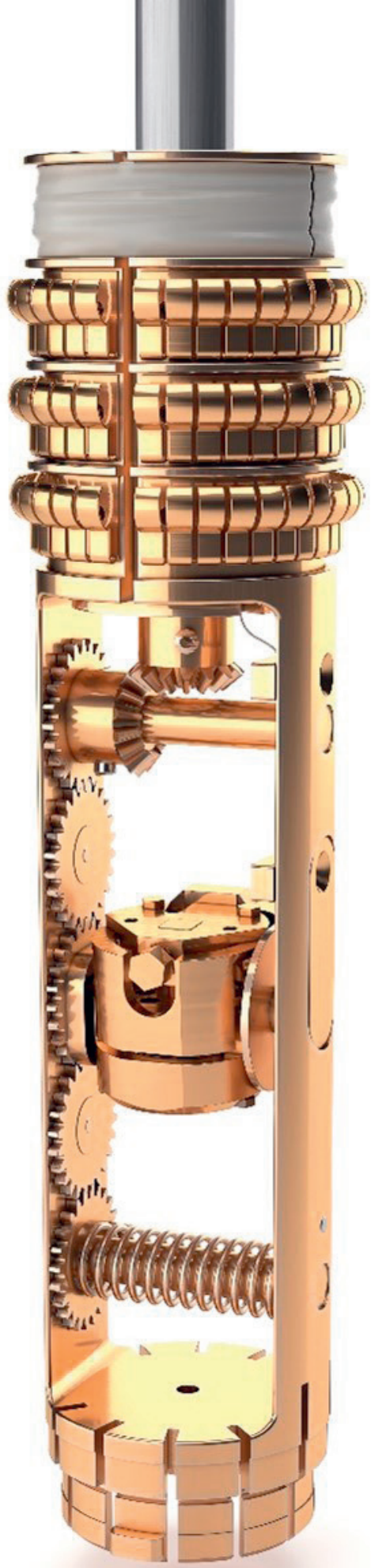
KUECHLER
Innovative Measurement Technology



MANUAL
IN-SITU PPMS-DILATOMETRY PROBE

Dr. Robert Kuechler
Frankenstraße 13 | 01309 Dresden
kuechler@dilatometer.info
www.dilatometer.info

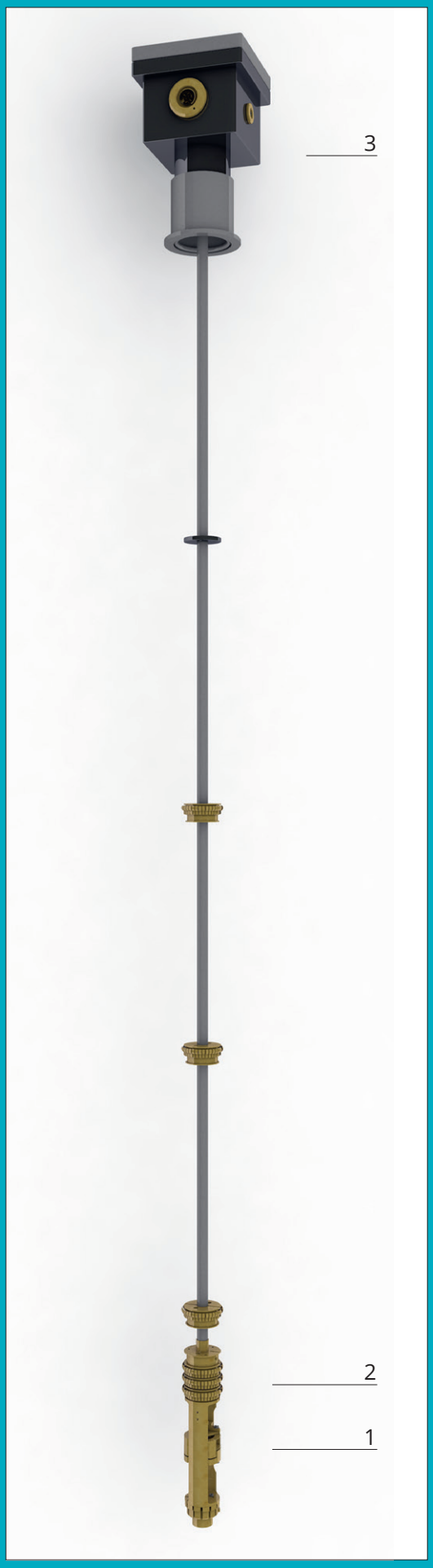
01

IN-SITU ROTATIONAL PPMS-DILATOMETRY PROBE

A mini-dilatometer is mounted and cabled one a mechanical rotator which allows manual in-situ rotation of the sample within the cell between -90° and +90° inside of the PPMS (rotation axis normal to the direction of applied field). This allows systematic anisotropy studies.

INTRODUCTION

The cage (1) of the in-situ PPMS-dilatometry probe contains a mechanical rotator on which a mini-dilatometer is mounted and cabled via miniature coax-wires. There is a rotary know on top of the probe head which allows manual in-situ rotation of the sample within the cell between -90° and +90°. The head (3) of the probe contains also two hermetic sealed Lemo-connectors, which can be connected to the capacitance bridge with the delivered pair of coax-cable. The PPMS-insert is thermally coupled to the annular region at the bottom of the PPMS, where heaters warm the helium gas to the correct temperature, via a pin connector. The PPMS-insert contains very effective gold plated thermal anchors (2), which are fixed on the upper part of the cage (1). These thermal anchors are mounted just above the dilatometer so that they touch the inner chamber of the PPMS cooling channel. Only the lowest part of the inner PPMS cooling channel is made of highly heat conductive material (cooper). The reason the thermal anchors' work platform with its extra-large surface is mounted at this level is to effectively improve the thermal coupling of the mini-dilatometer. The ultra-thin coaxial cables and the twisted Pair Beryllium Cooper Loom are wrapped around measurement probe and anchors to reduce the heat leak. The probe also contains radiation shields to prevent additional heating. The in-situ PPMS-dilatometry package also includes software. The dilatometry data can be read out with a LabVIEW program and the temperature and field sweeps can be controlled with the Quantum Design MultiVu program.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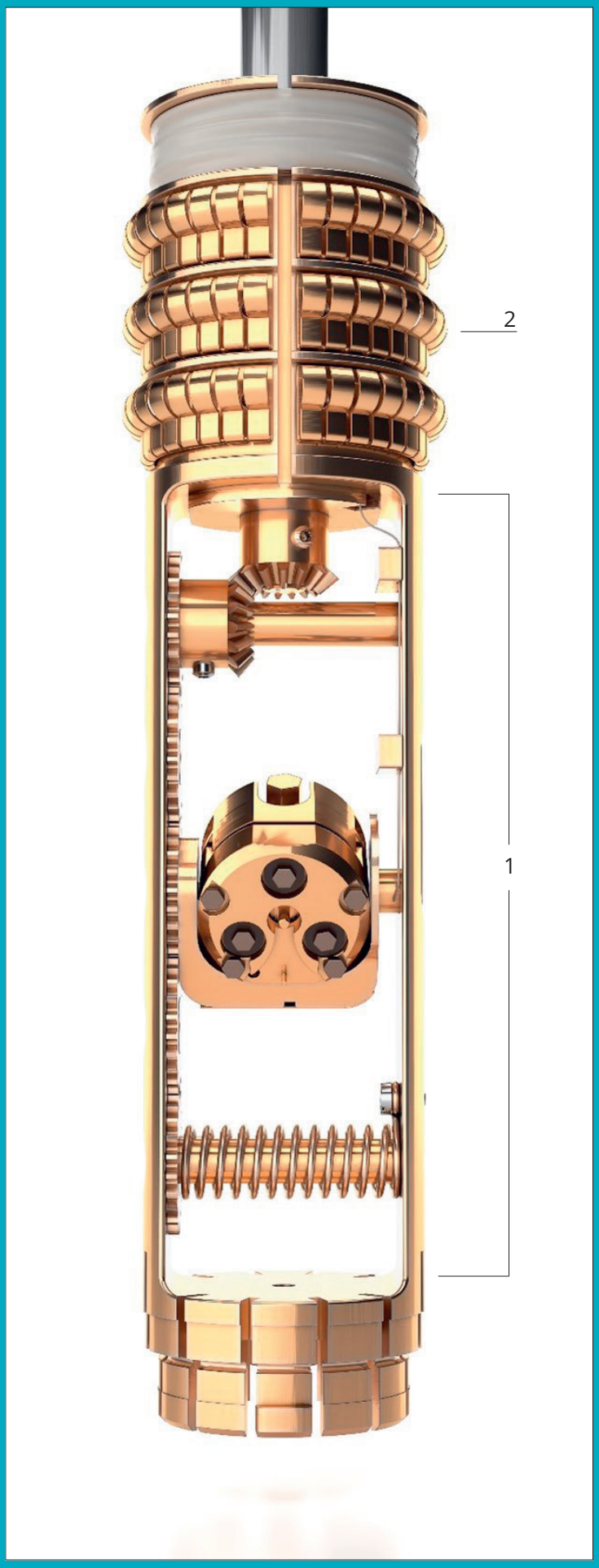
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02

IN-SITU ROTATIONAL PPMS-DILATOMETRY INSERT

WHAT ELSE IS NEEDED TO PERFORM MEASUREMENTS WITH OUR SPECIALDILATOMETRY PPMS INSERT

1 Computer or Notebook

2 Capacitance Bridge

We suggest using the Andeen Hagerling 2550 A (1 kH) or its predecessor model AH 2500 A (1 kHz), or at least a ultra-precision capacitance bridge.

