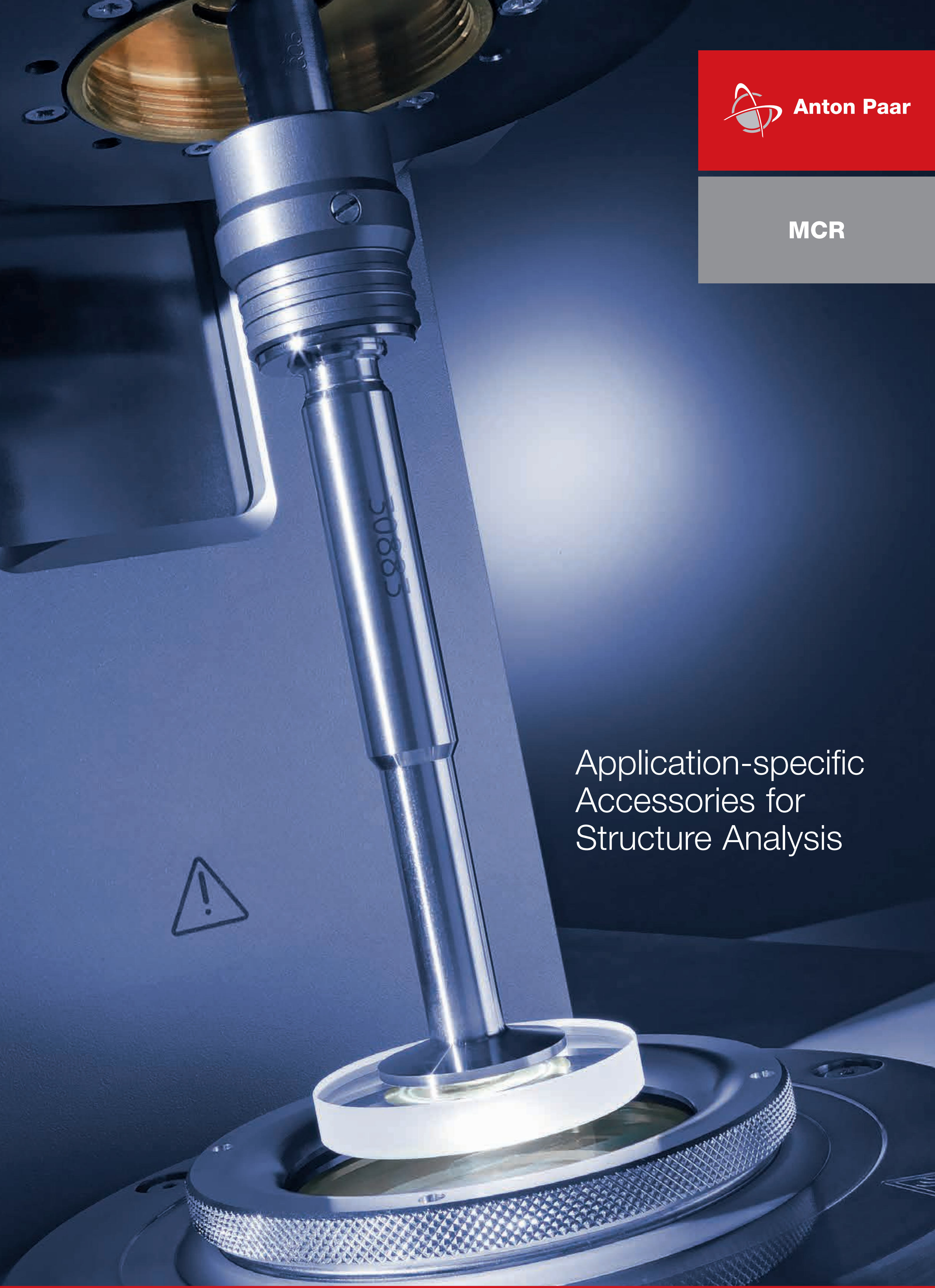
Anton Paar

MCR



Application-specific Accessories for Structure Analysis

Rheometry combined with …

Structure Analysis

Material research constantly calls for new analysis solutions. With their intrinsic modular concept, MCR rheometers from Anton Paar are easily configured with a wide variety of application-specific accessories to match your specific measurement needs.

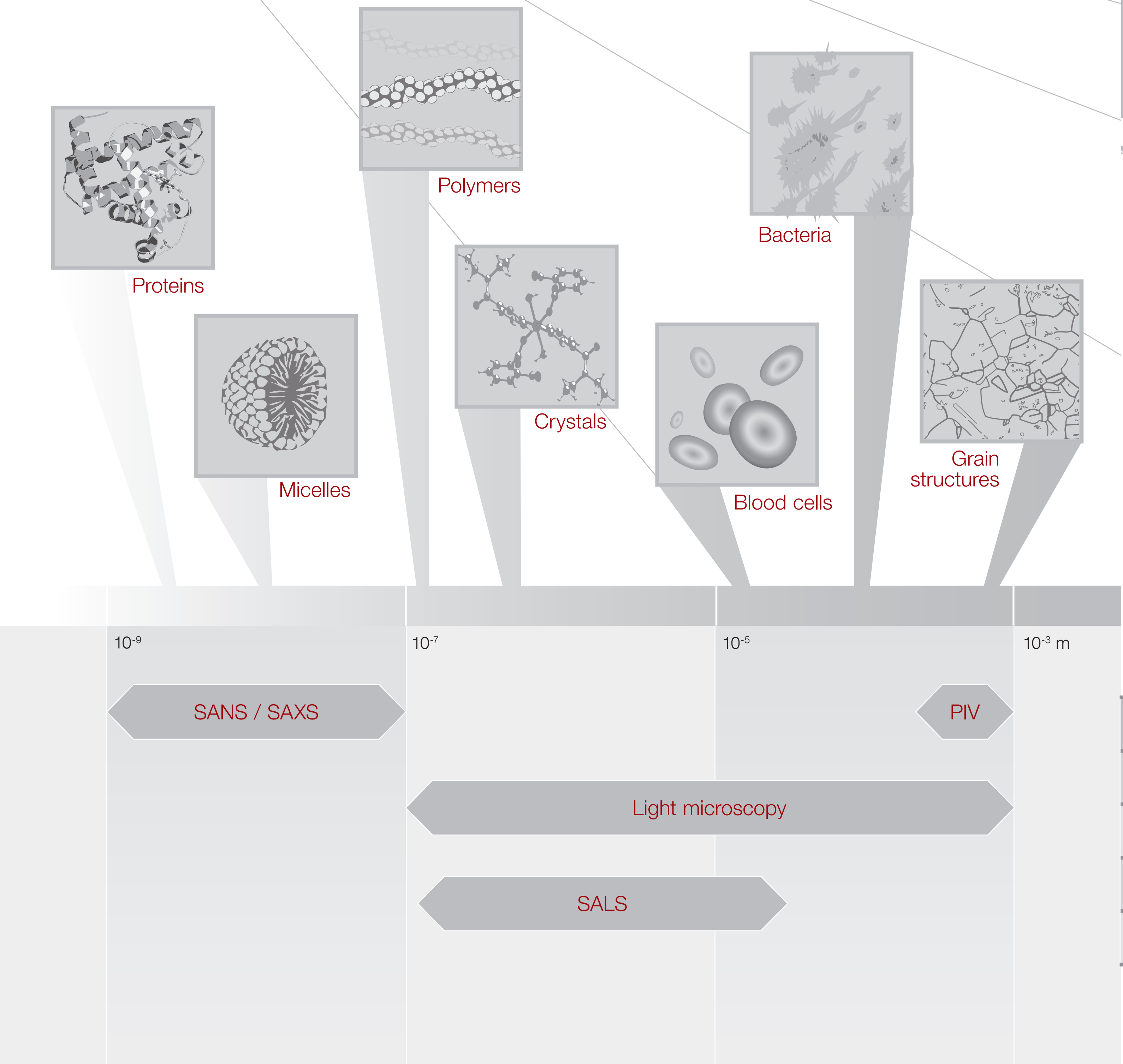
Anton Paar's range of MCR accessories for structure analysis enables you to perform optical measurements and rheological tests simultaneously.

