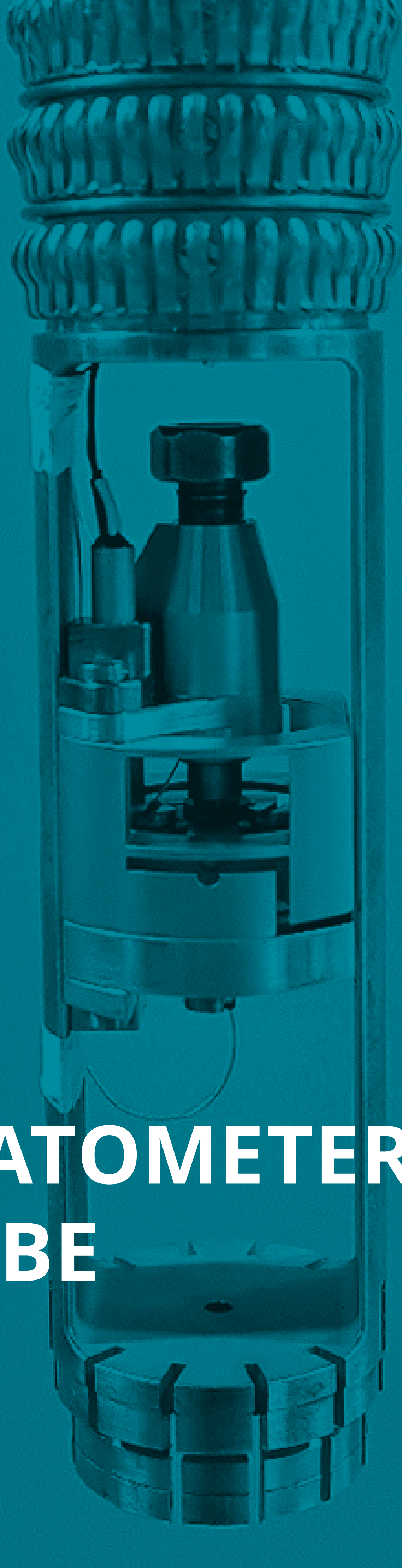
KUECHLER

Innovative Measurement Technology



MANUAL
STRESS-DILATOMETER PPMS – PROBE

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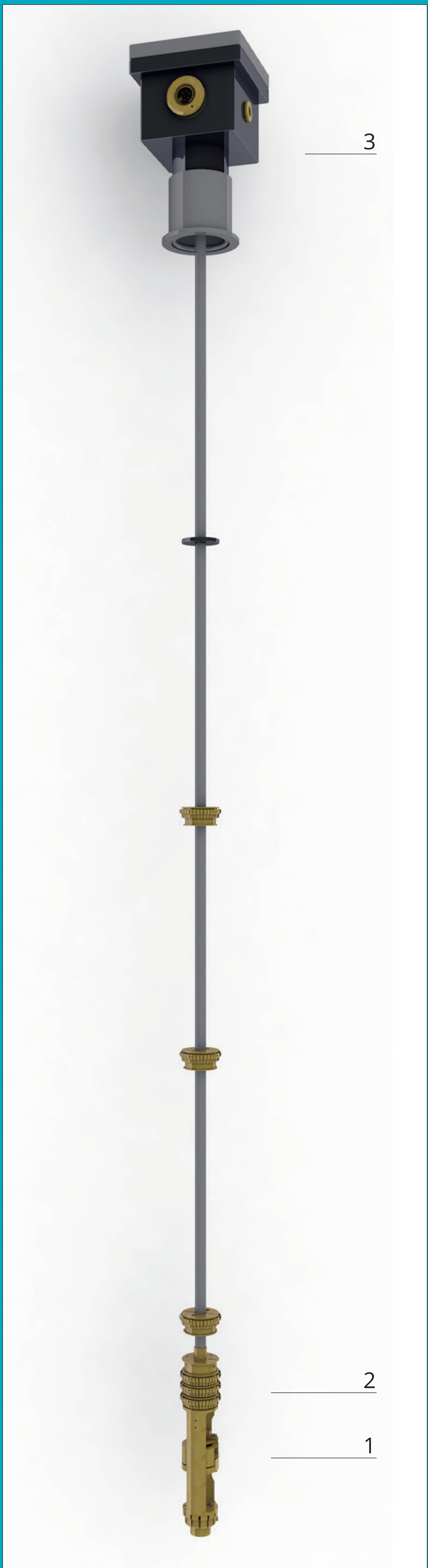
01

