CeDeD
CENTER OF DEVICE DEVELOPMENT

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The principal task of CeDeD is to develop scientific research systems and devices for use in both the characterization of new materials and the quality control within the production process.

CeDeD offers full scope of expertise in scientific development, planning, design, and construction of research systems. CeDeD covers the entire supply chain of development of Fraunhofer ISC’s research systems – from concept and design stages based on 3-D modeling programs right through to computer-based component production in the workshop. CeDeD is a central point of contact for internal and external research groups and also acts as a direct partner to industry. CeDeD develops prototypes, demonstrators and pilot plants for the manufacture and processing of innovative materials and of research instruments used for process control at the end of the value-added chain.

Particular emphasis is placed on thermo-optical measurement systems designed for in-situ characterization of materials during heat treatment. Measurements can be made under temperature conditions ranging from room temperature to more than 2350 °C. Demand for the CeDeD services is currently particularly strong in the specialty glass and high-tech ceramics industries, as well as in the ongoing development of refractories. The newly developed processes are expanded into industrial scale systems using vacuum engineering, laser technology and robotics. Thermo-optical measurement methods are an excellent choice for all groups of materials that undergo heat treatment during their manufacture, such as materials used in powder metallurgy and injection molding processes.

Certified under ISO 9001:2008, CeDeD guarantees full reproduction of the process chain and is annually audited for its quality management system. It is a reliable partner for organizations seeking to develop new technologies.

Ceramics.....
...are densified and strengthened by sintering. This process controls the quality of the final product – and considerably contributes to production costs. The sintering shrinkage is the most telling indicator for the sintering state. Close monitoring of shrinkage and warping during firing can be used to optimize firing conditions.

Our task
  • Monitoring of the sintering shrinkage without mechanical impact of the measuring device on the samples
  • Recording of any warping or adherence of the sample during sintering even if the sample shape is quite irregular
  • Measuring of creep properties under well defined constant or cyclic loads

Glasses......
...are molten and shaped at low viscosities. Numerous thermo-physical data are required on the way from the raw material to the melt to ensure the precise design of the thermal processes. The measuring of thermophysical properties of glasses around the softening temperature is particularly difficult.

Our task
  • Determination of the expansion coefficient in the range from low temperature to melting temperature, i. e. also around the softening temperature
  • Monitoring of the melting behavior as well as the wetting of refractories and metallic molds by the glass melt at high temperatures

TOMMI plus – Precise material data at high temperatures by thermooptical measuring

TOMMI plus is a follow-up model of its successful predecessor TOMMI with improved performance and more flexibility. It combines a high temperature furnace with an optical dilatometer. The crosslight silhouette of the sample is recorded by a CMOS camera. A special optical system provides distortion free imaging – even if the sample slightly shifts during the heat treatment, e. g. due to thermal expansion.

Dimensional changes of the sample are registered by a purpose-made image analysis software; the sample may be of any shape as long as its complete silhouette remains within the dilatometer window. Measurements are taken once every second so that rapid changes can be registered. Furthermore, melting and wetting phenomena can be investigated by examining the wetting angles and infiltration kinetics.

TOMMI plus is controlled by a standard PC and operated via a comfortable graphical user interface. Besides the data on dimensional changes, single images as well as time-lapsed videos of the thermal treatment can be obtained. The resolution of TOMMI plus is about 0.4 µm with very high reproducibility. It is optionally available with several loading stages and a balance for simultaneous gravimetric investigation of the sample.

TECHNICAL DATA OF TOMMI plus

Optical dilatometer for the control of thermal processes at oxide atmospheres

<table>
<thead>
<tr>
<th>Height x depth x length:</th>
<th>1700 x 700 x 1600 mm</th>
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<tbody>
<tr>
<td>Maximum temperature:</td>
<td>1750 °C or 1200 °C</td>
</tr>
<tr>
<td></td>
<td>(GLASS-TOM for glass applications)</td>
</tr>
<tr>
<td>Measuring window diameter:</td>
<td>adaptable from min 3 mm to max 60 mm</td>
</tr>
<tr>
<td>Resolution:</td>
<td>0.4 µm</td>
</tr>
</tbody>
</table>
Quality of sintering and other heat treatments

Heat treatments need precise conditions to yield best results and ideally the processes can be monitored in situ to gain a better understanding. Ceramics and powder metallurgical materials are densified and strengthened by way of sintering. This process determines the quality of the final product – and accounts for a considerable part of production costs. As sintering shrinkage is the most telling indicator of the sintering state, close monitoring of the shrinkage during the heat treatment can be used to optimize the processing parameters. Weight loss and dimensional changes during debinding provide information on how to optimize debinding cycles. Weight gain and optical inspection allow for the in situ measurement of infiltration processes.