The physical measuring principle of rheometry is used to determine macroscopic material functions – a sample's behavior. However, such functions are the sum result of the sample's microscopic structure parameters, and to analyze samples' microstructures, optical and dielectric methods are employed. A combination of rheometry and optical structure analysis forms a so-called rheo-optical setup – providing you with the macroscopic 'big picture' as well as information on smallest microstructure changes at once.

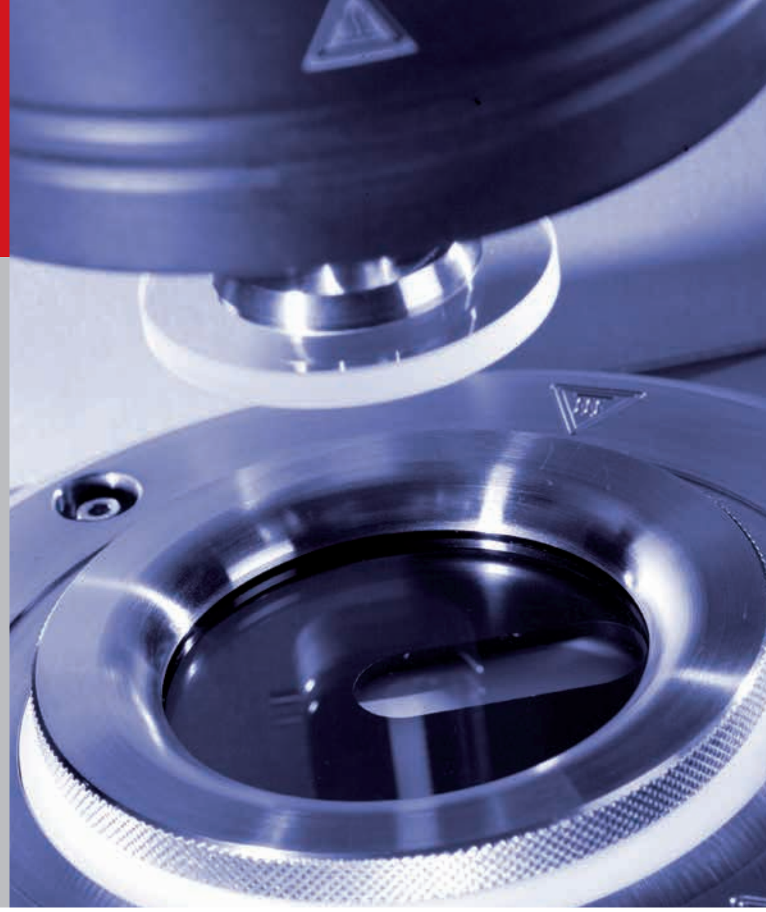
Structure Size Defines Optical Method

When using optical techniques, the wavelength determines the size of the structure that can be observed. Light microscopy and SALS techniques, for example, employ visible light in the wavelength range from 400 nm to 750 nm, which means these principles can resolve structures with sizes according to those wavelengths. Select your accessories in relation to the structure sizes of the materials you are testing.

Depending on the optical method you employ, the applied light either travels in transmission or is reflected. When transmission techniques are used, such as SALS or light microscopy, the samples' concentration and turbidity as well as the contrast (refractive index difference) between their matrix and structure has to be considered. Non-transmission techniques either require tracer particles or a non-transparent sample structure which scatters back the light.



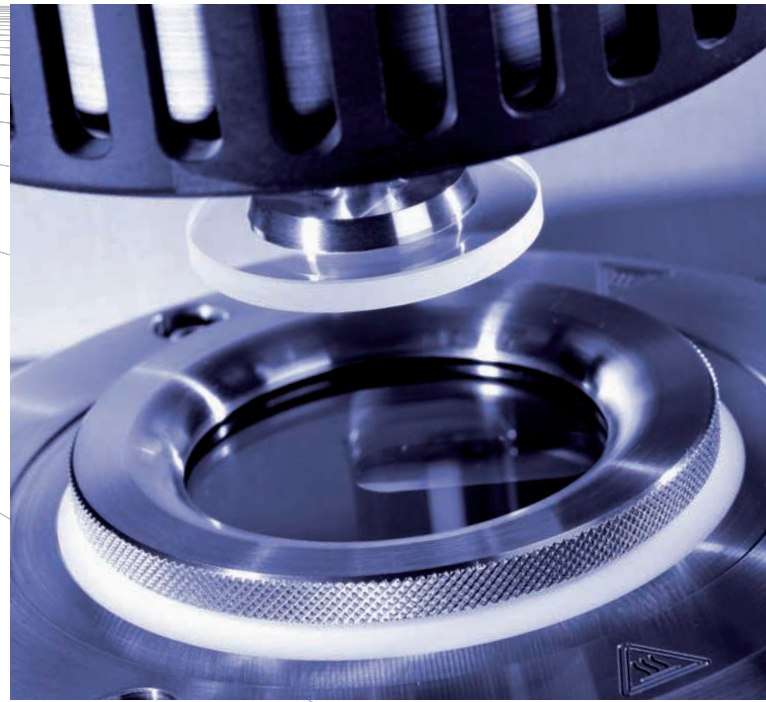
Rheometry combined with … Universal Temperature Control for Structure Analysis



Peltier Universal Optical Device (P-PTD 200/GL)

The Peltier Universal Optical Device (P-PTD 200/GL) is a modular unit that provides you with unprecedented temperature control options: it can be universally applied for Peltier temperature control, whether you attach the Rheo-Microscope, the SALS system, the Polarized Imaging Option, or self-designed optical systems, including spectroscopy systems.