STRESS-DILATOMETER-PPMS-PROBE

The stress-dilatometer is mounted and cabled within our special dilatometry PPMS-insert. This type of insert is designed and developed for best possible dilatometry measurements and can be used in PPMS systems.

INTRODUCTION

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 where they touch the inner chamber of the PPMS cooling channel. Only the lowest part of the inner PPMS cooling channel is made of very good heat conducting 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 stress-dilatometer. The ultra-thin coaxial cables and the twisted Pair Beryllium Cooper Loom are wrapped around measurement probe and anchors to reduce a heat leak. The probe also contains radiation shields to prevent additional heating. A second temperature can be measured using a Cernox resistance thermometer, which is enclosed in the dilatometer holder, near the sample. The head of the probe (3) contains one hermetic sealed Fischer- and two Lemo-connectors, which can be connected with the delivered cables. The stress-dilatometer PPMS insert 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. An additional resistance bridge is not required. Connecting the probe's-PPMS cable allows reading the internal PPMS resistance bridge.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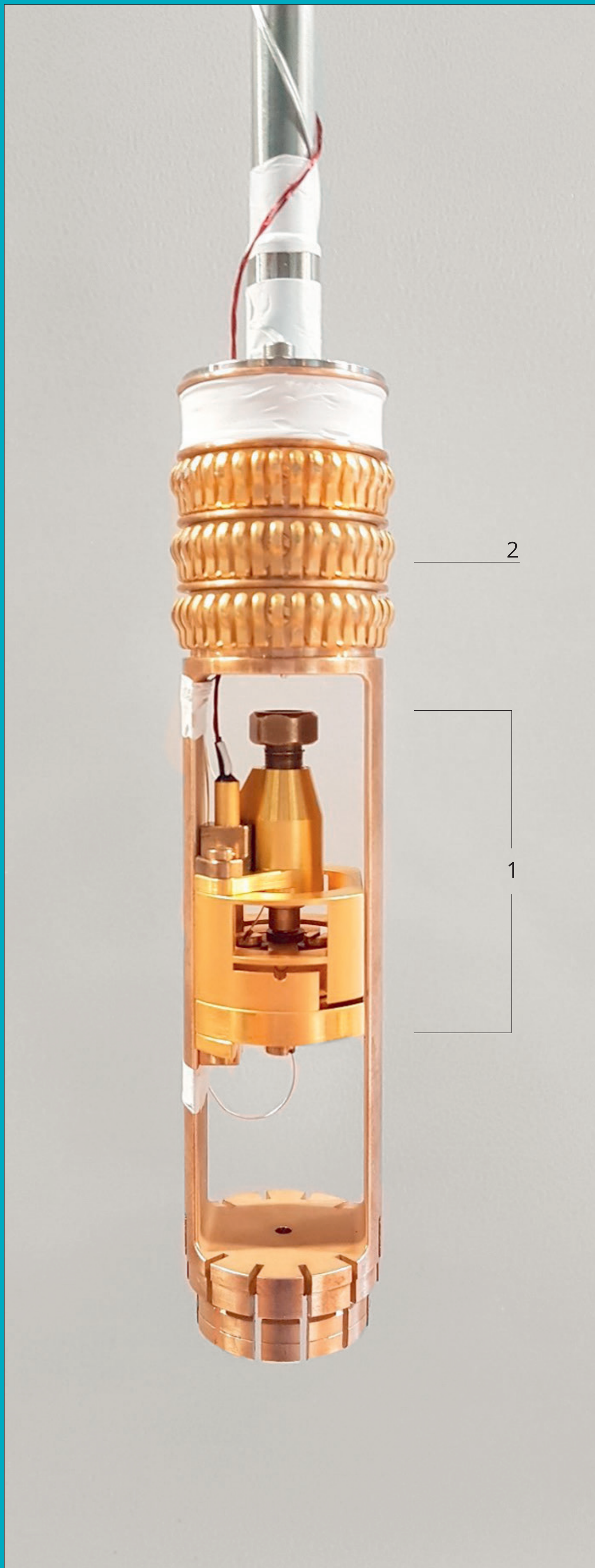
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02

