / optional		R-type / optional		
Film attachment accessory – set with 2 hooks, quartz glass	K-type / optional		R-type / optional		
0 to 10 mm sample holder, aluminum oxide	-		optional	R-type / included with standard equipment	
Megouring proboo	10/600	10/600	15/1100	UT/1600	
	10/000	LN/000		11/1000	
Measuring probe, ball-point, 3 mm, quartz glass	inc	luded with standard equi	pment	optional	
Measuring probe, ball-point, 3 mm, aluminum oxide	_		optional	included	
Measuring probe, flat, 3 mm, quartz glass	optional (can be used up to 1100 °C)				
Measuring probe, flat, 1.1 mm, quartz glass	optional (can be used up to 1100 °C)				
Measuring probe, knife-edge	optional (can be used up to 1100 °C)				
3-point bending accessory	optional (can be used up to 1100 °C)				
Swelling accessory	optional (can be used up to 1100 °C)				
Volume expansion accessory	optional (can be used up to 1100 °C)				

#### Defined furnace atmosphere, programmable gas flow and gas switching.

The furnace chamber can be purged with a defined gas. This process is software controlled, which makes it very easy to switch from an inert atmosphere to reactive conditions. The standard gas controler can be upgraded to a GC 302 or GC 402 for improved functionality and atmosphere control.





#### Key

- 1. Water cooling
  - 2. Parallel guidance with bending bearings
  - 3. Adjustment weight
  - 4. Transformer (LVDT)
  - 5. Force generator
  - 6. Height adjustment

- 7. Thermostatted measuring cell
- 8. Sample support
- 9. Measuring probe
- 10. Sample temperature sensor
- 11. Water-cooled furnace jacket
- 12. Furnace heating

Extremely precise mechanical system based on internationally acclaimed METTLER TOLEDO balance technology. Thanks to this development, the measuring probe can move up and down perfectly free from any frictional forces. The force applied is therefore extremely accurate.

# **Thermomechanical Analysis** For All Kinds of Materials

The TMA/SDTA 2+ can be used for a wide range of applications due it its broad temperature range and the wide choice of force parameters in compression and tension modes. As a result, the TMA/SDTA 2+ quickly provides characteristic information on numerous types of samples, for example very thin layers, large sample cylinders, fine fibers, films, plates, soft or hard polymers and single crystals.

TMA is the ideal addition to DSC. Besides the measurement of expansions coefficients, TMA is also an excellent technique for determining glass transitions that cannot be satisfactorily measured by DSC, for example materials with a high filer content. The penetration mode is ideal for characterizing the glass transitions of difficult samples such as very thin coatings.

#### Effects and properties that can be characterized using the TMA/SDTA 2+ system

- Viscoelsatic behavior (Young's modulus)
- Glass transition
- Expansion coefficient
- Expansion and shrink of fibers and films
- Softening
- Viscous flow
- Melting and crystallization

- Gelation
- Phase transitions
- Curing and crosslinking reaction
- Swelling behaviour
- Volume expansion
- Thermal effects of pharmaceuticals and foodstuffs





Thermomechanical analysis (TMA) is used to measure the dimensional changes of a material as a function of temperature. TMA allows you to determine expansion coefficients and softening temperatures, as well as measure relaxation effects that are often not detected by other thermal analysis techniques.

#### www.mt.com/ta-applications

#### **Delamination of composites**



A printed circuit board (PCB) is a laminate consisting of several layers of glass fibers embedded in a thermosetting resin matrix. Important characteristics of PCBs are their glass transition temperature ( $T_g$ ) and temperature stability. The diagrams show TMA curves of two different PCBs. The changes in the slope of the curves at 93 and 122 °C correspond to the  $T_g$  of the PCBs. Decompositions of the resin matrix is accompanied by outgassing. This forces the layers apart (delamination) and leads to spikes in the TMA curve. The curves show that PCB1 is more stable than PCB2.

#### Creep behavior of elastomers (TMA Mode)





An important property of a seal is its creep and recovery behavior. Creep deformation consists of reversible viscoelastic relaxation and irreversible viscous flow components. In this application, several styrene-butadiene rubber (SBR) samples with different degrees of vulcanization were investigated. Unvulcanized SBRO shows the largest elastic deformation (left arrow) and the largest irreversible deformation (right arrow). With increasing vulcanization, both the elastic deformation and the viscous flow decrease. Good sealing materials should not exhibit viscous flow.

#### Sintered high-performance ceramics





High-performance ceramics exhibit high temperature stability. This is shown here with measurements of two sintered  $SiO_2$  samples – a conventional  $SiO_2$  (Sample 1) and a second type (Sample 2). The cristobalite transition of Sample 1 can be clearly seen at 245 °C. This occurs rapidly and often leads to cracks in the material. Sample 2 exhibits a slower quartz transition at a higher temperature, with less risk of crack formation. Sample 2 also contains crystallization nuclei; crystallization occurs from about 1200 °C onward. These properties make Sample 2 a high-performance ceramic.

#### Moisture induced curing





Using the TMA sorption option together with dynamic load TMA (DLTMA), the behavior of a moisture induced curing reaction was measured. The sample was held at 90% RH at an isothermal temperature of 30 °C with an oscillating force of 10 mN. As curing occurs, the viscosity of the sample increases until it reaches a constant level (after about 200 minutes). The upper envelope of the DLTMA curve has two distinct curing steps with onsets at times T1 and T2 before reaching a near constant value. The lower envelope curve has only one distinct onset at time T3 before approaching the upper envelope curve.