P-PTD 200/GL controls the temperature of the bottom glass plate by Peltier elements and can be used with the patented, actively Peltier-controlled hood (US Patent 6,571,610) surrounding the sample to eliminate temperature gradients.
It provides flexible and accurate Peltier temperature control for optical systems. Various optical requirements – one Peltier temperature control solution.



Electrical Universal Optical Device (P-ETD 300/GL)

For rheo-optical investigations at high temperatures, a Universal Optical Device based on electrical temperature control is also available. Featuring the same modular setup as P-PTD 200/GL, the Electrical Universal Optical Device (P-ETD 300/GL) covers your temperature control requirements for the High Temperature Rheo-Microscope as well as the High Temperature Rheo-SALS system or any other self-constructed optical system at temperatures up to 300 °C.

P-ETD 300/GL is based on electrical temperature control, which is commonly used for higher temperatures reached at considerable heating rates. An additional heated hood prevents gradients in the sample due to the potentially large difference from the ambient temperature. P-ETD 300/GL's temperature range spans from room temperature up to 300 °C.

Specifications	P-PTD 200/GL	P-ETD 300/GL
Temperature range	-20 °C to 200 °C	RT* to 300 °C
Heating rate	30 °C/min	20 °C/min
Cooling rate	20 °C/min	15 °C/min
Maximum diameter of measuring plate	50 mm	50 mm

** Room temperature*

Rheometry combined with …
Light Microscopy

Rheo-Microscope

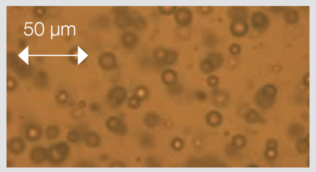
Attach the Rheo-Microscope to your MCR rheometer to gather insights into the inner structure of your samples during rheological measurements.

Light microscopy combined with rheology visualizes the influence of shear and deformation forces on a sample's microstructure. Structural parameters and rheological parameters can be studied simultaneously. For example, you can observe and record the effect of shear-induced structure changes or crystallization processes.

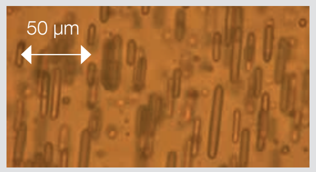
The setup

The Rheo-Microscope consists of a CCD camera, a long working distance objective, and a microscope tube with exchangeable modules. Depending on the desired application, a module with or without polarizers or a module with filters for fluorescence microscopy can be used. The illumination integrated into the microscope tube lights the sample from below. Due to the system's modular design, the light source, CCD camera, and objectives can all be exchanged. The microscope can be moved in the y- and z-directions for focusing on the sample and searching for areas of interest.

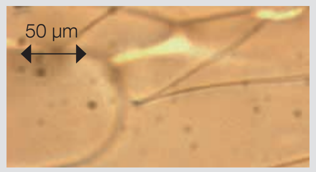
The combination of a heated bottom plate and a hood ensures low temperature gradients in the sample and makes the purging with nitrogen possible, thus preventing sample oxidation. For parallel-plate measurements, measuring systems made of glass are available to prevent reflection and the cover glass plate option allows you to employ thin microscopy glass, e.g. when using the uncorrected objectives. When working with turbid samples, a polished stainless steel measuring system can be used to shed more light on the sample.



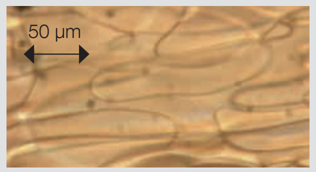
Polymer blend at rest …



… and relaxing after a strain step



Domains of liquid crystals at low …



… and high shear rates

Specifications	
Long working distance objective, standard magnification 20x	
Working distance	20 mm
Numerical aperture	0.42
Resolving power	0.7 µm
Depth of field	1.6 µm
Field of view 2/3" CCD camera (20x)	0.44 mm x 0.33 mm
Further options	
Objectives: 5x, 10x, 50x magnification	
Objective 20x with correction for the optical properties of the lower glass plate	
Cover glass option	
High-intensity LED light source	
Polarizers	
Fluorescence	
Temperature range	
Peltier Universal Optical Device	-20 °C to 200 °C
Electrical Universal Optical Device	RT* to 300 °C
Measuring system	
Parallel plate made of glass	43 mm
Parallel plate / cone-plate stainless steel polished	Up to 50 mm

** Room temperature*

Rheometry combined with …
Small-angle Light Scattering

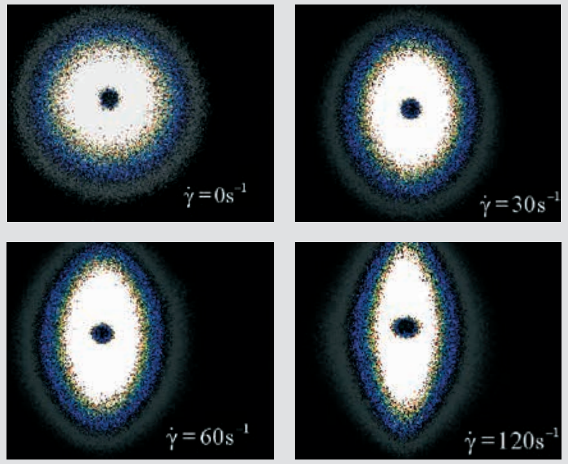
Rheo-SALS System

Connect the Small-Angle Light Scattering (SALS) system to your MCR rheometer to form a Rheo-SALS setup; this setup enables you to investigate shear-dependent microstructure changes or crystallization during rheological measurements.

SALS is one of the most widely used techniques for structure analysis in combination with rheology. A primary laser beam is focused on a sheared sample that scatters this light. The resulting scattering patterns visualize structural changes and contain information about the microstructure. If the patterns are analyzed with respect to angle and light intensity and suitable calculations are employed, size distributions can be estimated.