SPECIAL DILATOMETRY PPMS INSERT

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

1 Computer or Notebook

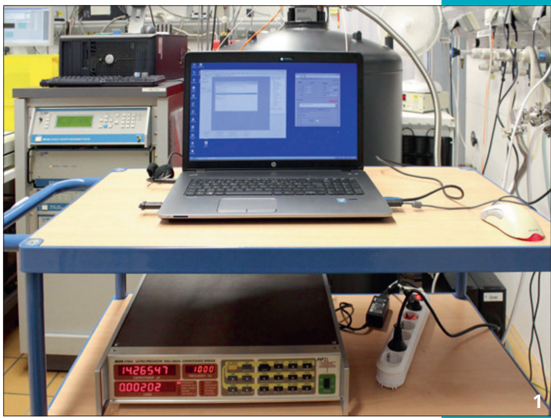
2 Capacitance Bridge
We suggest using the Andeen Hagerling 2550 A (1 kH) or its predecessor model AH 2500 A (1 kHz), or at least a ultra-precision capacitance bridge.

3 GPIB-USB-HS Converter
To connect the measuring bridge with the computer, we use the National Instruments converter.

4 Probe – PPMS connecting cable.
Is included in the delivered PPMS dilatometry probe package.

5 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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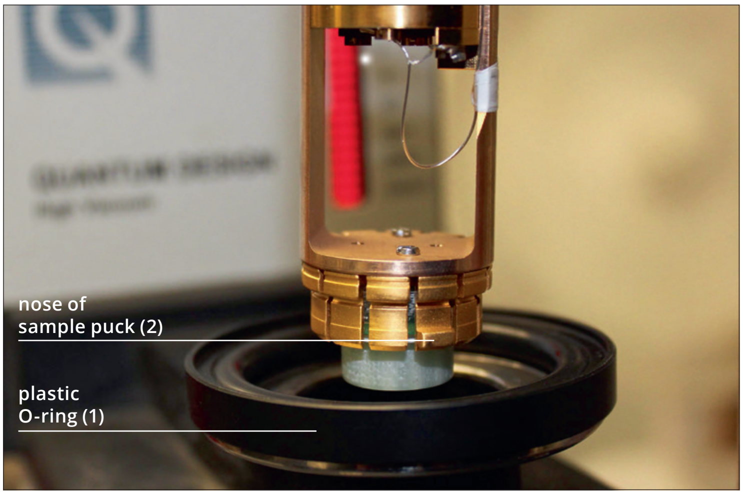
03