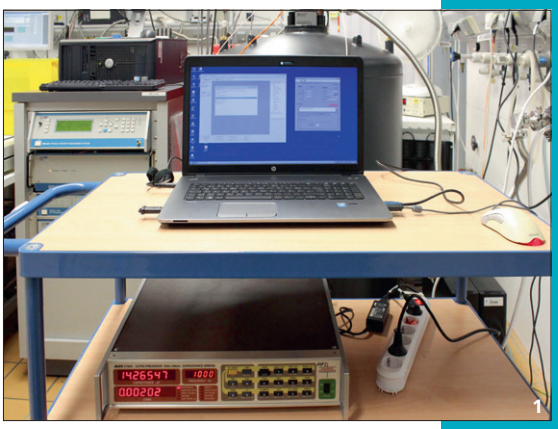
3 GPIB-USB-HS Converter

To connect the measuring bridge with the computer, we use the National Instruments converter.

4 Pair of probe – Capacitance Bridge connecting coax-cable

Is included in the delivered PPMS-dilatometry probe package.

Our LabVIEW software can also be installed directly on the PPMS computer. In this case, the GPIB-USB-HS Converter and an additional Notbook are not needed.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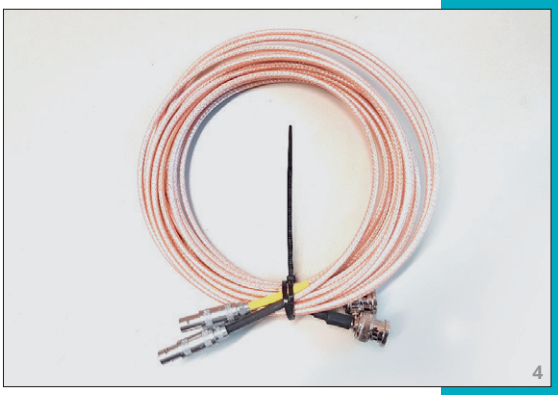
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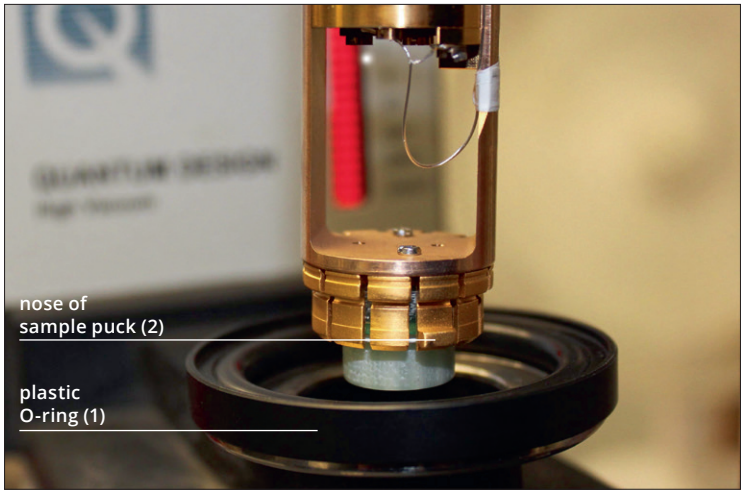
03

DILATOMETRY MEASUREMENT SET-UP

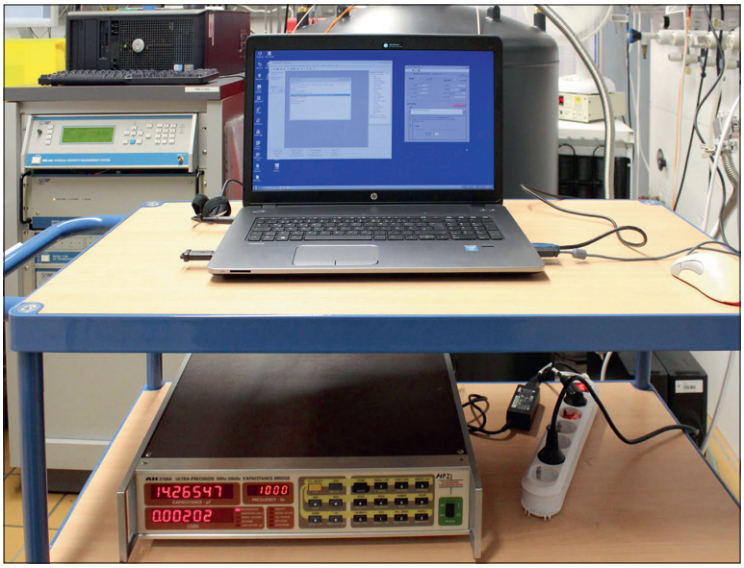
THE PPMS-DILATOMETRY MEASUREMENT SET-UP

First, place the plastic O-ring (1) on the PPMS-port, then cautiously insert the PPMS-dilatometry probe into the PPMS-system. Make sure that the nose (2) of the sample puck points forward and locks in place.

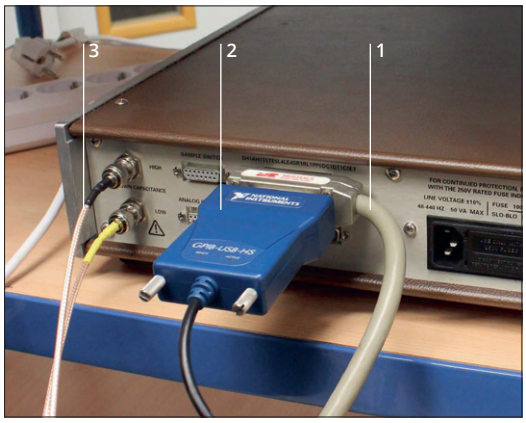
The flange on the probe head glides over a rubber seal ring and can be moved carefully up and down to adjust the correct probe height.

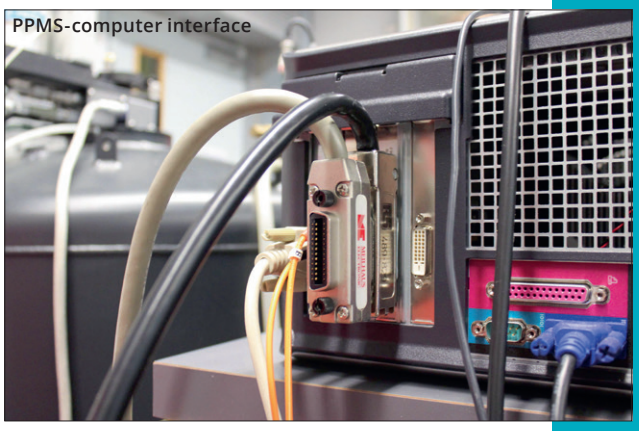


Place your computer and capacitance measuring bridge on a suitable rack.

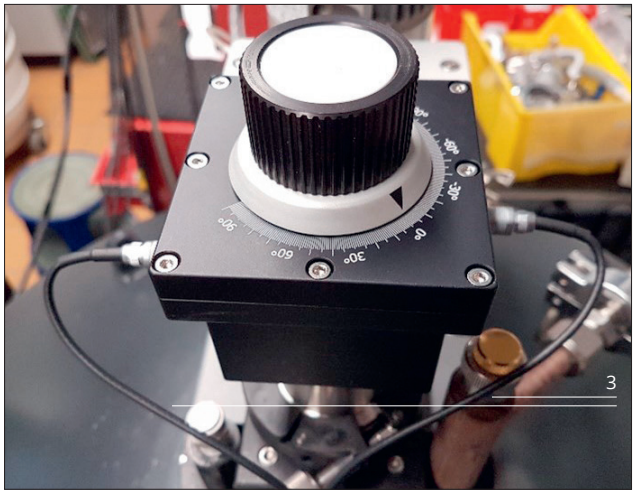


Connect the capacitance measuring bridge by means of a standard GPIB-cable (1) to the PPMS-computer interface and via GPIB-USB-HS converter (2) to your computers USB-interface.





Connect the capacitance measuring bridge by means of the delivered pair of capacitance bridge-probe coax cable (3) with the head of the PPMS-probe.



NEWER TYPE OF PPMS

Unlike in older PPMS-systems, the Magnetic Field Control System is not operated by using a standard GPIB cable in the newer systems. The PC software now communicates with CAN modules for field control, using CAN over USB via the CANOpen-USB dongle. Therefore, the PPMS-dilatometry Measurement set-up has to be assembled as described above but, additionally, the USB-CAN Module has to be connected to your computer's USB-interface.