The setup

The modular Rheo-SALS system features Peltier and electrical temperature control; Sample oxidation can be prevented thanks to the combination of a heated bottom plate and a hood that ensures low temperature gradients in the sample and makes purging with nitrogen possible. A small moveable laser diode emits light which is then transferred towards the sample by a small prism. Depending on the sample's viscosity, a parallel-plate or a double-gap system can be used. Polarizers before and after the sample allow measurements with polarized and depolarized light. A lens system collects the scattered light and transfers it directly onto the CCD chip of a camera. This optical train also contains the beamstopper and maintains the scattering angle as well as the light intensity for a scattering pattern analysis without further corrections. In addition to the laser diode, the optics can be moved as well, allowing flexible focus on different points of the sample.



Scattering patterns of a polymer blend at different shear rates

The large range of scattering angles allows measurements of both small and large structures. The scattered light is focused directly onto the CCD chip resulting in a high light intensity resolution.

Despite all this flexibility, the system does not require any major user alignments – the measuring systems are swiftly adjusted for parallel-plate and double-gap measurements. A dedicated software package for scattering pattern analysis is available.

Specifications	
Laser class 1	
Integrated linear bearing for movements of 15 mm to 20 mm in x-direction from center	
Direct transfer of the scattered light onto the CCD chip	
Laser wavelength	658 nm
Minimum scattering angle	~ 1°
Maximum scattering angle	~ 25°
Minimum scattering vector	~ 0.17 1/µm
Maximum scattering vector	~ 4.17 1/µm
Laser power	390 µW
Temperature range	
Peltier Universal Optical Device	-20 °C to 200 °C
Electrical Universal Optical Device	RT* to 300 °C
Measuring systems	
Parallel plate	43 mm
Double gap	32 mm

** Room temperature*

Rheometry combined with …

Radiolucent Convection Temperature Device (CTD 200/GL)

CTD 200/GL is a radiolucent temperature device based on convection temperature control. Combined with an MCR rheometer, this device provides a basis for various rheo-optical measurements at temperatures ranging from -50 °C to 200 °C (optionally up to 300 °C).

Rheo-SAXS

The Small-Angle X-ray Scattering (SAXS) technique is based on the recording of elastic X-ray scattering by samples with inhomogeneities in the nanometer range. This provides information about the shape and size of macromolecules, characteristic distances of partially ordered materials, pore sizes, and other data. Rheology combined with SAXS techniques enables investigations of shear-induced nanostructure changes in complex samples – from flow- and temperature-induced crystallization to shear banding and many more.

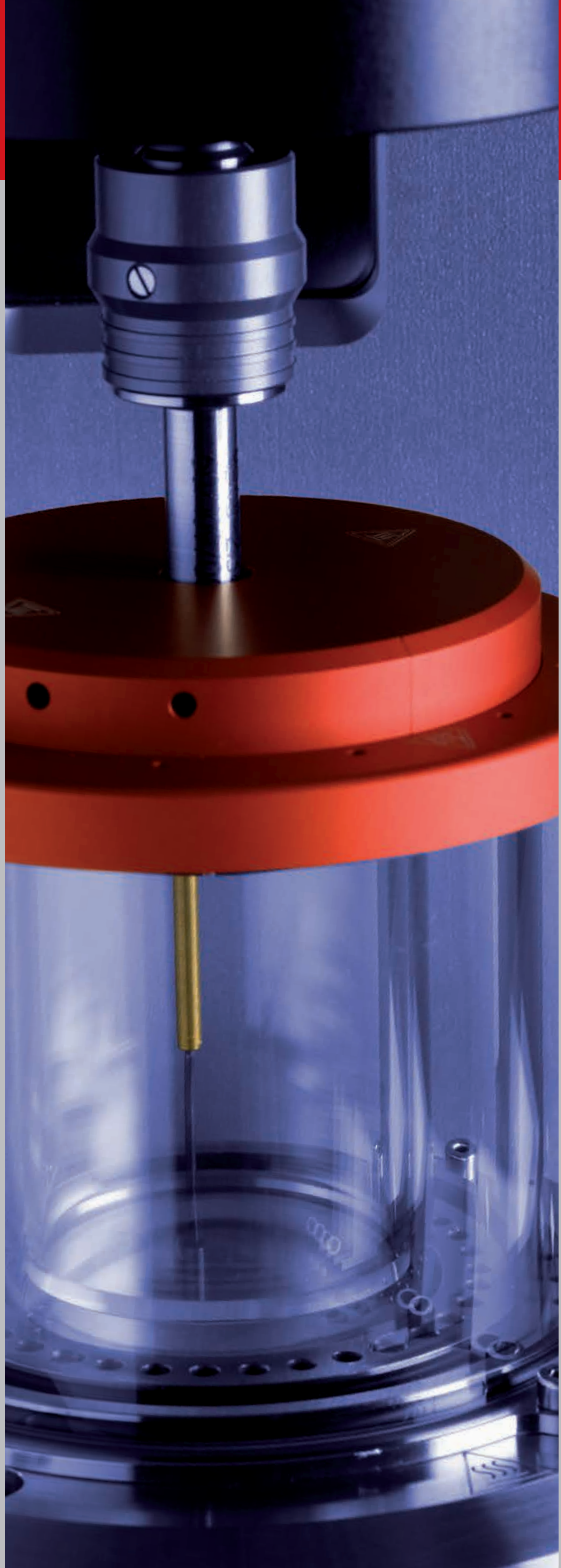
CTD 200/GL is ideal for the combination with synchrotron X-ray radiation, as this highly energetic beam allows a fast time resolution of the scattered beam – a potential requirement for following fast structure transitions induced by flow.

Rheo-SANS

By attaching CTD 200/GL to an MCR rheometer, Small-Angle Neutron Scattering (SANS) experiments can be performed simultaneously to rheology. Neutrons are scattered by nuclei or by the magnetic moment of unpaired electron spins in magnetic samples.

Unlike an X-ray photon with a similar wavelength, which interacts with the electron cloud, neutrons interact with the nucleus itself. Since the neutron is an electrically neutral particle, it deeply penetrates and therefore probes the bulk material.

A special measuring setup for grazing incident small-angle neutron scattering (GISANS) or neutron reflectometry can be integrated into CTD 200/GL.