DILATOMETRY MEASUREMENT SET-UP

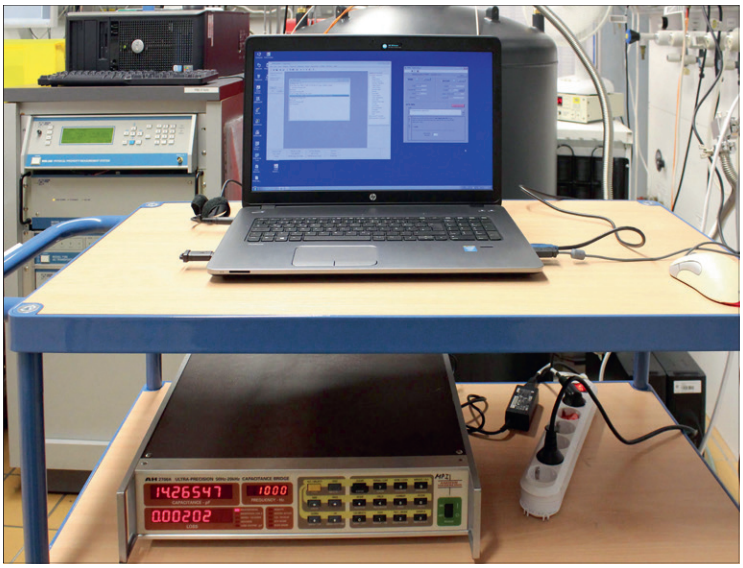
THE PPMS-DILATOMETRY MEASUREMENT SET-UP

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.

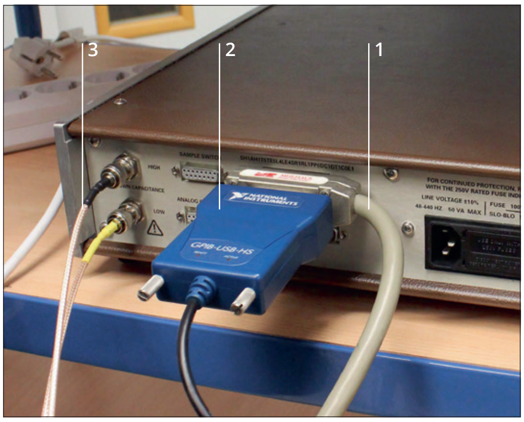
The flange on the probe head glides over a rubber seal ring and can carefully be moved up and down to adjust the required probe height.

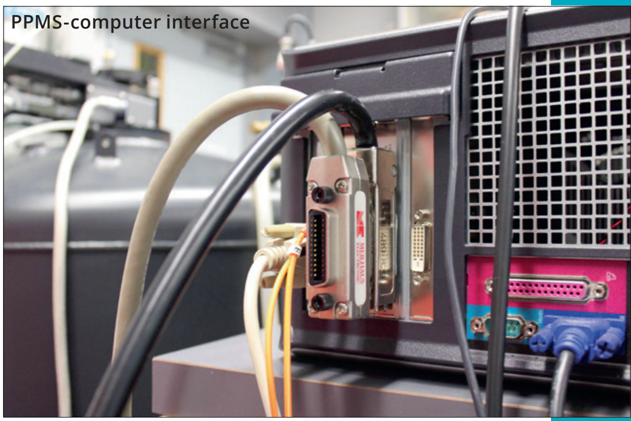


Place your computer and capacitance measuring bridge on a suitable rack.



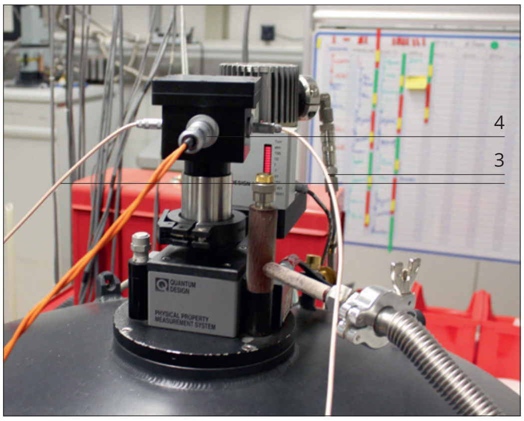
Connect the capacitance measuring bridge by means of a standard GPIB-cable (1) to the PPMS-computer interface and via GPIB-USB-HS converter (2) to your computers USB-interface.