Our task
- Monitoring the sintering shrinkage without mechanical impact of the measuring device on the samples
- Recording of any warpage of the sample during sintering even if the sample shape is quite irregular
- Heat treatment in vacuum, inert or reducing atmosphere
- Measurement of weight changes in controlled atmosphere
- Determining the wetting behavior of melts on solid substrates
- Monitoring infiltration processes in situ

Our solution: TOM-AC

Furnace with controlled atmospheres and dilatometer

TOM-AC is the combination of a high temperature (2000 °C), a high temperature graphite furnace (2400 °C) or a full metal molybdenum furnace (1650 °C) with an optical dilatometer. The crosslight silhouette of the sample is recorded by a CMOS camera. A special optical system provides distortion free imaging even if the sample is displaced, e.g. due to thermal expansion.

Special image analysis software

Dimensional changes of the sample are registered by special image analysis software; the sample may be of any shape as long as its complete silhouette remains within the measuring window (diameter 50 mm). The volume of samples which are rotationally symmetrical can be optionally calculated.

A measurement every second

Measurements are taken every second so that rapid changes can be registered. Furthermore, melting and wetting phenomena can be investigated by examining the wetting angles and infiltration kinetics.

Comfortable use

TOM-AC is controlled by a standard PC and operated via a comfortable graphical user interface. Besides the data on dimensional changes, saved as ASCII-files, single images and time-lapsed videos of the thermal treatment can be obtained. The resolution of TOM-AC is about 2 µm with very high reproducibility.

Benefits

Non-contact optical measuring mode
- No mechanical impact on the sample
- No moveable mechanical parts

Two-dimensional silhouette recording
- Measuring of anisotropic sintering processes and warpage
- Monitoring of wetting, melting and infiltration phenomena
- Simultaneous recording of up to 20 parameters

Image processing software
- Automatic compensation of shifts within the measuring window
- Optimization of the image contrast by illumination control
- Measurements every second in order to record rapid dimension changes
- High reproducibility

Atmosphere control
- Automatic switching between pumping and gas flow
- Computer control of gas mixtures (Ar, N₂, H₂, CO)
- High vacuum conditions

TOM-AC is optionally available with a weight sensor for the simultaneous gravimetric investigation of the sample. A further option is the equipment for measuring samples loaded with uni-axial force.

TECHNICAL DATA OF TOM-AC

Optical dilatometer for the control of thermal processes with controlled atmosphere

Height x depth x length: 2500 x 1000 x 2600 mm

Maximum temperature: 2000 °C - 2400 °C

Measuring window diameter: 50 mm

Resolution: 0.4 µm

TOM-AC – Precise material data at high temperatures to optimize heat processes
IR TOM – Applying thermal shocks

Refractory material is needed in all devices that handle hot materials or open flames. From a medieval clay oven to a giant steel smelting furnace, everything depends on reliable heat protection. But while a broken refractory lining of a clay oven is relatively easy and quickly to be fixed by a skilled craftsman, such malfunctions can cost vast sums of money when occurring in industrial plants.

It is hence vital for this application to characterize refractory materials in terms of their resistance against thermal shocks, which are the common reason for breakage.

Our solution: TOM IR

Within a chamber, floodable with different gasses, nine samples are held with a minimum of their surface being in touch with the surrounding holder. One after the other they get heated in their centre until damages of a certain extent occur. The process is observed visually, acoustically and via two pyrometers facing different areas of the sample.

Benefits

- Controllable rate of heating and cooling
- Selectable environment
- Complete and chronologically correct optical, acoustic and thermal recordings
- Statistical certainty instead of one-shot statements

Our task

- Heating small areas of material samples very quickly in a defined atmosphere
- Acquiring data on temperature, heat conduction and the emergence and progression of cracks
- Repeated measurements at a time on multiple samples of the same type to obtain statistical certainty

SPEEDCAL mobile SCM®

Calibration of multi-channel piston pipettes

Quality control in chemical, pharmaceutical and medical laboratories requires a regular calibration of multi-channel piston pipettes. This is usually a very time-consuming procedure as standards prescribe that every single channel must be tested several times. Many laboratories self-organize this calibration with high expenditure of time or it is outsourced to third party calibration services.