Flow Visualization, SAXS and SANS Measurements

The setup

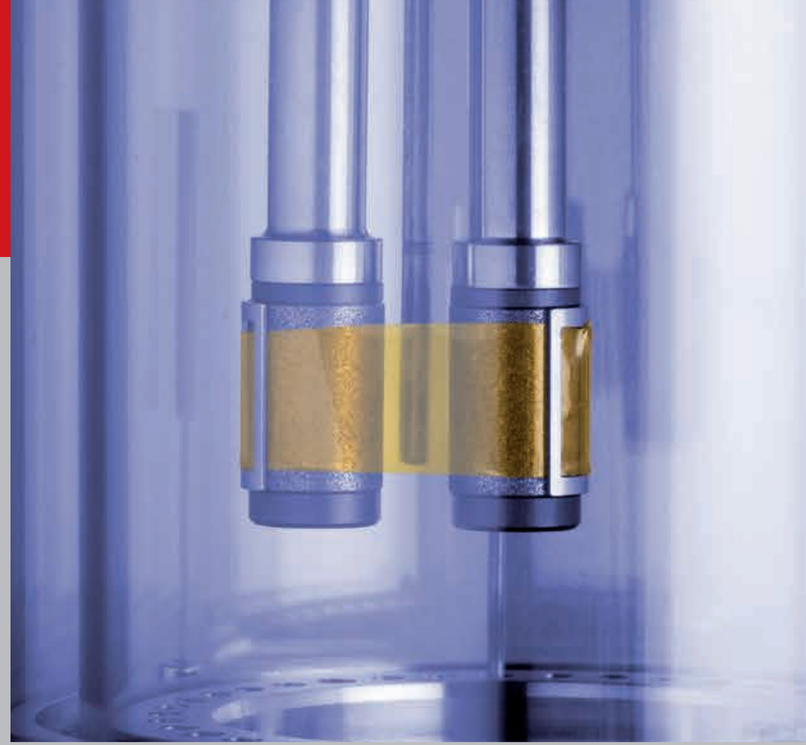
CTD 200/GL is a radiolucent convection temperature device. The standard setup with conventional optically transparent glass measuring systems enables light scattering, birefringence/dichroism measurements, PIV, or simple flow observation alongside rheological tests.

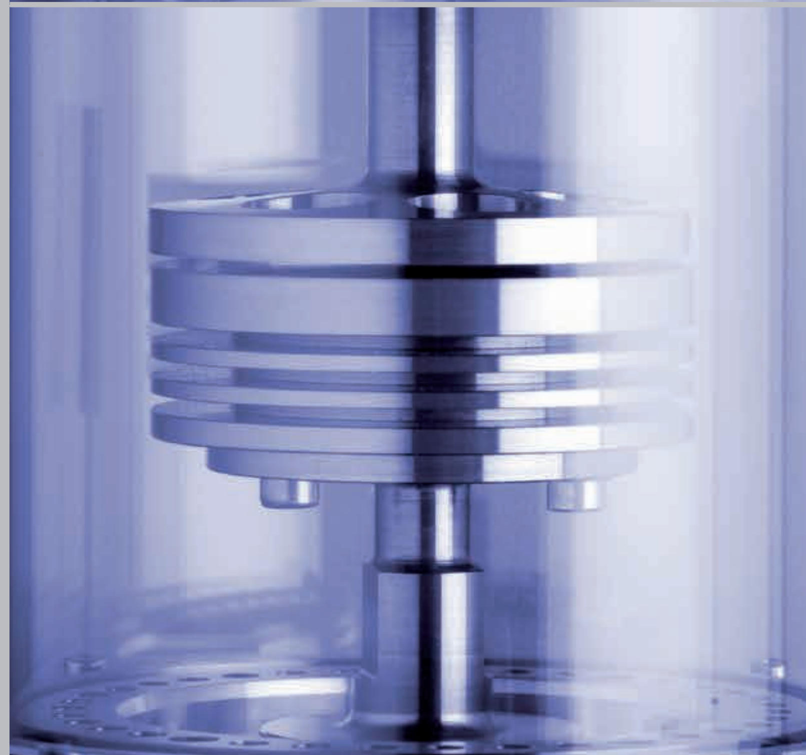
Replacing the glass with available polycarbonate systems, you can use the device to turn your MCR rheometer into a Rheo-SAXS system, and equipped with optionally available quartz glass or titanium rings and cups, CTD 200/GL also facilitates Rheo-SANS applications.
The system can be outfitted with various measuring systems: parallel-plate, ring-plate, and concentric-cylinder systems for standard rheological tests, solid torsion bar and film fixtures for DMA/DMTA measurements, and extensional fixtures for extensional rheology.

With the concentric-cylinder system a simple, fast, and accurate positioning routine is employed for cup alignment. The system allows for various beam positions with respect to the shear flow, providing scattering information at different planes.

Specifications

Convection temperature control	
Temperature range	-50 °C to 200 °C (optional: up to 300 °C)
Maximum heating rate	15 °C/min
Maximum cooling rate (with liquid nitrogen)	5 °C/min
Measuring systems	
Concentric cylinder	
Mooney Ewart type ME20, ME48, ME49	Made of quartz glass, titanium, or polycarbonate
Parallel plate	
Diameter plates	Up to 50 mm, stainless steel
DMTA and extensional fixtures (SRF, SCF, UXF, SER)	





Rheometry combined with …

Transparent Concentric Cylinder (C-LTD 70/PIV)

C-LTD 70/PIV is an optically transparent concentric cylinder system mounted to allow optical access to the sample from the side and from below. The system is ideally suitable for Particle Image Velocimetry (PIV) applications combined with rheology. This combination of techniques allows the visualization of flow fields during rheological tests, such as shear bands, flow instabilities, or startup flow behavior.