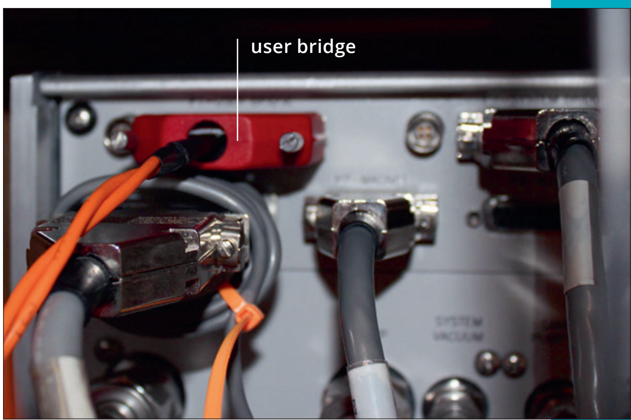




Connect the Capacitance measuring bridge by means of the delivered pair of capacitance bridge-probe coax cable (3) with the head of the PPMS-probe.

Connect the delivered PPMS-probe cable (4) to the PPMS-user bridge (5).





NEWER TYPE OF PPMS

USB-CAN module

noise-surge-protector

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.

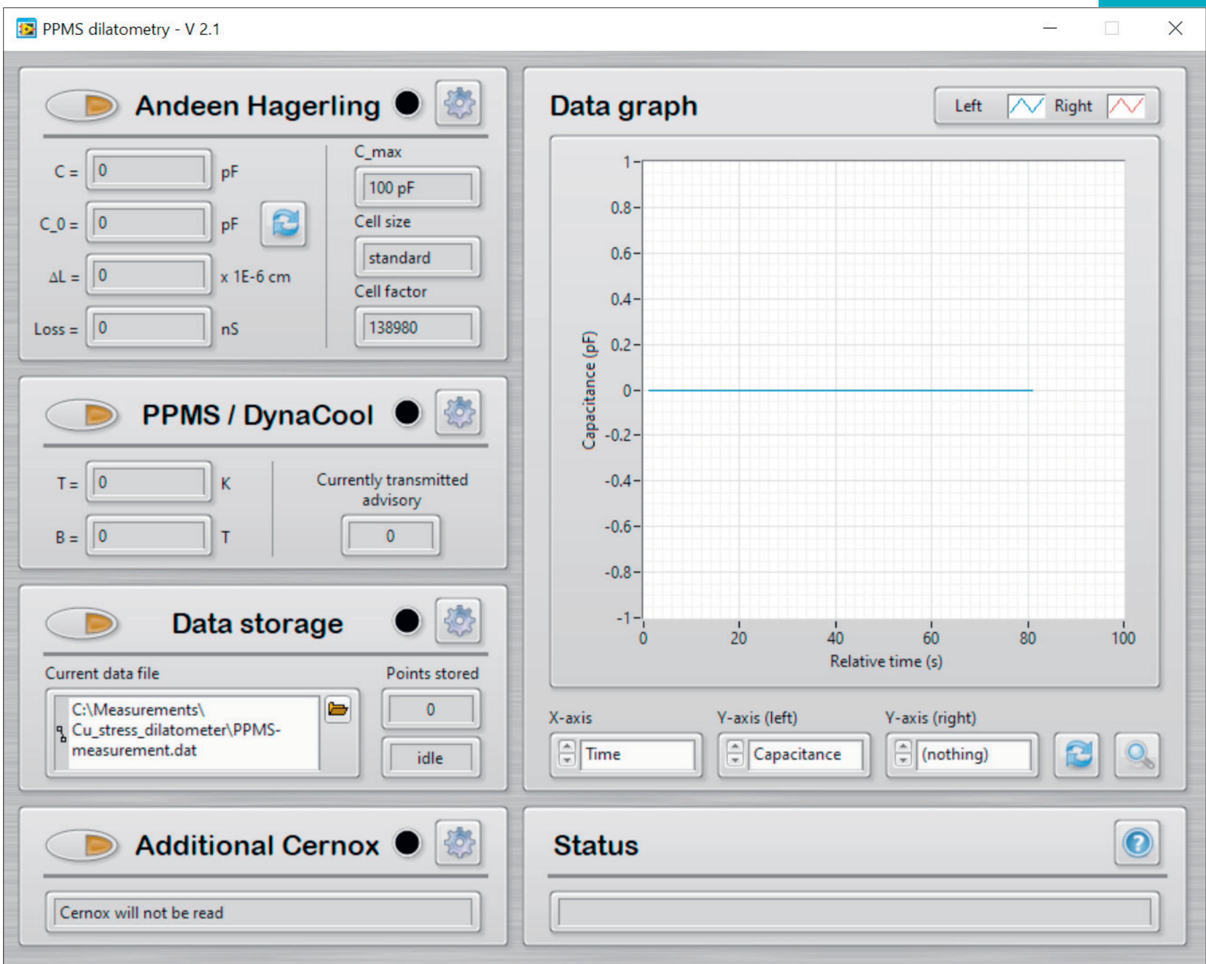
In case the electric power supply in your lab is not filtered properly, we suggest using a noise-surge-protector. To reduce noise during the measurement, all devices of your measurement set-up (like the capacitance measuring bridge and your computer) should be plugged in to the noise-surge-protector.