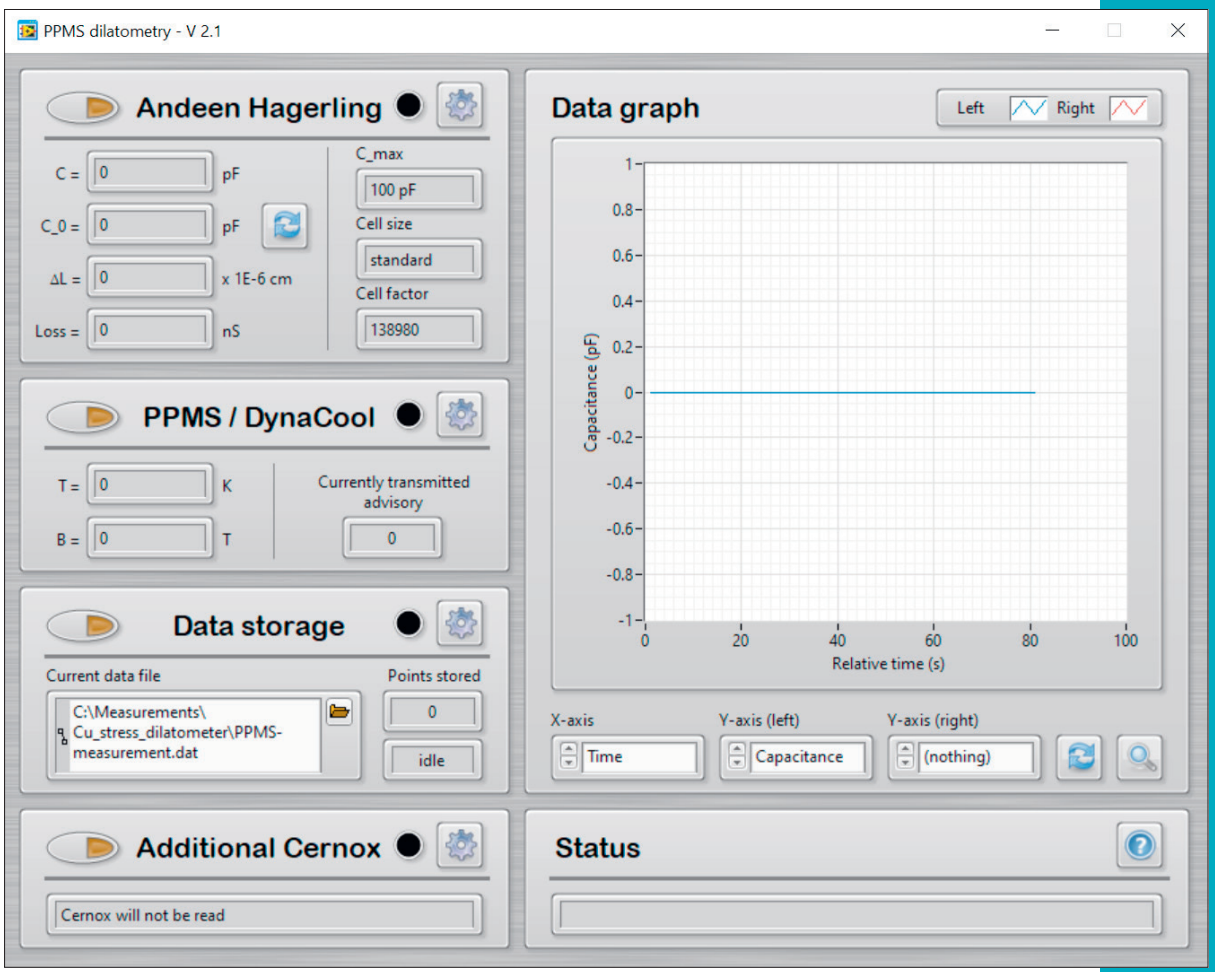
In case the electric power supply in your lab is not filtered properly, we suggest using a noise-surge-protector. To reduce noise during the measurement, all devices of your measurement set-up (like the capacitance measuring bridge and your computer) should be plugged in to the noise-surge-protector.

04

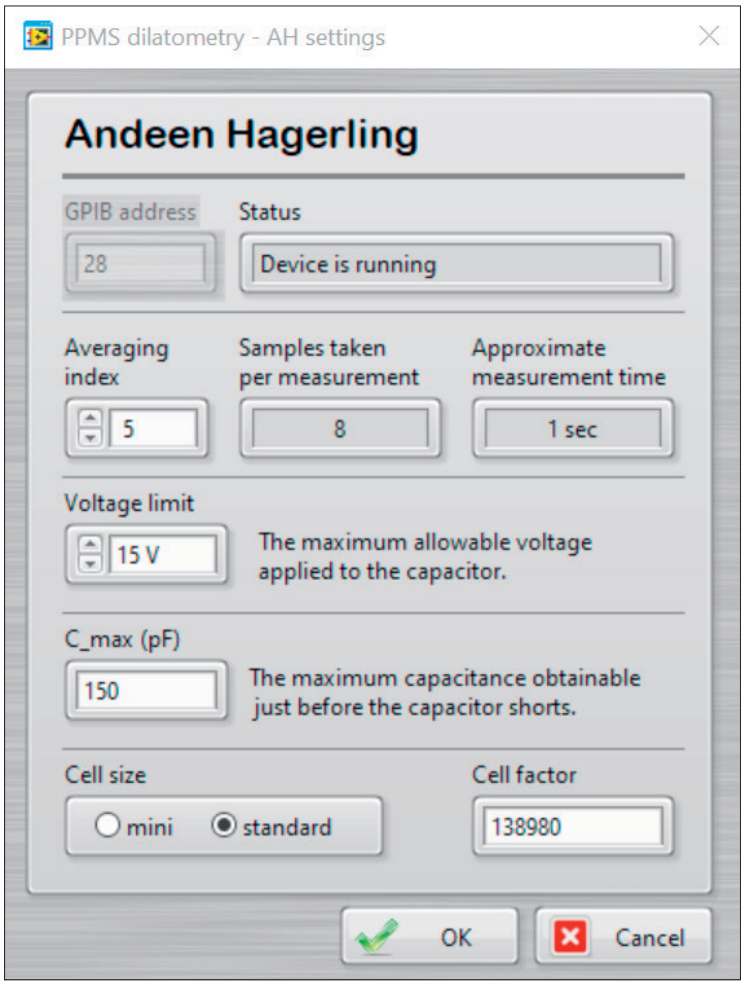
PPMS DILATOMETRY SOFTWARE INSTALLATION

First install the Quantum Design MultiVu and the appropriate LabView Runtime Engine on your computer. You can download it for free from this website
http://www.ni.com/download/labview-run-time-engine2014/4887/en/

To get our newest LabView software (2020 version) running, LabView2018 runtime must be installed.
After that, you can install and start the LabView program. When the program has started, open the settings windows for all the devices and define the appropriate settings:



ANDEEN-HAGERLING SETTINGS



GPIB address:

In the AH-settings, first, the right GPIB address of the Andeen Hagerling Capacitance Bridge has to be assigned: 28

Averaging index:

The Averaging index allows choosing the approximate measurement time over which the capacitance bridge is averaging measured samples. Since noise is proportional to the inverse of the square root of the measurement time, increasing the measurement time will reduce the effect of noise. On the other hand, increasing the measurement time will yield fewer data points.

Voltage limit:

The Voltage limit allows you to specify a maximum signal level to be applied. The bridge will measure the sample at a voltage equal to or lower than this maximum. The bridge will report the actual voltage at which the measurement is taken. The maximum Voltage limit of 15 V produces the least noise effect.

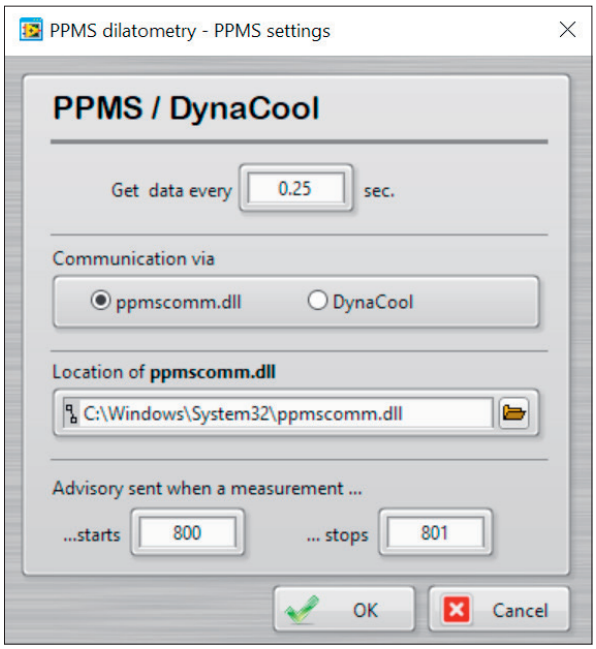
C_max(pF):

In the C_max(pF) setting, the dilatometers specific short circuit capacitance (C_max) has to be entered. You will find the c_max-value on your delivery note. This value yields the maximum capacitance obtainable just before the capacitor shorts.

Cell size:

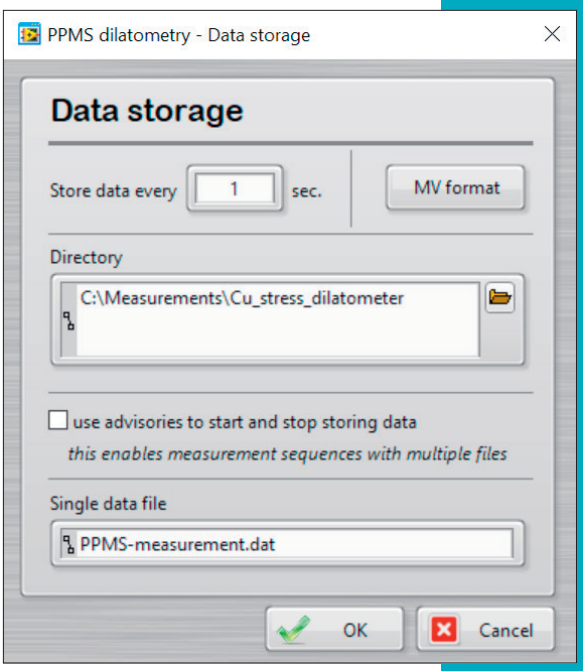
In this section you can choose between the standard (standard and stress-dilatometer) and the mini-dilatometer. The cell size determines the cell factor $f = \varepsilon_0 \pi r^2$ (ε_0 = electric field constant, r = capacitor radius). The cell factor only differs in the different radius of the circular shaped capacitor plates (r = 5 mm for mini-dilatometer and r = 7 mm for standard-dilatometer).