Your solution: Efficient testing with SpeedCal and SCM® - SpeedCal mobile

Simultaneous pipetting with up to 12 channels and automated simultaneous precise measurement (weightings) using 12 weighing cells in the stationary unit SpeedCal or 4, 8 or 12 cells in the SCM® - SpeedCal mobile allow for significant time savings and reliable results.

The new SCM® - SpeedCal mobile was conceived for flexible use. The compact unit may easily be moved and used at different locations. The reduction to only 4 weighing cells in the basic configuration makes the SCM® - SpeedCal mobile a cost-efficient investment for laboratories and service providers who wish to check a high volume of multi-channel pipettes in a cost-efficient and time saving way.

The design enables the testing of 12 channel pipettes, testing 4 channels per segment at a time. A later upgrade to 12 weighing cells is possible. With this, calibration of 12 channel piston pipettes becomes possible in only 30 steps.

The output of measurement results will be adapted to your pipetting software.

Testing according to DIN EN ISO 8655 with SCM® - SpeedCal mobile

According to your type of SCM®, 4, 8 or all channels of a 12 channel pipette will be measured simultaneously. Only 30 pipettings are required with a 12 channel SCM®, 4 channel SCM® requires 90 pipettings. Results are displayed live on PC screen and are available for further handling. The scales reset automatically after each pipetting. Full weighing containers are emptied within seconds with the attachable extraction device.

SCM® - SpeedCal mobile advantages at a glance

- Gravimetric accuracy testing according to DIN EN ISO 8655 in your own lab
- Up to 360 individual values for a 12 channel pipette in less than 10 minutes using the SCM® - SpeedCal mobile 12 channel version
- Portable unit, easy to use
- Available with 4, 8 or 12 weighing cells

SpeedCal and SCM® - SpeedCal mobile also allow for speedy testing of single pipettes. After pipetting into one weighing cell it is possible to use the next cell without waiting for transient effects. This allows testing single channel pipettes in half the time.

SpeedCal mobile - SCM® was developed by the Fraunhofer ISC and is distributed under license by the company Sartorius
A CeDeD invention:
Screening apparatus for glass development

Motivation
The production or introduction of new speciality glasses requires a long and expensive period of development. Time-intensive melting processes needed to analyze properties and melting behavior of the new glasses still further increase the costs. It is a great effort to technologically enable accurately reproducible casting processes. Even a delay of only a few seconds during the casting can significantly change some of the properties.

The automatic approach
The automation of the melting process for a systematic development of glass systems reduces costs significantly and improves the reproducibility.

The automatically produced samples can be characterized quickly and efficiently in our laboratories; some properties can be determined in situ during the melting process.

The screening apparatus
The worldwide unique screening apparatus developed at Fraunhofer ISC consists of a weighing unit for 10 components and an electrically heated furnace (up to 1700 °C) and 5 independently working furnaces (up to 1750 °C).

A robot performs the powder mixing and then transfers the homogeneous powder into the furnace chamber. The glass is melted and homogenized in a platinum 100 ml crucible. Then the liquid glass is poured out into a stainless steel crucible. The glass is cooled down slowly in a separate furnace to relieve stress. This allows for a production of 16 different samples of glass with a defined batch composition of high optical quality within 20 hours.

The following parameters are variable:
• Automatic raw material dosing of up to 10 components
• Adjustable melting program for individual samples
• Selection of thermodynamic models for possible glass systems

Our range of services
• Advice on the selection of appropriate glass systems for the development of new types of glasses according to your specifications
• Screening of new glass systems
• Characterization and interpretation of the properties of the samples produced by the glass screening apparatus according to your specifications
KLIMATOM – In-situ characterization of materials with climate change conditions

The Fraunhofer ISC’s Center for Device Development CeDeD has evolved a new analytical device for in-situ characterization of materials and material combinations with climate change conditions: KLIMATOM.

In a unique way the KLIMATOM enables a non-contact and non-destructive contour analysis of different materials, whereby the shadow image of the sample is investigated. Furthermore, it is possible to analyze in-situ material changes of transparent and translucent surfaces using transmitted light. In this occasion dimensional changes within defined temperature and humidity ranges can be measured and images in high-resolution CMOS technology can be obtained.