04

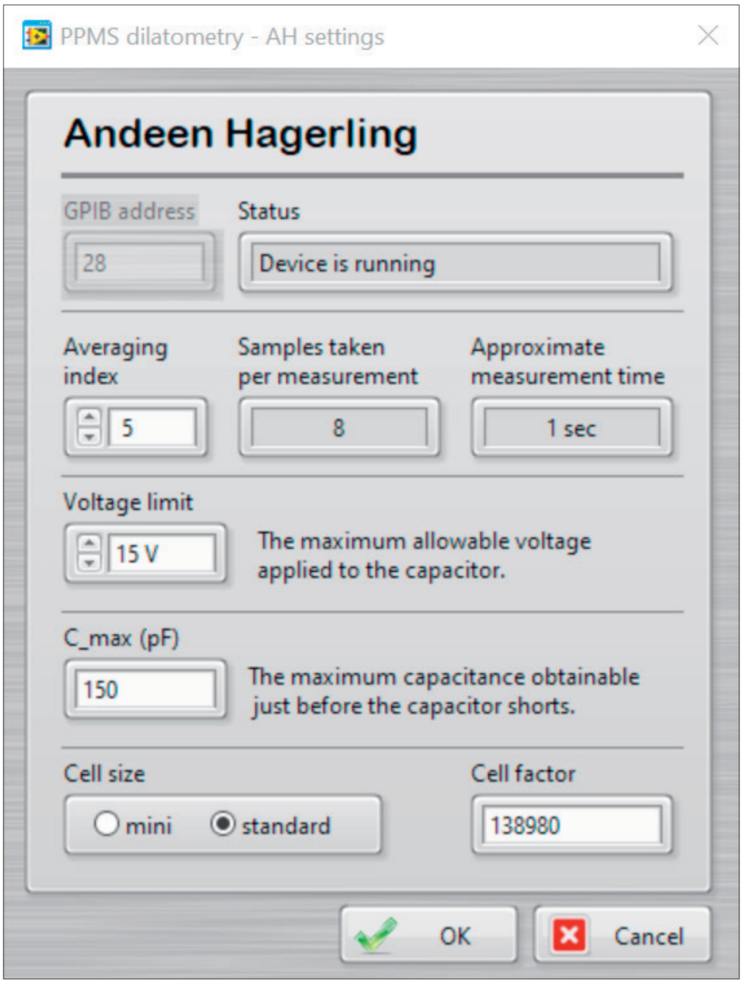
PPMS DILATOMETRY SOFTWARE INSTALLATION

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:

