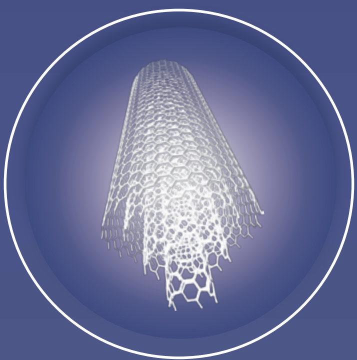
Advanced Materials

High Temperature Thermal Analysis and Calorimetry Solutions



calvet 3D inside

SETARAM Instrumentation
KEP Technologies

Inspiring Imagination for Material Science

Sintering techniques were first used for the preparation of ceramics, but are now also used for the development of high value alloys. Precise knowledge of a reliable and reproducible temperature profile is essential to the development of a quality sintered product. TMA allows for the measurement of sintering of even the finest powders under controlled atmospheres and non-linear and reactive temperature profiles.

Oxidation and corrosion of materials in highly demanding applications is critical for long term performance of materials used in everything from re-entry vehicles to nuclear reactors and power plants. Using TGA under corrosive atmospheres, and with the ability to measure long isotherms it is possible to quantify the materials performance.

Heat capacity (Cp) is one of the critical properties of a material that needs to be assessed. Cp can be estimated by DSC, however 3D Calvet based techniques provide higher precision measurement of Cp.

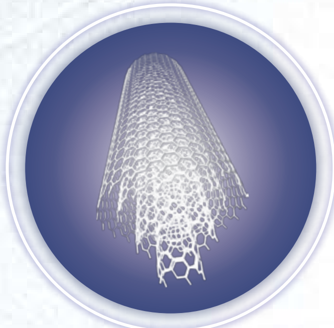
For high temperature Cp measurement drop calorimetry is the most accurate alternative.

Phase diagram and formation enthalpies of alloys and oxides are also essential thermochemical data for the understanding of materials compatibility and reactivity.

They are obtained by using high temperature calorimetry in DSC or drop mode.

During the development of Advanced Materials it is essential to understand production challenges as well as to characterize the performance of a material under the most challenging and demanding experimental conditions. Setaram presents a comprehensive range of techniques and instrumentation for the Advanced Materials laboratory with the ability to quantify all types of behavior, including dimensional, mechanical, mass variation and phase changes under the most demanding of experimental conditions. Only Setaram can offer temperatures down to -196 °C and up to 2400 °C and also the ability to work under corrosive, oxidative, controlled humidity and high vacuum atmospheres.





Advanced Materials

Our Products

The **LABSYS evo** system is a highly flexible TGA / STA (Simultaneous Thermal Analysis) system that also offers a unique 3D Calvet based Cp rod for materials characterization up to 1600 °C. This highly flexible system features a powerful furnace, a thermostated balance designed specifically for thermal analysis applications and plug and play rod connectors for ease of use and rapid configuration for different applications.

LABSYS evo

- Ambient to 1600 ° C
- 0.01 to 100 °C/min
- 0.2 µg, 0.02 µg balance resolution
- 20 g maximum balance capacity
- Up to 3 carrier gases
- TGA, DSC and DTA interchangeable rods
- 3D Cp rod for Cp measurements within 2 %

The **SETSYS evolution** is a high performance TGA / STA (Simultaneous Thermal Analysis) and TMA system designed for applications that demand the most challenging of experimental conditions. The heart of the system is Setaram's unique symmetrical balance beam that allows for the direct suspension of even large sample sizes and a highly robust furnace that can operate all the way to 2400 °C and under the most aggressive atmospheres, from high vacuum, high humidity, reductive and oxidative atmospheres.

SETSYS evolution

- Ambient to 2400 °C
- 0.01 to 100 °C/min

TGA / STA (Simultaneous Thermal Analysis)

- 0.03 µg / 0.3 µg balance resolution
- 35 g / 100 g balance capacity
- TGA, DTA and DSC interchangeable rods

TMA

- Resolution to 0.2 nm
- Zero force TMA capability
- Controlled rate sintering

TAG

The **TAG** system from Setaram is the world's most precise isothermal TGA system. It features the same symmetrical balance beam and aggressive atmosphere control seen in the SETSYS Evolution system. In addition it incorporates a pair of matched furnaces to achieve a truly symmetrical performance and therefore eliminate buoyancy effect.