PPMS / DYNACOOL SETTINGS:



For the PPMS, the required location of the ppmscomm.dll has to be assigned: C:\\Windows\System32\ppmscomm.dll

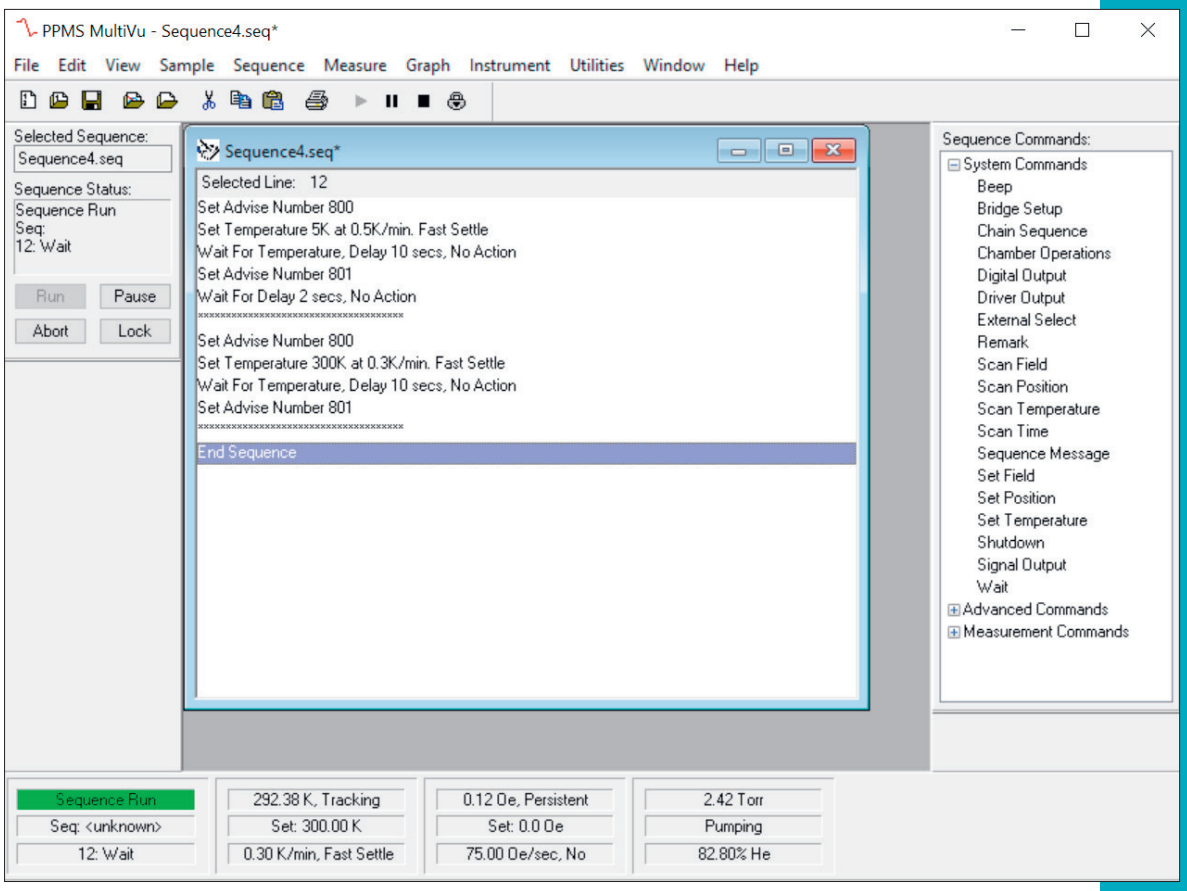
The number shown in the first window determines the number of seconds after which data from the PPMS will be transmitted.



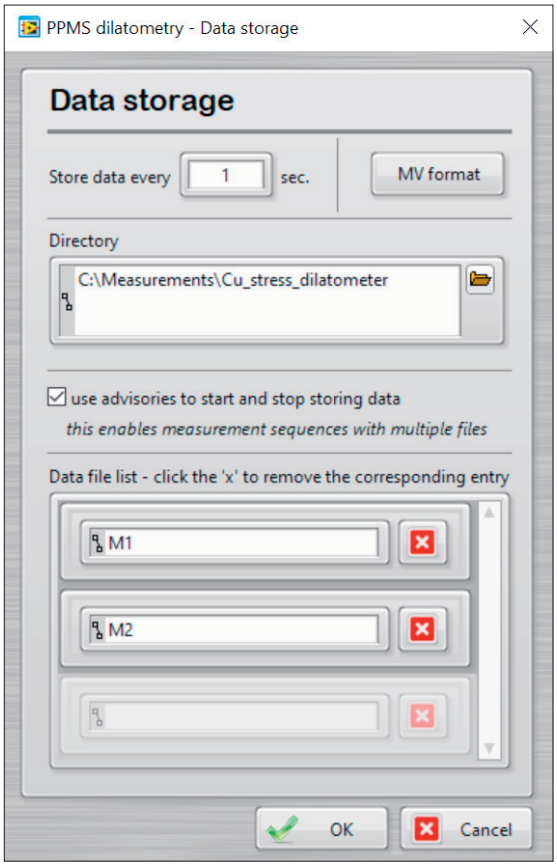
DATA STORAGE:

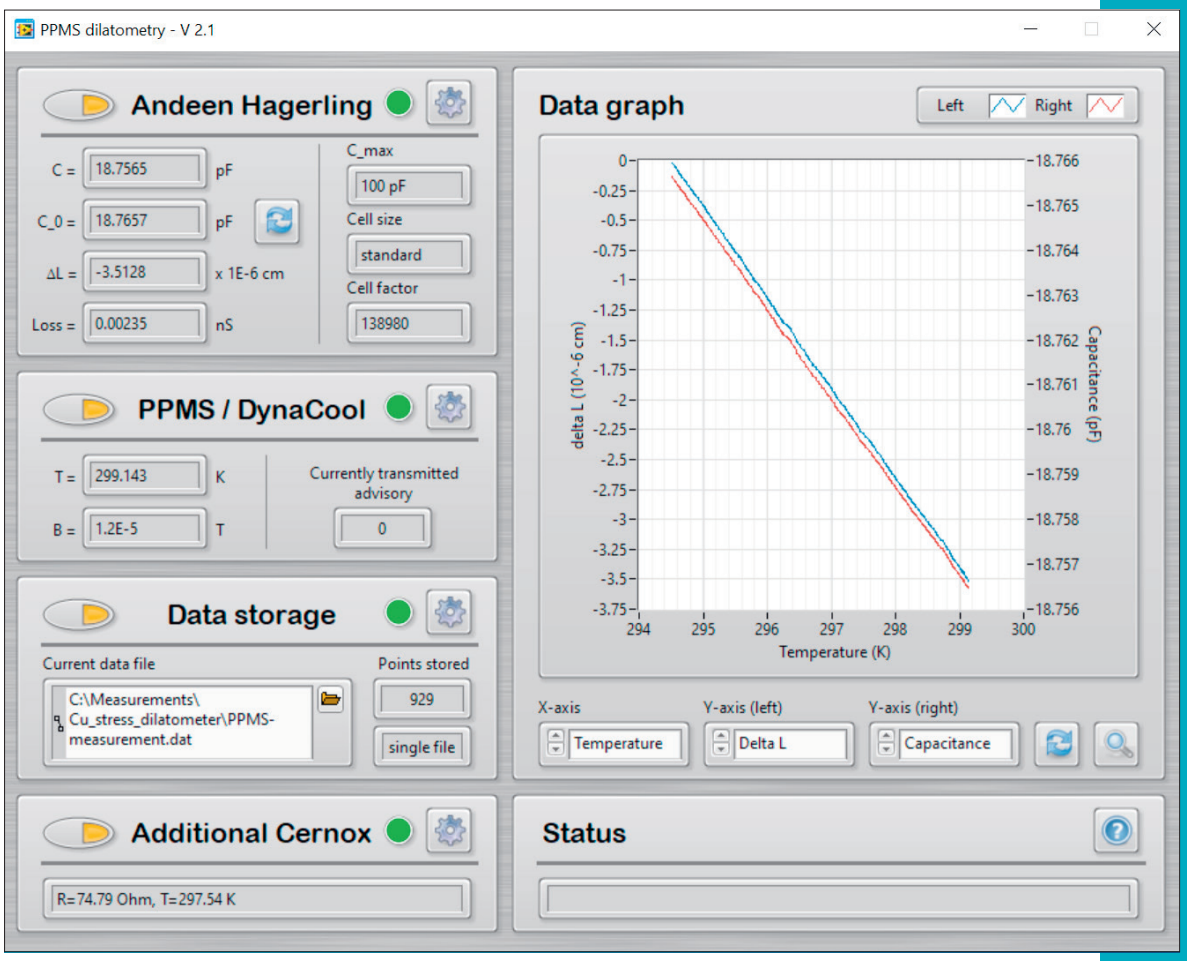
The number displayed in the first window determines the number of seconds after which data obtained from the PPMS and the capacitance bridge will be stored.