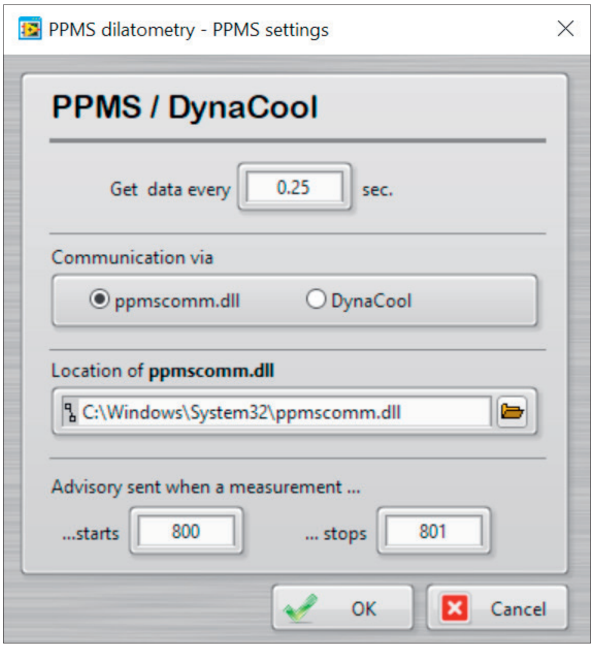
The voltage limit allows you to specify a maximum signal level to be applied. The bridge will measure the sample at a voltage equal to or lower than this maximum. The bridge will report the actual voltage at which the measurement is taken. The maximum voltage limit of 15 V produces the least noise effect.

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

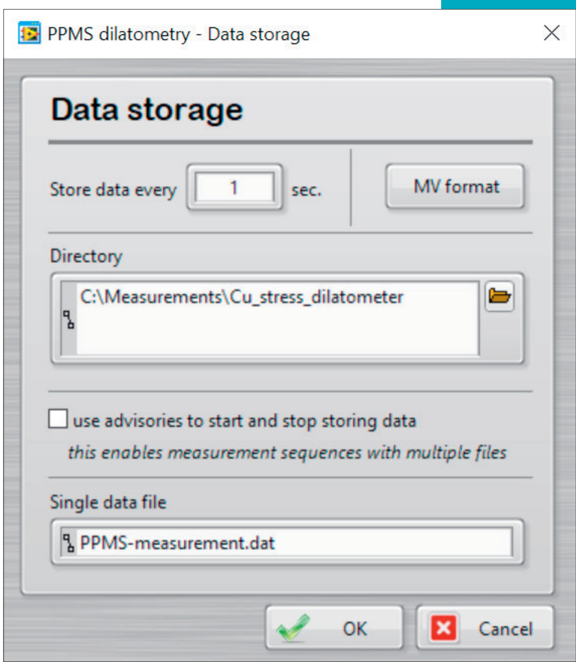
The number shown in the first window determines the number of seconds after which data from the PPMS will be transmitted.