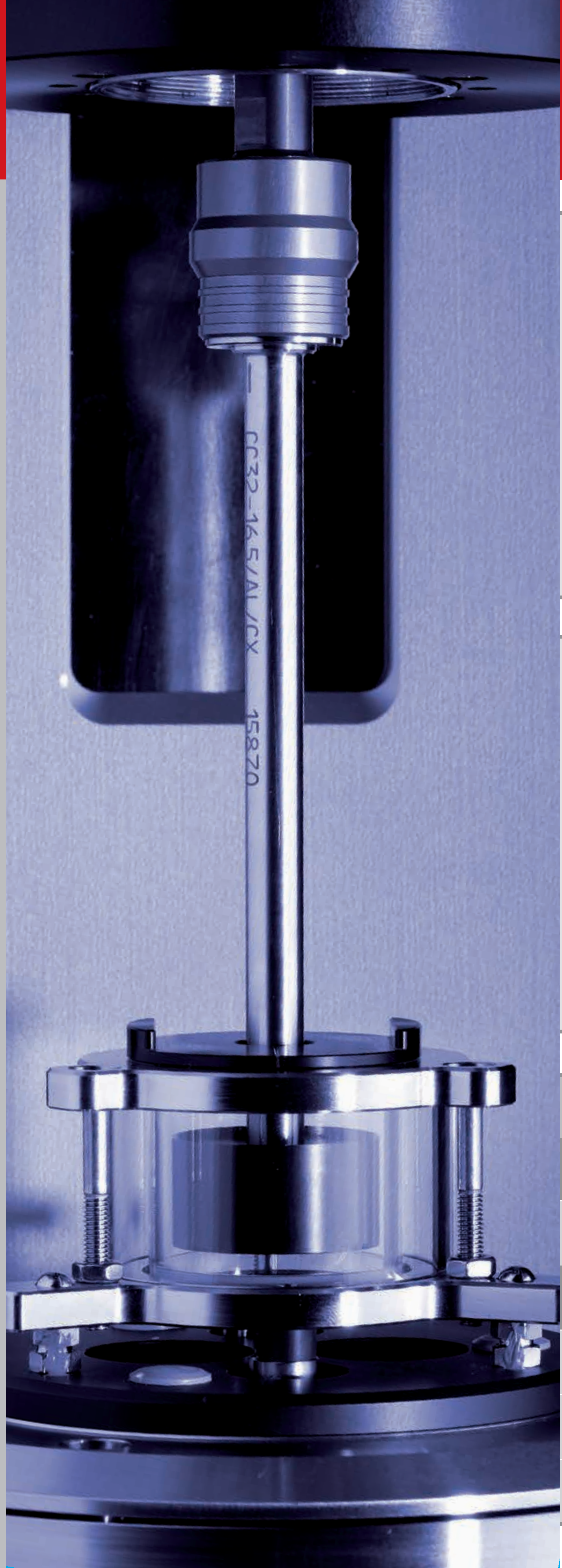
Particle Image Velocimetry (PIV)

A typical PIV device consists of a CCD camera, a pulsed laser, and an optical arrangement to convert the laser light to a light sheet. In addition, tracking particles with reflective coating are suspended in the fluid.

The laser acts as a photographic flash for the digital camera, and the particles in the fluid scatter the light. This scattered light is detected by the camera. In order to measure the velocity of the fluid, at least two separate exposures must be recorded. These two images are then analyzed with respect to the motion of the tracking particles, resulting in flow field vectors (as shown in figure 2).

The setup

C-LTD 70/PIV is available in different sizes (on request). All of them have a glass jacket and a glass bottom and are mounted on stilts. The system is temperature-controlled via a fluid circulator in the range from 10 °C to 70 °C. The measuring system is a black anodized aluminum cylinder which prevents reflections when a laser sheet is used. Cleaning is easy, since the glass cup can be completely removed. The optical setup for PIV is not included.



Particle Image Velocimetry

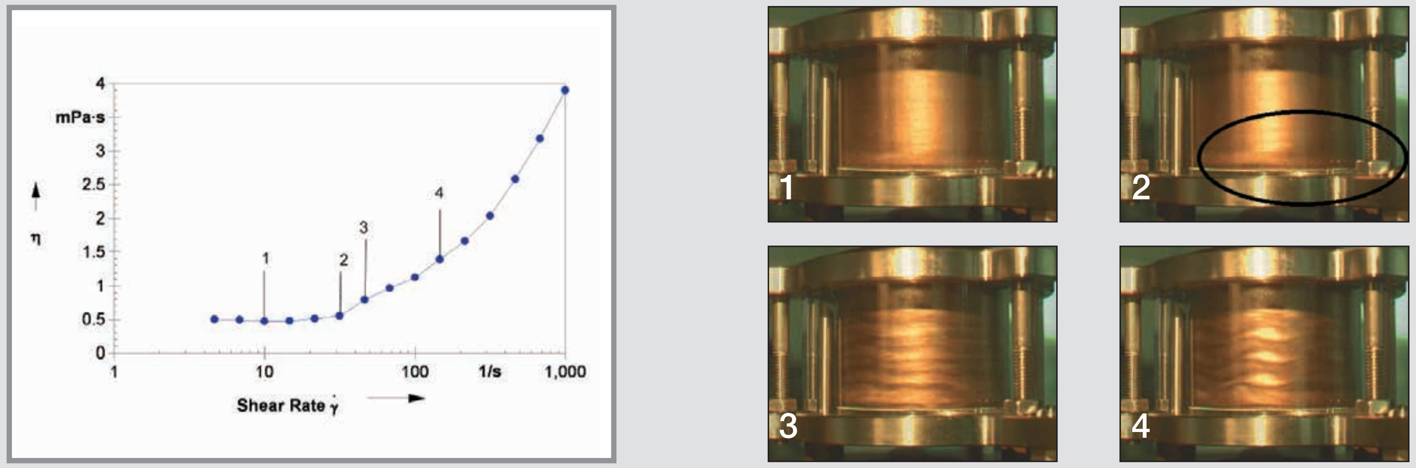


Figure 1: Flow curve of an acetone/gold suspension with related pictures of Taylor vortices

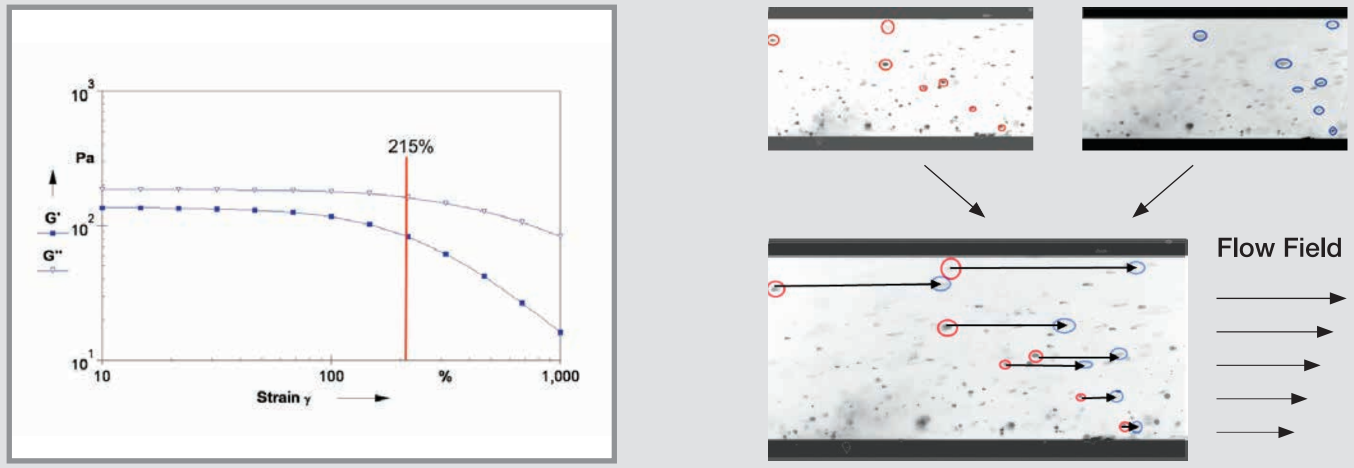


Figure 2: Particle Image Velocimetry (PIV) of a polymer solution in a strain sweep (PP50). The flow field is determined at a strain of 215 %.