The Directory defines the place where the data will be collected.
It is possible to select either a single data file or measurement sequences with multiple files. For the latter, you have to use MultiVu advisory to start and stop storing data. At first, the appropriate sequence has to be written with MultiVu. Note that each time two advisory have to be set. The first advisory is sent when the measurement starts (800) and the second (801) when the measurement finishes.



Only then can the LabView program recognize which file is to be stored. If you press this button , the devices will start running or data storage starts.
Note that starting the MultiVu sequence also automatically starts data storage.





The readout of the dilatometry data can be carried out with the LabView program and the temperature and field sweeps can be controlled with the Quantum Design MultiVu program. The LabView program displays and records the following data:

From Andeen Hagerling (Capacitance Bridge):

1. C (pF) shows the currently measured capacitance
2. C_0 (pF) indicates the initial capacitance
3. delta L (1E-6 cm) displays the already calculated absolute length change in units of 10^{-6} cm based on the formula for slightly non-parallel capacitor plates:

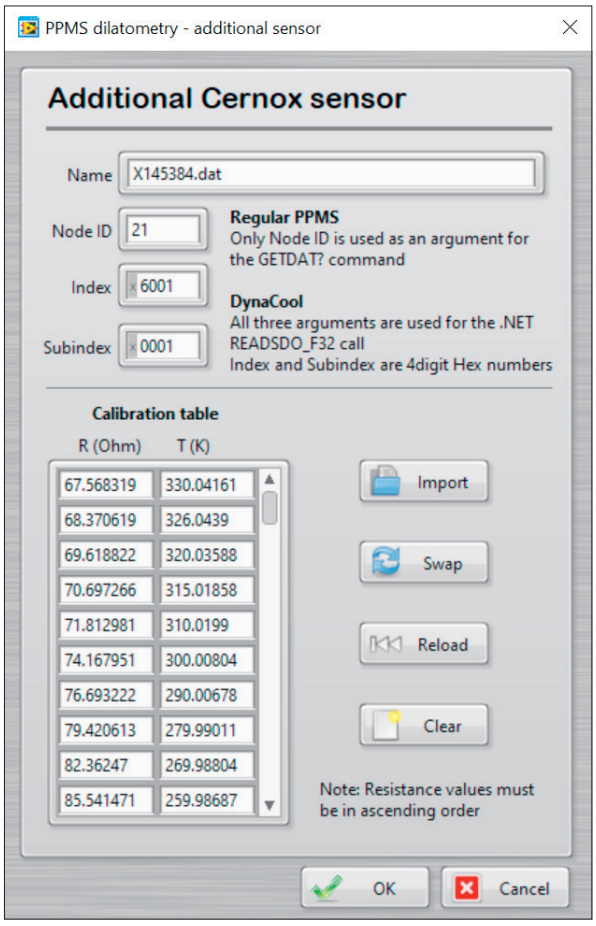
$$\Delta L = \varepsilon_0 \pi r^2 \frac{C - C_0}{C \cdot C_0} \left(1 - \left(\frac{C \cdot C_0}{C_{max}^2}\right)\right)$$

4. Loss [nS] indicates the measured loss

From PPMS:

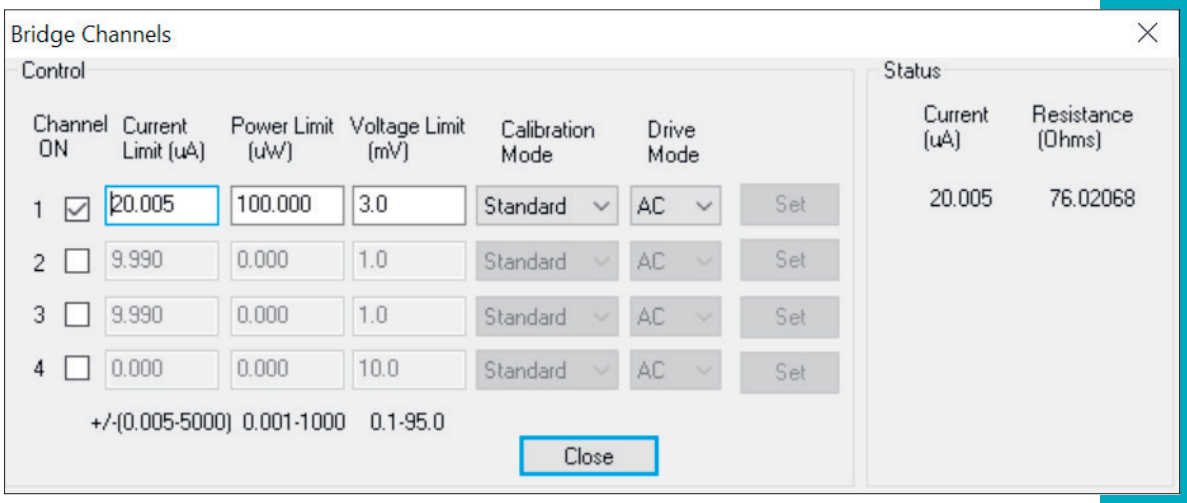
T (K) displays the temperature at the PPMS bottom pin connector.
B (T) displays the current magnetic field.

ADDITIONAL CERNOX SENSOR:



The addition Cernox Sensor is only used in the stress- and standard dilatometer probe but not in the in-situ rotational PPMS-dilatometry probe. For the much smaller and light-weight mini-dilatometer the temperature gradient across the dilatometer is extremely small when sweeping the temperature slowly (0.3 up to 0.5 K/min.) and can be neglected.

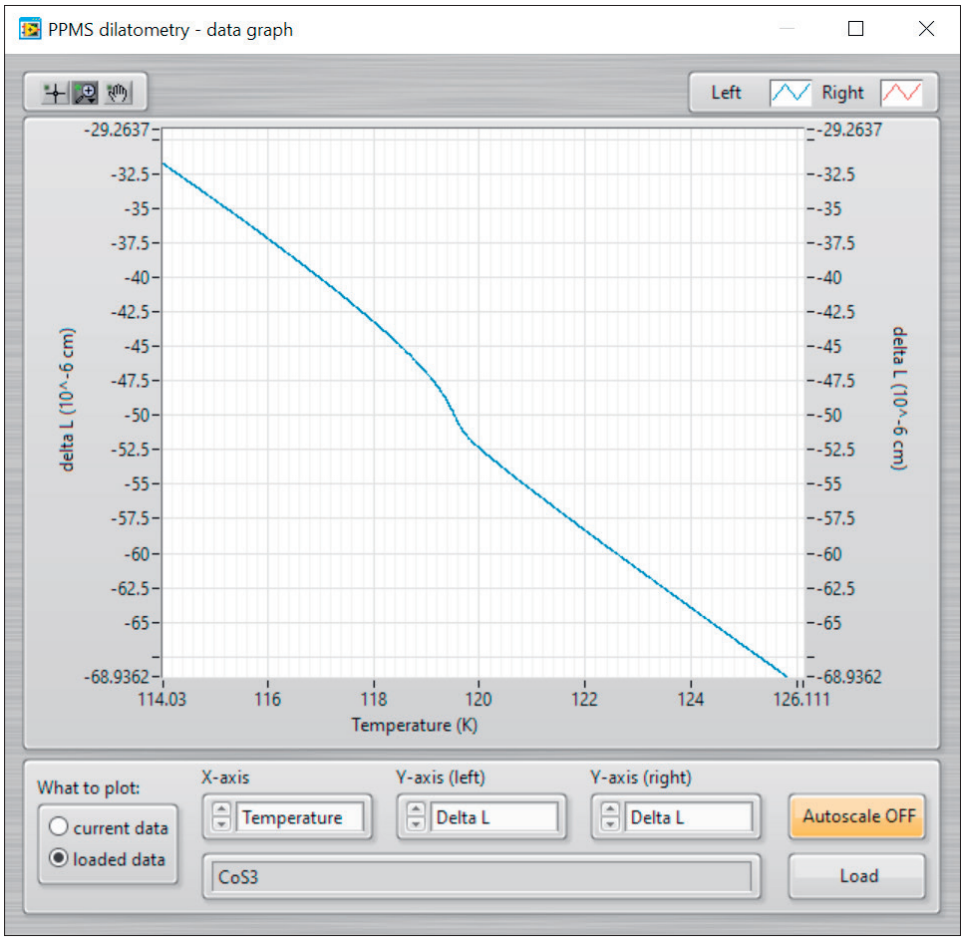
The standard- and stress-dilatometer probes contains an additional Cernox-Resistance sensor, which is enclosed in the dilatometer holder, near the sample. The readout of this additional Cernox thermometer carried out by using the resistance bridge integrated in the Quantum Designs PPMS-system. In the additional Cernox Sensor setting, you will be requested to upload the calibration curve of the corresponding Cernox Sensor. You will find this dat.- file in your software package in the chapter Cernox Sensor. If the provided calibration curve shows the temperature in the left column and the resistance in the right column, please use the swap button to exchange the two.