If you are studying long term corrosion and/or oxidation then nothing comes close to the TAG's performance.

- Ambient to 1600 °C / 1750 °C
- 0.01 to 100 °C/min
- 35 g balance capacity
- TGA, DTA and DSC interchangeable rods

The **96 LINE** is a powerful modular system for the precise characterization of materials up to 2100 °C using drop-calorimetry, DSC, DTA, TGA and TMA. The system is unique in that each module can use large, even bulky and heterogeneous samples to give data about materials in real life conditions.

- Ambient to 2100 °C
- TGA - samples up to 100 g
- DSC - crucibles up to 450 µl
- Drop calorimetry - crucibles up to 5700 µl
- TMA - samples up to 18 mm diameter

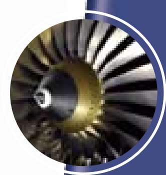
96 LINE

Setaram offers a range of 3D Calvet High Temperature Calorimeters that are used for applications such as high precision Cp measurement and the study of alloy and oxide formation using drop techniques.

The **NEW AlexSys** high temperature in-situ reaction calorimetry system is designed to measure heat of formation, heat of solution, Cp and other characteristics of actinides, oxide melts, and other materials. Its unique gas mixing system removes the need for mechanical stirring.

- Up to 800 °C or 1000 °C
- Vessel volume - 28 ml (quartz cell) or 20 ml (platinum crucible)

ALEXSYS



C600 calorimeter is available for calorimetric measurements up to 600 °C.

- Up to 600 °C
- Cell Volume 8.5 ml

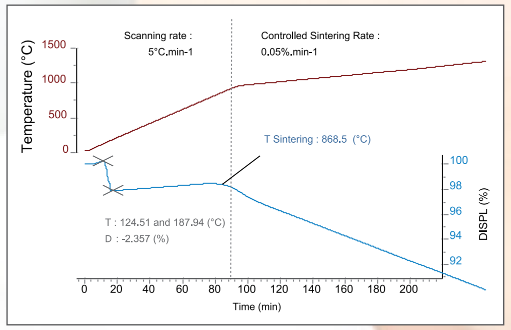
Applications

TMA/Dilatometry

Low load and controlled SINTERING using SETSYS Evolution TMA

The vertical setup of SETSYS Evolution TMA allows applying loads as low as 1 g. In the following example, a polyethylene-coated metallic powder was tested under different loads, in the controlled sintering mode. This mode consists in controlling the temperature of the sample so that sintering happens at a set rate.

The thermogram below shows the heating of the sample between 25 °C and 1350 °C at 5 K/min, with a set load of 2 g. The sintering rate is set to 0.05 %/min. PE starts to melt at 124 °C, leading to a shrinkage of 2.36 %. The sintering starts at 868.5 °C.



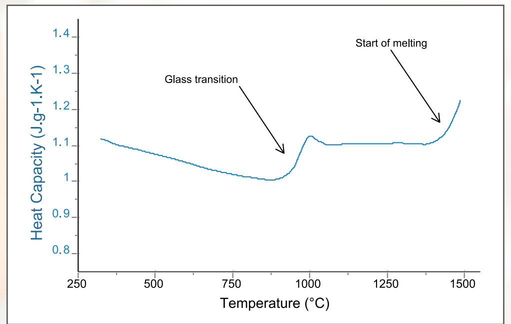
The same test with 20 g load leads to an irreversible deformation of the sample

High Temperature Calorimetry

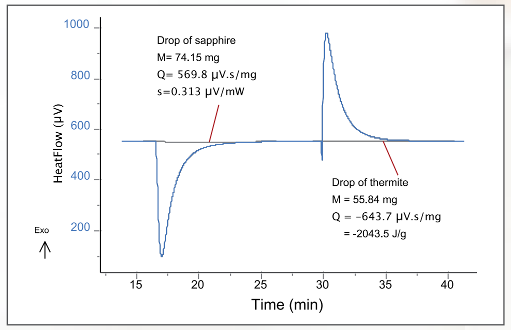
Heat capacity (Cp) determination of glasses with MultiHTC (HF-DSC)

Together with the other potassium feldspars, orthoclase ($KAlSi_3O_8$) is a common raw material for the manufacture of glasses, ceramics (such as porcelain) and is a constituent of scouring powder. Some intergrowths of orthoclase and albite have an attractive pale luster and are called moonstone when used in jewelry.