Specifications	C-LTD 70/PIV
Liquid temperature control	
Temperature range	10 °C to 70 °C
Measuring systems	
Concentric cylinder	
Diameter cup	35 mm, transparent
Diameter bob	32 mm, black anodized

Rheometry combined with …

Dielectro-Rheological Device (DRD)

Combine the Dielectro-Rheological Device (DRD) with an MCR rheometer to investigate the influence of mechanical deformation on the samples' conductivity, capacity, and permittivity. This setup enables you to analyze the influence of flow and deformation forces on the dielectric spectra of the sample as well as material properties in a range less accessible to mechanical analysis.

Dielectric spectroscopy is an investigation technique based on the study of a material's response to an applied electric field. The electric current flowing through a sample as a response to an alternating electric field is measured as a function of the field frequency. The interpretation of the obtained dielectric spectrum provides information on the structure and behavior of the analyzed material.

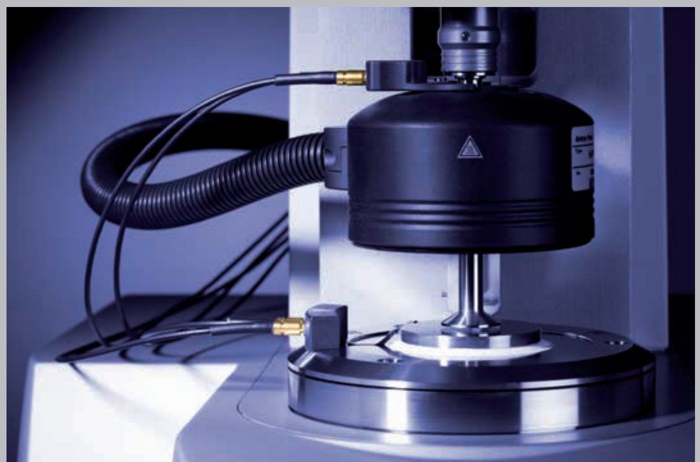
The setup

The DRD setup consists of a LCR meter connected to an electrically insulated and Peltier- or convection-temperature-controlled parallel-plate measuring system. The two plates form a capacitor which can be temperature-controlled in the range from -40 °C up to 200 °C (Peltier) and from -160 °C to 600 °C (convection). The software ensures flexible test programming in rotational and oscillatory modes and synchronizes the LCR meter and rheometer via trigger impulse.

Example

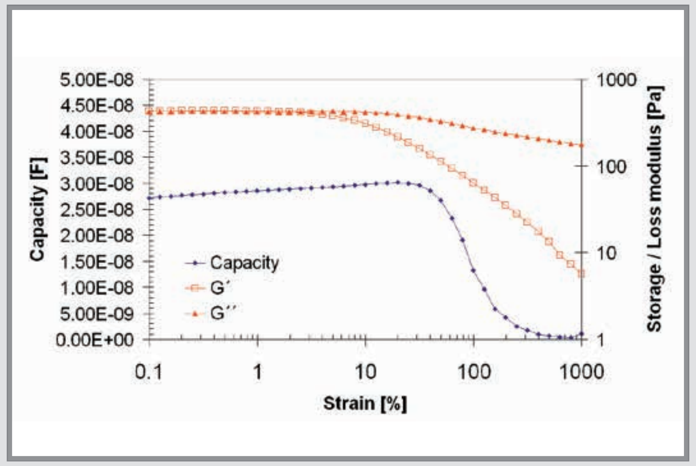
An epoxy resin filled with carbon nanotubes was measured using DRD. A strain sweep and a flow curve test were performed, while an alternating field with constant voltage (1 V) and frequency (1 kHz) was applied.

Result: A significant decrease in capacity is observed when the linear viscoelastic range is exceeded or the shear rate is increased.

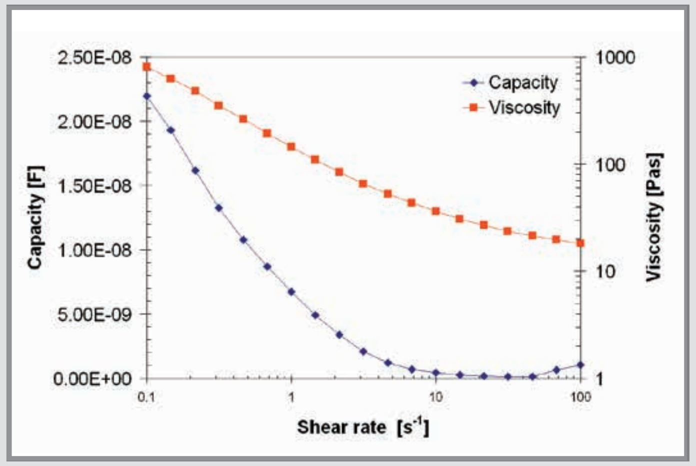


Peltier-temperature-controlled Dielectro-Rheological Device with parallel-plate measuring system

Dielectric Spectroscopy



Strain sweep of an epoxy resin filled with carbon nanotubes and simultaneous capacity measurement



Flow curve of an epoxy resin filled with carbon nanotubes and simultaneous capacity measurement

Technical Data	DRD Option P-PTD 200/DI	DRD Option CTD 180, CTD 450 TDR, CTD 600 MDR
Temperature range	-40 °C to 200 °C (CMT) Electrolyte contact -40 °C to 100 °C	Spring and wire shaft contact: -160 °C to 600 °C (SMT, CMT, Counter Rotation) Electrolyte contact -50 °C to 100 °C
Contact options	Spring Contact Electrolyte Contact Wire-Shaft-Clip	Spring Contact Electrolyte Contact Wire-Shaft-Clip
Min. measurable capacitance	Depending on used contact option and measuring temperature	Depending on used contact option and measuring temperature
Recommended electrolyte	KCl	KCl
Frequency range	Depends on the LCR meter	Depends on the LCR meter
Voltage range	Depends on the LCR meter	Depends on the LCR meter
Test mode	Only single drive	For single and TwinDrive™
Measuring systems	Parallel plate (PP, made of titanium), disposable PP	Parallel plate (PP, made of titanium), disposable PP
Trigger of the LCR meter	Yes	Yes
Standalone dielectric measurements	Yes	Yes
Toolmaster™	Yes	Yes