In order to use the resistivity measuring bridge of your PPMS-system, you need to activate the suitable bridge channels. You will find the bridge channel parameter in the MultiVu program, in the chapter Instruments – Bridge Channels.

Data Graph:

This button on the bottom right in the data graph allows you to load stored measurement data. On the X-axis Time, Temperature, Field, Cernox-R can be displayed. On Both, the left and the right Y-axis as well Capacitance, Delta L, Loss, Field and Cernox-R can be displayed.



MEASSURING PROCEDURE:

The best results can be obtained by sweeping up and down the temperature slowly with 0.3 K/min or comparable sweeping rates. A slow sweeping rate ensures that the temperature gradient within the dilatometer and the sample is very small. For the magnetic field sweeps, we suggest to use the lowest possible sweep rate.

Note for DynaCool system:

The use of the In-situ Dilatometry probe in a DynaCool system requires to install our LabView program and the appropriate LabView Runtime Engine directly on your DynaCool PPMS computer. Additionally the Capacitance bridge has to be connected with a GPIB-USB Converter to the DynaCool PPMS computer.

Here the data can currently be collected only in a single data file. For multiple files our program works with MultiVu advisory to start and stop storing data. These are not available in the DynaCool MultiVu program.

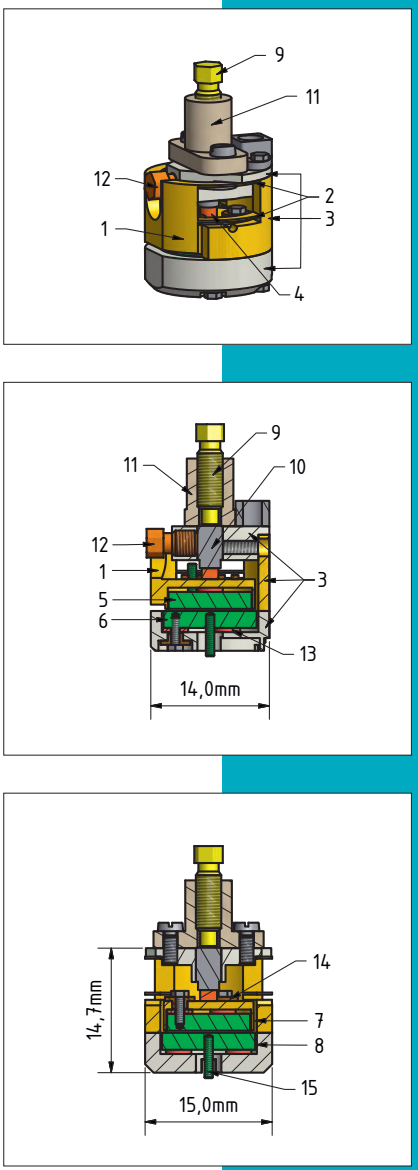
05

MEASURING PRINCIPLE

Measuring principle (capacitive technique) and sample mounting is shown in movie on http://www.dilatometer.info

The mini-dilatometer design is represented schematically in the figure on the right. All of the parts (except for the electrically insulating washers) were produced from ultrapure Be–Cu to reduce eddy currents induced by the time variation of the magnetic field. The Be component of 1.84 % produces an electrical conductivity which is much lower than that of the pure material. The eddy currents induced with the field sweeps are much smaller than those with cells produced from pure copper or silver. The considerable reduction in the dilatometer size and weight has a favorable impact. Less material means less surface area in which the ring currents can flow. The new dilatometer design also includes many recesses: on the one hand to eliminate the screw heads and to thus minimize the external dimensions. On the other hand, the recesses prevent the flow of continuous ring currents and thus minimize the formation of eddy currents.

The mini-dilatometer essentially consists of four main parts: (a) a lower component (shown in grey in Fig. 1), which contains the lower capacitor plate (6). (b) The body (the golden center part), which contains both the two leaf springs and the upper capacitor plate (5). This body essentially defines the shape and function of the dilatometer. (c) The cover (the thin grey part), which is screwed onto the body. This cover's new design includes a lock screw (12, orange), whose function is further described below. (d) The sample-adjusting aid (11, grey). This includes a fine-threaded adjusting screw (9) which can be used to clamp the sample (4, red). The sample can be easily inserted as follows: Unscrew and remove the sample-adjusting aid (11, grey) as well as the cover. Now the sample can be placed very easily on the center of the body from the top. Then the cover is screwed back onto the body, followed by the sampleadjusting aid. The sample is initially still freestanding and is clamped in the next step by a tightening of the adjusting screw (9). In this process, the screw does not press directly against the sample, but rather against a cubic piston (10) that can move horizontally inside the cover. This prevents the sample from twisting when it is inserted. Once the sample is clamped, the lock screw (12) is used to lock the cubic piston (10) in place. The sample-adjusting aid can then be unscrewed and removed. This innovation enables the dilatometer to achieve



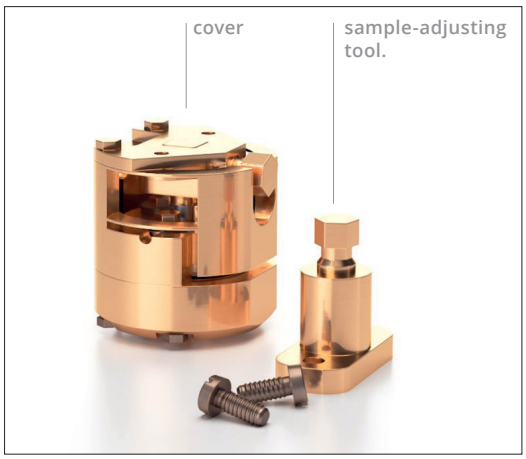
A schematic view of the mini-dilatometer. The upper panel shows a 3D-view, the middle panel shows a side cut-away view, and the lower panel shows a front cut-away view of the cell.
(1) mobile part
(2) Be–Cu flat springs
(3) external frame
(4) sample
(5) upper capacitor plate
(6) lower capacitor plate
(7 and 8) guard rings
(9) adjusting screw
(10) cubic piston
(11) sample adjusting-aid
(12) lock screw
(13) sapphire-washer
(14) insulating piece of Vespel
(15) electrical connection

the preferable low construction height of just 1.47 cm. The dilatometer operates in accordance with the Pott and Schefzyk principle: it consists of an external C-shaped frame (3) and a mobile part (1). The lower capacitor plate (6) is located on the lower grey part of the external frame (3). The upper capacitor plate (5) was mounted at the bottom on the mobile part (1). The external frame and the mobile part are connected by two leaf springs (2) with a thickness of 0.3 mm. The sample can be clamped between the mobile part (1) and the external frame (3) by the adjusting screw. A small force of 4 N against the spring force is required for this. If the length of the sample then changes during the experiment due to a temperature change or a magnetic field sweep, the distance between the capacitor plates also changes in the same manner. A commercial capacitance measuring bridge can be used to record the measured change in capacitance; the simple formula of the plate capacitor can then be applied to calculate this change in capacitance back to the length change caused by the sample. Thanks to the parallelogram suspension, the movement of the upper capacitor plate is exactly perpendicular to that of the lower plate. The mini-dilatometer can be used to measure samples less than 1 mm up to 2.75 mm in size. Each of the two capacitor plates is electrically insulated by three pieces of Vespel (14) and three sapphire washers (13) and surrounded by protective rings (7 and 8) for preventing stray fields. An essential production step is the polishing of the capacitor plates installed in the protective rings. This is done after the capacitor plates have been assembled. The quality of the dilatometer greatly depends on how parallel and vertically the capacitor plates and the external contact surfaces of the housing can be polished. We have developed a patented polishing device for this purpose. Thanks to this device, we achieve a nearly absolute parallel orientation of the capacitor plates towards each other. The two capacitor plates are 0.25 mm apart in the initial position. This corresponds to a capacitance of around 3 pF. Once the sample has been inserted, an adjusting screw (9) is used to clamp the sample even tighter. All of the mini-dilatometers manufactured cause a short circuit at a capacitance exceeding 40–50 pF. This makes it possible to work with a very high measuring capacitance between 10 and 20 pF. At a measuring capacitance of 20 pF, the two capacitor plates are merely 0.025 mm apart and a force of 3 N, which is negligible for most materials, is applied to the sample. At such a high measuring capacitance, the absolute value of the capacitance can be measured with a resolution of 10^{-6} pF, which corresponds to a sensitivity of $\Delta L = 0.02$ Å in the absolute length change. The new mini-dilatometers are extremely high-resolution dilatometers despite their tiny dimensions. In the PPMS a resolution of $\Delta L = 0.2$ Å can be resolved.

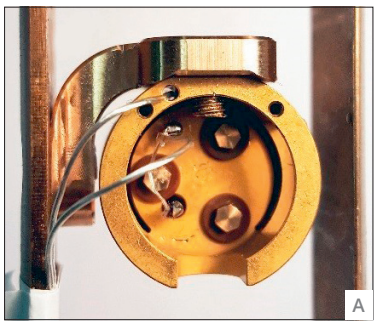
06

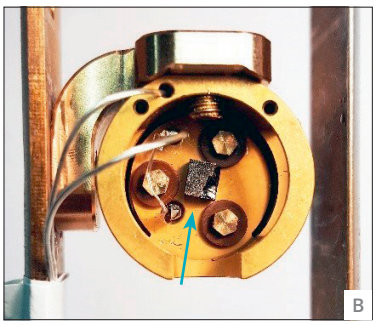
SAMPLE MOUNTING IN MINI-DILATOMETER

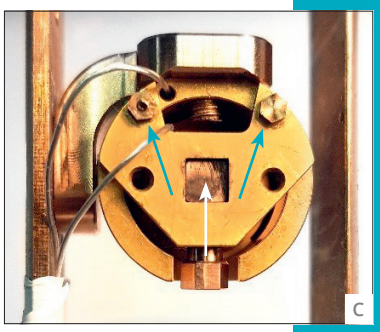
First, put your In-situ PPMS-dilatometry probe on a table. Before you start mounting the sample, connect the PPMS-probe with your capacitance measuring bridge so that you can read the capacitance during the sample mounting. The initial value shows you the capacitance of your empty dilatometer and should be about 3 pF.