A large sample (1040.22 mg) of orthoclase was heated from 300 °C up to 1500 °C at 10 °C/min in a multiHTC HF-DSC sensor. A blank and a reference tests then allowed calculating the heat capacity of the mineral, and detecting its glass transition temperature (972.8 °C) and melting (1 451.7 °C).



Heat of thermite reaction with MultiHTC (drop cell)



During a thermite reaction aluminum reacts with an oxide of another metal, most commonly iron oxide Fe_2O_3. Although the reactants are stable at room temperature, when exposed to sufficient ignition heat, they burn with an extremely intense exothermic reaction and as a result aluminum oxide Al_2O_3 and iron are formed.

Drop calorimetry is used to investigate this reaction: pellets of Fe_2O_3 + 2Al are dropped into a boron oxide crucible, placed at 1550°C, under an inert atmosphere. Calibration is done with sapphire.

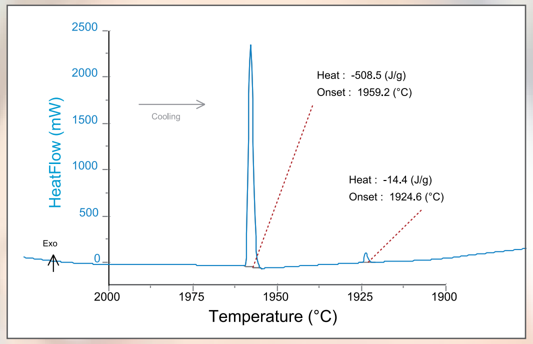
Knowing the heat of formation of Al_2O_3 and heat capacity of Fe_2O_3, the enthalpy of the thermite reaction can be easily determined.

High Temperature DTA

Phase diagrams in a wide temperature range: crystallization of a high-end application ceramic

Yttrium aluminum garnet (YAG), in its neodymium-doped form ($Nd{:}Y_3Al_5O_{12}$), is widely used in solid-state lasers. Nd- doped YAG lasers have biomedical applications in photocoagulation for patients suffering from ulcers, in correction of complications linked with cataract surgery, or in removal of skin cancers.

They are also used for engraving, etching, or marking a variety of metals and plastics. They are extensively used in manufacturing for cutting and welding steel and various alloys.



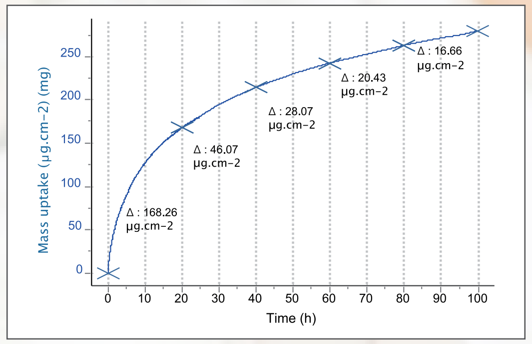
In the present example, a powdered sample of $Nd{:}Y_3Al_5O_{12}$ was placed in a tungsten crucible. It was heated up to 2100 °C under helium flow in a SETSYS Evolution equipped with a high temperature furnace and tungsten DTA rod. Then it was cooled down at a controlled rate of 20 K/min. The thermogram exhibits two crystallization peaks above 1900 °C.