#### Solid-solid transitions by TMA and DSC



Solid-solid transitions are generally accompanied by volume changes. Transitions like this are observed as steps in the TMA curve. This is demonstrated here by using a single grain of ammonium nitrate, a substance used in many fertilizers and explosives. The curves show that the structural changes occur quite rapidly. The transition temperatures depend on internal stresses of the sample related to its thermal history. This explains the different shapes of the curves measured in the first and second hearing run. The DSC curve (second heating run) is shown for comparison.

#### **CTE** determination



The coefficient of thermal expansion (CTE) can be determined from TMA measurements in the DLTMA mode. The diagram shows dilatometric curves and the resulting expansion coefficients of three different materials. Borosilicate glass has a CTE of about 3.3 ppm in the glassy state and a glass transition at about 550 °C. Invar is a iron-nickel alloy, which shows practically no thermal expansion up to 150 °C. Crystalline  $\alpha$ -quartz expands with a continuously increasing expansion coefficient. A solid-solid transition to  $\beta$ -quartz occurs at about 575 °C. On further heating, the sample then starts to shrink.

#### Swelling of elastomers



The swelling behavior of sealants in solvents is often important for their practical use. Swelling behavior can be measured using a TMA/SDTA 2+ equipped with a special swelling accessory. The diagram shows the swelling curves of four different elastomers in toluene at 30 °C. A fluoroelastomers (FPM) swells only about 2% in one direction. FPM is clearly resistant to toluene and can be used as a sealant when it is exposed to this solvent. The other three elastomers swell significantly more, for example silicone rubber (MQ) swells by more than 35% in one direction in 35 minutes.



#### Curing of an epoxy resin by DLTMA

A precured epoxy resin was measured by DLTMA. In the glassy state, the resin is hard and the displacement amplitude with the alternating load used is small. The amplitude increases at the glass transition. After this, the resin becomes liquid and beings to flow; the amplitude remains constant but then decreases at about 190 °C due to curing of the resin. The curing process can also be seen as an exothermic peak in the simultaneously measured SDTA curve. In the second DLTMA heating run, the glass transition of the fully cured sample is observed at about 110 °C.

# Simple, Intuitive Operation Straightforward, Efficient and Secure

STAR<sup>e</sup> software has been expanded to include new features that help you prepare your TMA/SDTA 2+ instrument for specific experiments, develop methods for advanced analyses and perform flexible result evaluations. Complex measurement programs are set up within minutes and the vast range of available tools permit curves to be evaluated both accurately and efficiently.



#### DLTMA



All records are stored and linked in a secure, protected data archive, shielding raw data from unintentional deletion or modification. The database can easily be filtered by sample name, date, time, user, project name, instrument used, and more.

# Elatic behavior of a laminate

The powerful STAR<sup>e</sup> software can be used to evaluate Dynamic Load TMA (DLTMA) measurements. Detailed information about viscoelastic properties (Modulus) is determined with great sensitivity.

#### CTE – Dilatometry



The most common use of a TMA is to determine the coefficient of thermal expansion (CTE). S**TA**R<sup>e</sup> has many different functions to calculate and evaluate CTE, which will provide the most relevant results.

#### Complete Thermal Analysis System





A complete thermal analysis system consists of the basic six complementary measuring techniques, each of which bring fast and accurate results. Additional knowledge can be obtained by means of several hyphenated techniques.

#### www.mt.com/ta-software

# World-Class Service and Support Provide Results You Can Trust

METTLER TOLEDO's portfolio of services is designed to ensure the continuous performance and reliability of your thermal analysis systems. Factory-trained in Switzerland, our worldwide teams bring the professional expertise and know-how needed to provide you with the highest level of after-sales support, as well as the experience necessary to optimize services for your own particular needs.





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#### TMA/SDTA 2+ Specifications

Temperature data	LF/1100	HT/1600	IC/600	LN/600	
Temperature range	RT to 1100 °C	RT to 1600 °C	–80 to 600 °C	-150 to 600 °C	
Temperature accuracy (RT to max. temperature)	±0.25 °C	±0.5 °C	±0.25 °C	±0.25 °C	
Temperature accuracy (–70/–100 °C to RT)	n.a.		±0.35 °C	±0.35 °C	
Temperature accuracy (–150 to –100 °C)	n.a.		n.a.	±0.5 °C	
Temperature reproducibility	±0.15 °C	±0.35 °C	±0.25 °C	±0.25 °C	
Heating (RT to max. temperature)	8 min	22 min	< 6 min	< 6 min	
Heating (-70/-150 to 600 °C)	n.a.		< 7 min	< 6 min	
Cooling (max. temperature to RT)	20 min	< 40 min	13 min	< 15 min	
Cooling (RT to -70/-150 °C)	n.a.		22 min	15 min	
Length data					
Maximum sample length	20 mm				
Measurement range	±5 mm				
Resolution	0.5 nm				
Noise (RMS)	5 nm				
Reproducibility	±100 nm	±300/±500 nm (1100/1600 °C)	±100 nm	±50 nm	
Force data					
Force range	-0.1 to 1.0 N				
DLTMA data					
Frequencies	0.01 to 1 Hz				
SDTA®-(Single differential Thermo	al Analysis)				
SDTA resolution	0.005 °C				
SDTA noise (RMS)	0.01 °C	0.01 °C	0.02 °C	0.02 °C	
SDTA sensor type	R type K ty			уре	
SDTA signal time constant	33 s	33 s	38 s	38 s	
Data sampling					
Sampling rate	max. 10 data points per second				

IEC/EN61010-1, IEC/EN61010-2-010 CAN/CSA-C22.2 No. 61010-1-04 & -2-010 IEC61326-1 / EN61326-1 (class B) IEC61326-1 / EN61326-1 (Industrial requirements) FCC, Part 15, class A AS/NZS CISPR 11, AS/NZS 61000.4.3 Conformity Mark: CE, CSA, C-Tick

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