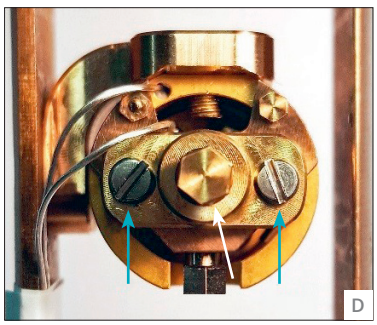


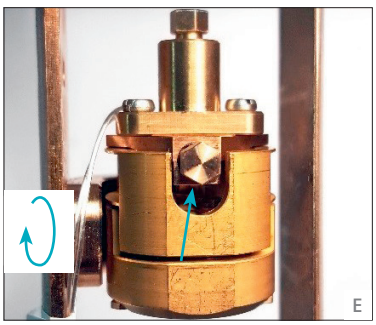
For better handling, Mounting tools are provided:
M1.8 and M3-socket wrenches. The M1.8 socket wrench is not a standard tool since it has to be machined to allow dilatometer mounting.

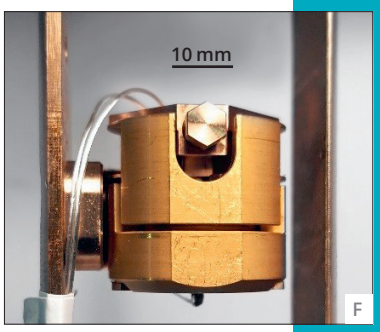










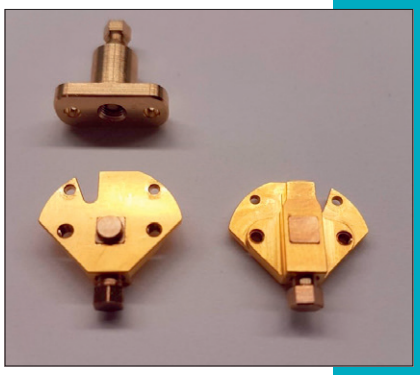


In the following, the sample mounting is explained systematically using the figures above. As a first step, unscrew the sample-adjusting tool and the cover. Fig A shows the open dilatometer. Place the sample carefully in the center of the dilatometer body (see blue arrow in Fig B). Make sure that the sample doesn't

touch the pieces of vespel which insulate the screw. Samples with complicated geometries can also be stabilized with some epoxy or glue (e. g. GE Varnish) so they stay upright. Then take the two M1 screws (see blue arrows in Fig C) to re-attach the cover to the body. Before attaching the cover, take out the cubic stamp (white arrow in Fig C) in order to be able to check the position of the sample after having mounted the cover. The sample remains unattached in this process. Afterwards, insert the cubic stamp into the cover and slightly press it down to lock the sample in place. In the next step, use the two M1.6 screws to mount the sample-adjusting tool (blue arrows in Fig D). Then further tighten the adjusting screw to increase the force to the sample (white arrow in Fig D). During this process, the screw does not press directly against the sample, but rather against the cubic stamp, which can only move vertically. This prevents the sample from twisting. The adjusting screw has a very fine threading and can be adjusted most precisely by a screwdriver M3-socket wrench. Tighten the adjusting screw carefully until the desired measurement capacitance of 15 up to 20 pF has been achieved. After that use the lock screw (blue arrow in Fig E) to lock the cubic stamp in place. The sample is now clamped under a force of about 3 N. Such a force can be neglected for most materials. As the final step, unscrew the sample-adjusting tool and remove it (see Fig F).

Note on the sample mounting:
The cubic stamp (piston) can be inserted in the dilatometer cover in two ways (see picture below).

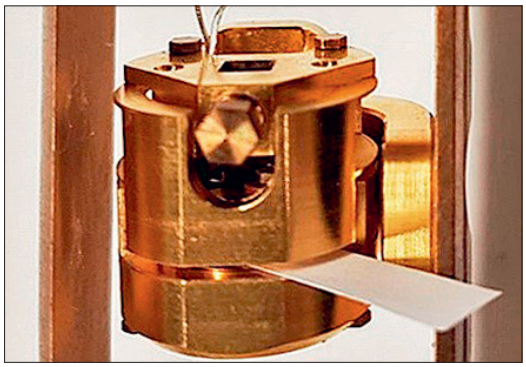
Only if the stamp is inserted with the round cylinder pointing up (left configuration) samples of up to 2.25 mm can be measured. The cylindrical part of the stamp fits inside the thread hole of the sample-adjusting aid. If the sample's cross section is smaller than the stamp's cross section, even samples of up to 2.75 mm can be measured. We provide two different pistons. The smaller one is designated for the measurements of larger samples.



Sample mounting is also shown in the video at
http://www.dilatometer.info

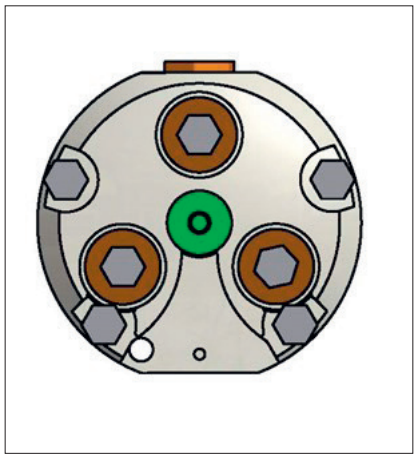
07

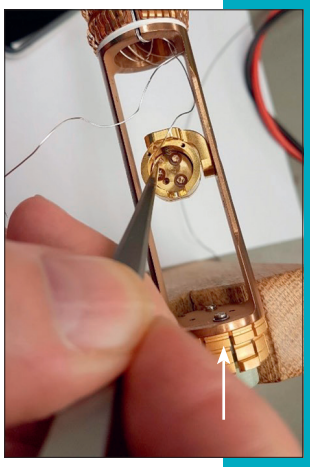
CLEANING THE DILATOMETER



It can happen that sample particles or dirt get in between the two capacitor plates and therefore create a short circuit. In such a case, the dilatometer has to be cleaned. First try to remove the sample particle's by cleaning the space between the two capacitor plates using a piece of paper. The paper can be soaked with Isopropanol.

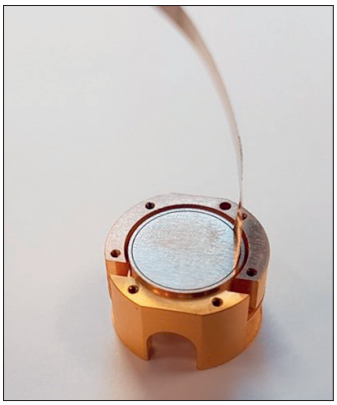
If this method does not work, unscrew the sample-adjusting tool and the cover. Unscrew the four outer bottom screws, but not the three inner screws, which are insulated from the dilatometer body by vespel parts and hold the lower capacitor plate.



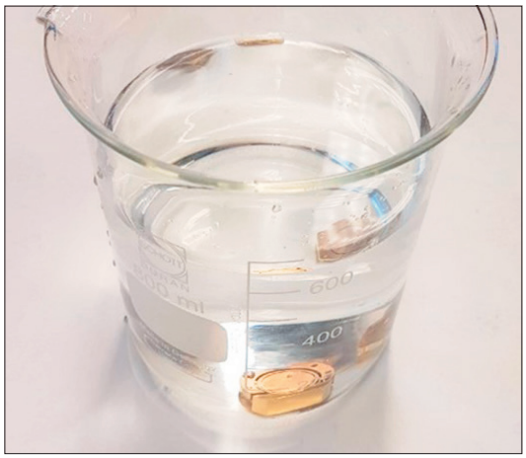


Then unsolder the coax-wires from its electrical connection

Caution: During unsoldering, please hold coax-wires with a tweezer in order to release the heat through the tweezer but not through the coax-wires. Otherwise, the coax-wires can be damaged.

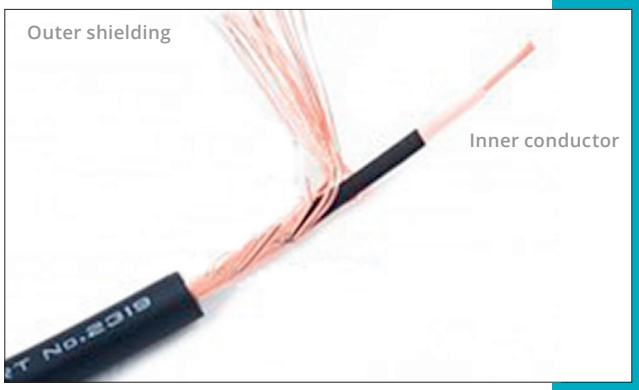


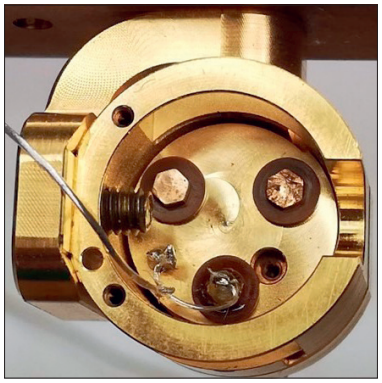
Now the gap between the capacitor plates and its surrounding guard rings can be cleaned by using a very thin and precisely cut metal foil. Use a microscope to check the cleanness of the dilatometer part. If after cleaning there is still a short circuit in between the plate and the cell body, some very small particles of dirt could be hidden below the capacitor plate. Then the dilatometer part has to be cleaned in an Ultrasonic cleaner.