General benefit of KLIMATOM

- On-line investigation of heat and/or cold effects
- In-situ characterization of expansion, swelling and bending
- On-line characterization of damage caused by extreme climatic conditions
- Analysis of material combinations with different expansion coefficients
- Determination of moisture sensitivity
- Investigation of crystallization processes (e.g. precipitation in aqueous solutions)

Benefit of KLIMATOM for plastics

- On-line characterization of plastics under changing and extreme climatic conditions
- Analysis of plastic composites combining materials with different expansion coefficients
- In-situ alteration and on-line comparison of separate plastic components

Benefit of KLIMATOM for building materials

- Characterization of swelling and shrinking behavior of insulation materials, concrete and composite materials
- Analysis of bending fracture and deflection under climatic conditions
- Climatic stability test of insulation materials depending on the binder content
- Examination of the functionality of different surface finishing processes

Benefit of KLIMATOM for coatings

- Surface characterization of coatings for quality control of layer application and functionality (e.g. anti-fog, anti-ice, anti-dust)
- Analysis of wetting behavior
- Delamination of varnish systems under influence of temperature and/or humidity

Motivation of using KLIMATOM

- You understand what happens with your material under climatic conditions
- You are able to recognize inconsistencies just in time of the production process
- You have the possibility to start the optimization of your products immediately

Our services for you

- Performing reliable tests of your materials or products
- Scientific support for understanding the material behavior
- Consulting for product optimization
- Individual factoring of KLIMATOM devices

Technical data of KLIMATOM

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Specification</th>
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<tbody>
<tr>
<td>Temperature range</td>
<td>-40°C to +160°C</td>
</tr>
<tr>
<td>Humidity range</td>
<td>30% to 95% r. h.</td>
</tr>
<tr>
<td>CMOS camera system</td>
<td>resolution of 0.3 µm and 20 images per second</td>
</tr>
<tr>
<td>Lighting</td>
<td>100 W LED array (wavelength selective)</td>
</tr>
</tbody>
</table>

Implemented measurement algorithm

Analysis/evaluation of height, width, area and volume
The specially designed computer-controlled unit Autojust® CAM is a semi-automatic machine for adjusting volumetric glassware (volumetric pipettes, measuring pipettes and burettes (tubes)). The system measures the pipettes/burettes (tubes) volume by using demineralised water (in accordance with ISO 3696 grade 3).

The machine has one station and can be expanded by a second station, which can be controlled from the same computer. The stations can be operated by one person. The operator inserts a pipette/burette (tube) into the station and pushes a button to activate the calibration station. During the calibration process a defined amount of water is filled into the pipettes/burette and a high precision camera system searches automatically the meniscus and a marking will be set. When a station cycle is completed, the operator removes the calibrated volumetric glassware from the station, the next one is then inserted into the station and the calibration process is repeated.

The marking will be performed from the calibration unit by a pen as a line or a ring mark. Depending on the ordered equipment the marking can also be made with ceramic paint or by means of a grinding wheel.

A manual will be provided which includes e.g. installation, setup for job changes and maintenance requirements. This general description is according to the standard of March 2014. We make constant efforts to improve and to adapt the machine to the latest state-of-the-art. Therefore it is possible that this description slightly deviates from the actual supply of the machine.

On the customer’s request the machines will be modified to suit the individual aspects desired.

<table>
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<td><strong>Type</strong></td>
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<td><strong>Dimensions 1st station</strong></td>
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<td><strong>Dimensions 2nd station</strong></td>
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<tr>
<td><strong>Weight:</strong></td>
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<tr>
<td><strong>Volume range:</strong></td>
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<tr>
<td><strong>Number of marks:</strong></td>
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</table>

on request the machine can produce up to 5 marks and a volume of more than 50 ml

Volumetric Accuracy

- Burettes: According DIN 12700

The system is common with the latest CE-standards and conceived for production purposes. The scope of supply includes start-up and training for the system at Bronnbach Branch (For destinations in the European Union and Switzerland: at the customers plant) for a duration of five days. A manual will be provided which includes e.g. installation, setup for job changes and maintenance requirements. This general description is according to the standard of March 2014. We make constant efforts to improve and to adapt the machine to the latest state-of-the-art. Therefore it is possible that this description slightly deviates from the actual supply of the machine.

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on request the machine can produce up to 5 marks and a volume of more than 50 ml

Volumetric Accuracy

- Volumetric flasks: According ISO 1042
- Graduated cylinders: according DIN EN ISO 4788

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