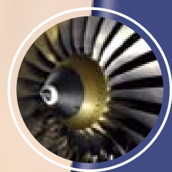
High Temperature TGA

High temperature oxidation / corrosion

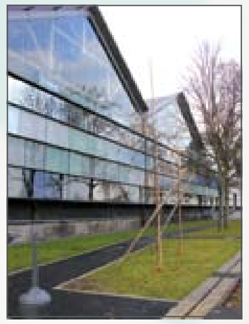
The symmetrical thermoanalyzer TAG was used to study the resistance of a metallic plate to corrosion at high temperature.

The sample weight is measured under an oxygen flow (30 ml/min) at 1100 °C during a 100 h experiment. The observed mass increase is due to surface oxidation of the sample. The outstanding long term stability of TAG thermobalance allows recording a very clear signal, even in the case of small mass change over a long period of time.





Customer testimonial



«At Saint-Gobain central research centres (in France and USA) - where we study glass, ceramics and other high performance materials - we have set up a laboratory to study the thermal behaviour of the various raw materials we use as well as our finished products. Our goal is to accurately characterize variations in CP (glass transition), crystallization, melting and/or any other phase transformation experienced by our materials during heating. The unique design of the 3D sensor of the Multi HTC Line 96 has enabled us to study high temperature phenomena with greater precision. SETARAM answers our needs in terms of reliability and robustness and provides us with the technical support necessary for the development of procedures customized for our specific materials.»

Sophie PAPIN - Groupe Minéralogie Appliquée
Service Expertises Analyses Réfractaires- SAINT-GOBAIN RECHERCHE - France

Some International references

Air Liquide - France
Alstom - Switzerland
BAM (Bundesanstalt für Materialprüfung) - Germany
BARC (Bhabha Atomic Research Centre) - India
Brookhaven National Laboratory - USA
Cambridge University - UK
CEA Saclay - France
Chimie ParisTech - France
CIEMAT - Spain
CMTR (Chimie Métallurgique des Terres Rares) **CNRS** - France
Cogne Acciai Speciali - Italy
CSIC - Instituto Cerámica y Vidrio - Spain
CSIRO - Australia
CTTC (Centre de Transfert de Technologies Céramiques) - France
Endesa - Spain
ICMMO (Institut de Chimie Moléculaire et des Matériaux d'Orsay) - France
IFP (Institut Français du Pétrole) - France
IGCAR (Indira Gandhi Centre for Atomic Research) - India
IIT (Illinois Institute of Technology) - USA
JAEA (Japan Atomic Energy Agency) - Japan
Johnson Matthey - UK
JRC (Joint Research Centre / European Comission) - Germany
KAERI (Korea Atomic Energy Research Institute) - South Korea
KIMS (Korea Institute of Machinery & Materials) - South Korea
Kyoto University - Japan
LCSM (Laboratoire de Chimie du Solide Minéral) - Université Henri Poincarré - France
Lockheed Martin - USA
MINES ParisTech - France
NIMS (National Institute for Materials Science) - Japan
NRC (National Research Council) - Canada
NSTR (Nippon Steel Technoresearch Corporation) - Japan
Nuclear Fuel Industries, Ltd. - Japan
Oxford University - UK
Saint Gobain High-Performance Materials - USA
Saint-Gobain Recherche - France
Sheffield Hallam University - UK
Snecma - France
Standford University - USA
Thales R&D - France
TU Bergakademie Freiberg - Germany
TU Clausthal - Germany
Universität Wien - Austria
University of California, Davis - USA
Veolia Environnement - France

SETARAM Instrumentation
KEP Technologies

Offices in United Kingdom, Germany, Italy, Switzerland and Singapore

www.setaram.com
sales@setaram.com

SETARAM INSTRUMENTATION
7 rue de l'Oratoire
69300 Caluire - France
Phone +33(0)4 72 10 25 25
Fax +33(0)4 78 28 63 55

SETARAM Inc.
8430 Central Ave. Suite C
Newark, CA 94560 - USA
Phone +1 (510) 793 3345
Fax +1 (510) 402 4705

SETARAM China
Rm.201, Building D, Block 7,
No.128 Huayuan Rd,
Shanghai 200083 – PR China
Phone: +86 21 36368319
Fax +86 21 36368094

Inspiring Imagination for Material Science