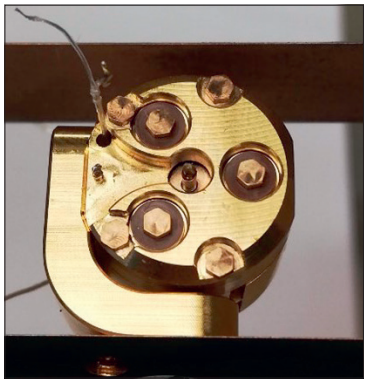


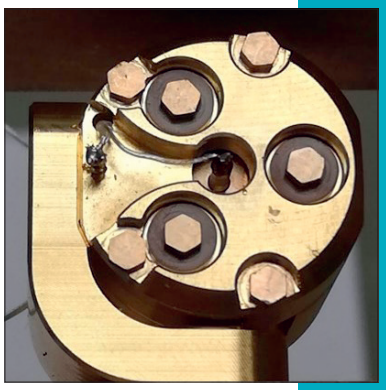
Place the corpus or the lower dilatometer part carefully at the bottom of a large glass (Hereby the capacitor plates should look upwards) and fill carefully up the glass with Isopropanol. Put the filled glass in an Ultrasonic cleaner and clean the dilatometer parts 30 minutes. Take care that during cleaning the glass cannot overturn since in this case the polished capacitor plates could be damaged. Finally gentle rinse the cleaned parts in calcium- and salt-free water and let the parts dry completely.

Finally solder the two inner conductor of the coax-wires to its electrical connections (screw in left picture and pin in right picture) and the outer shielding of the coax-wires to the cell-ground.









08

CALIBRATION OF THE CELL BACKGROUND

The use of different materials in the dilatometer assembly leads to a temperature-dependent background due to different thermal expansion coefficients. We minimized this effect in our cell design, where nearly all components are machined from Be-Cu alloy. The only exceptions are sapphire washers and electrically insulating parts made of vespel. In the following, the remaining cell background is calibrated by a reference measurement of a copper sample. In a thermal expansion experiment, both the sample length and the length of the dilatometer cell itself vary with temperature. The measured length change of the sample ΔL^{sample}_{meas} is the difference between the actual length change ΔL^{sample} of the sample and the length change of the cell ΔL^{cell}:

$$\Delta L^{sample}_{meas} = \Delta L^{sample} - \Delta L^{cell}$$

To calibrate the cell effect ΔL^{cell}, we measure the thermal expansion of a reference sample with a thermal expansion coefficient close to that of the cell body material $Cu_{0.98}Be_{0.02}$. We can use high-purity copper (99.999 %) as a reference, where the thermal expansion in the relevant temperature range is well known from literature [F. R. Kroeger and C. A. Swenson, J. Appl. Phys. 48, 853–864 (1977)].

$$\Delta L^{Cu}_{meas} = \Delta L^{Cu}_{lit.} - \Delta L^{cell}$$

The cell effect ΔL^{cell} is therefore the difference of the length change of the copper sample $\Delta L^{Cu}_{lit.}$ and the measured copper sample:

$$\Delta L^{cell} = \Delta L^{Cu}_{lit.} - \Delta L^{Cu}_{meas}$$

The cell effect results mainly from the fact that by measuring a sample with a length L_0 a respective part of the BeCu cell of the same length is missing. The cell effect is quite small, the thermal expansion of the cell deviates only slightly from that of a block of pure copper, demonstrating the high quality of the cell. To obtain the true length change of a sample measured with this dilatometer, the calibrated cell effect is added to the measured length change ΔL^{sample}_{meas}:

$$\Delta L^{sample} = \Delta L^{sample}_{meas} + \Delta L^{cell}$$

$$\Delta L^{sample} = \Delta L^{sample}_{meas} - \Delta L^{Cu}_{meas} + \Delta L^{Cu}_{lit.}$$

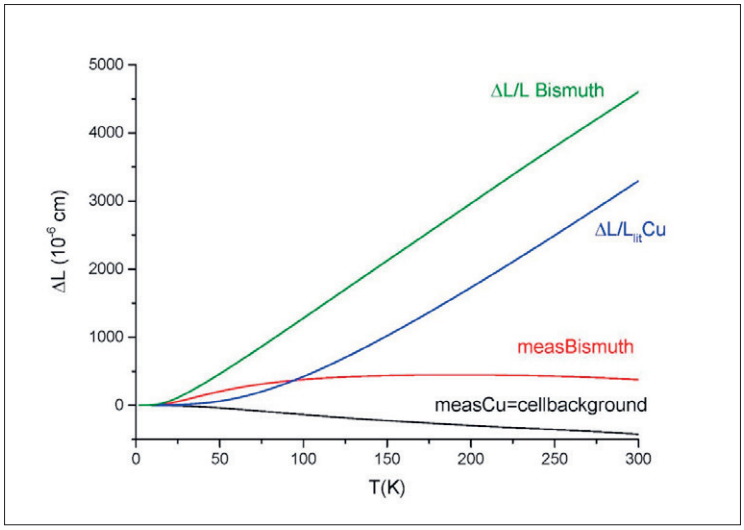
The relative length change of the sample normalized to its room temperature length L_0 is therefore given by:

$$\frac{\Delta L^{sample}}{L_0} = (\Delta L^{sample}_{meas} - \Delta L^{Cu}_{meas})/L_0 + (\Delta L/L)^{Cu}_{lit.}$$

where the last term represents the literature value for the relative thermal expansion of pure copper, which is independent of the sample length.

In the following, we show an example of a large bismuth single crystal measurement. Note that the copper sample must have the same length as the investigated sample:

$$\frac{\Delta L^{Bismuth}}{L_0} = (\Delta L^{Bismuth}_{meas} - \Delta L^{Cu}_{meas})/L_0 + (\Delta L/L)^{Cu}_{lit.}$$

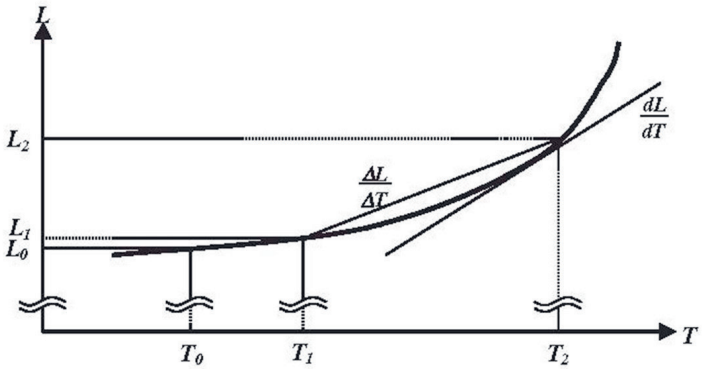


The cell background of the dilatometer is linear with the measured copper sample length. Therefore, only a small (0.5 mm) and a large (2 mm) copper sample have to be measured for the background subtraction. Then, all other sample length between them can be calculated. This is explained in detail in the appendix.

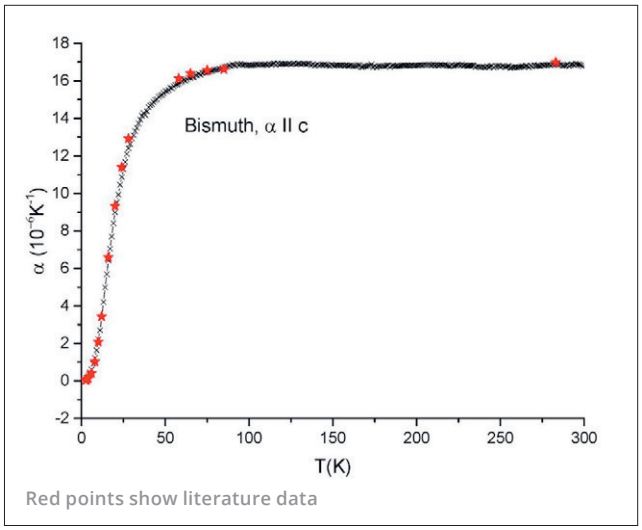
From the resulting $\frac{\Delta L^{Bismuth}}{L_0}$-data the thermal expansion coefficient

$$\alpha(T) = \frac{1}{L_0}\,\frac{d\,\Delta L(T)}{dT}$$

can be calculated based on an interval derivative using an interval in between 0.1 up to 0.5 K:



$$\alpha_1 = \frac{(L_2 - L_1)/L_0}{T_2 - T_1} \qquad \text{where } T_m = \frac{T_1 + T_2}{2}$$



Calculated thermal expansion coefficient using a derivative interval of 0.5 K.