Rheometry combined with …
Polarized Imaging

Polarized Imaging Option

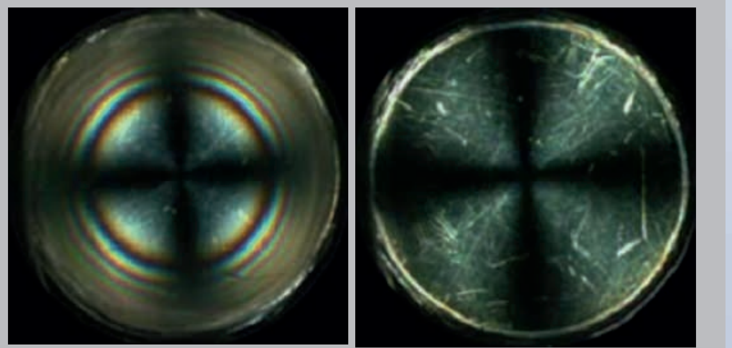
Crystallization processes and different types of crystals are a constant presence in our daily life. Liquid crystals in screens or other optical devices and polymers with different physical properties are present in a lot of products. Knowledge about crystallization processes and crystal orientation is therefore essential for material, process, and product development as they strongly influence the products' final properties.

Utilizing polarized light, the Polarized Imaging Option allows you to observe flow-induced crystallization processes or rearrangements in crystals induced by the shear load of a rheometer.

The setup

The temperature control of the system is ensured either by the Peltier-heated or the electrically heated universal optical device (P-PTD 200/GL or P-ETD 300/GL) and the corresponding hood in a temperature range from -20 °C to 200 °C or from room temperature up to 300 °C, respectively. The optical parts consist of a color CCD camera, an optical tube which transfers the image telecentrically onto the CCD chip, and an inlet for light. A light source emits light which is transferred by the light guide into the optics. This white monochromatic light travels through a polarizer to the beam splitter, where it is deflected towards the sample, which is thus illuminated with polarized light. Different polarization states can be generated by rotating the built-in polarizer. The image of the illuminated sample is transferred telecentrically to the CCD chip, allowing the observation of local effects which may occur in parallel-plate measurements due to the shear rate gradient or due to shear bands induced by the sample.

Shear-induced crystallization in polymers is a very interesting potential application of the Polarized Imaging Option, as the mechanisms leading to the different morphologies are not yet fully understood and the influencing factors, e.g. the molecular weight, are not yet clear.



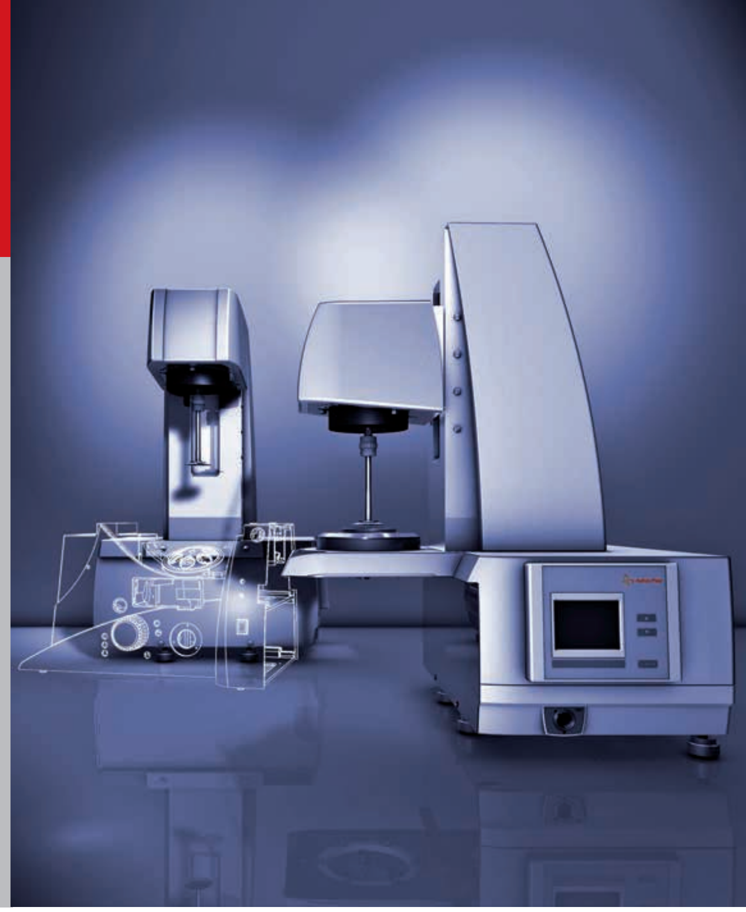
Shear-induced crystallization of HDPE recorded with Polarized Imaging Option

Rheometry combined with …
Further Structure Analysis Applications

Special MCR setups

Self-constructed optical or mechanical setups are often found in the field of rheology. Adapting these systems to the instrument is sometimes difficult, as the working space within or around the instrument may not be sufficient.

Anton Paar offers a solution: the customized MCR rheometer versions WESP and WSP. The enlarged working space underneath the instrument enables you to adapt a confocal microscope or any other optical or mechanical setup, including spectroscopy systems.



MCR WSP (Without Supporting Plate)

In the MCR WSP version the front panel is shifted to the right side of the instrument, resulting in a maximum working space all the way down the motor drive. This instrument features the same technical specification as a standard MCR, even the lift motor is fully functional. All conventional test modes can be performed.

MCR WESP (With Exposed Supporting Plate)

The WESP version is a fully functional MCR rheometer with enlarged working space underneath the instrument.The front panel of the MCR WESP is also shifted to the right side, yet the supporting plate is kept to ensure an optimal parallelity between the measuring system and the devices mounted on the supporting plate. All accessories of the MCR series can still be attached.

DSR measuring head

Some applications only require an accurate rheometer drive or the integration of a rheological measurement into an automation process. The DSR (Dynamic Shear Rheometer) measuring head gives you the flexibility to mount the measuring unit far away from the electronics, as these are separated from the drive. This is especially useful for rheological measurements in areas with limited access around the instrument and for work with self-constructed rheological devices. All standard MCR measuring capabilities are available, with the exception of functions that require the lift motor.

Specifications

For further details and specifications refer to the MCR Rheometer Series brochure.

www.anton-paar.com