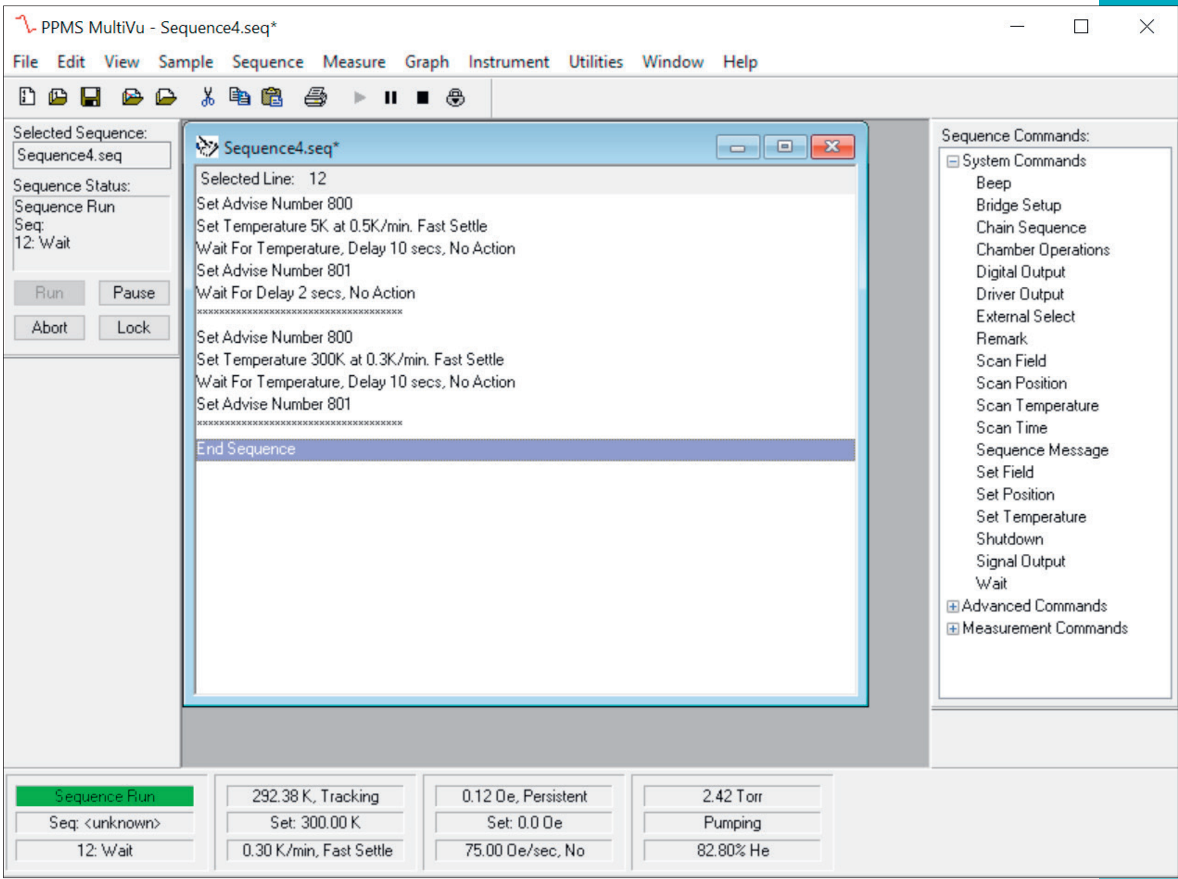
DATA STORAGE:

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.

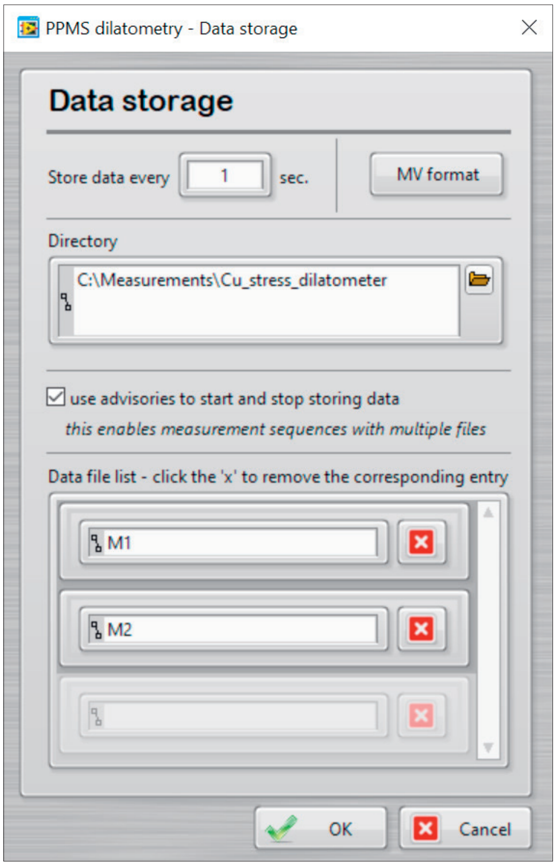
The Directory defines the place where the data will be collected.

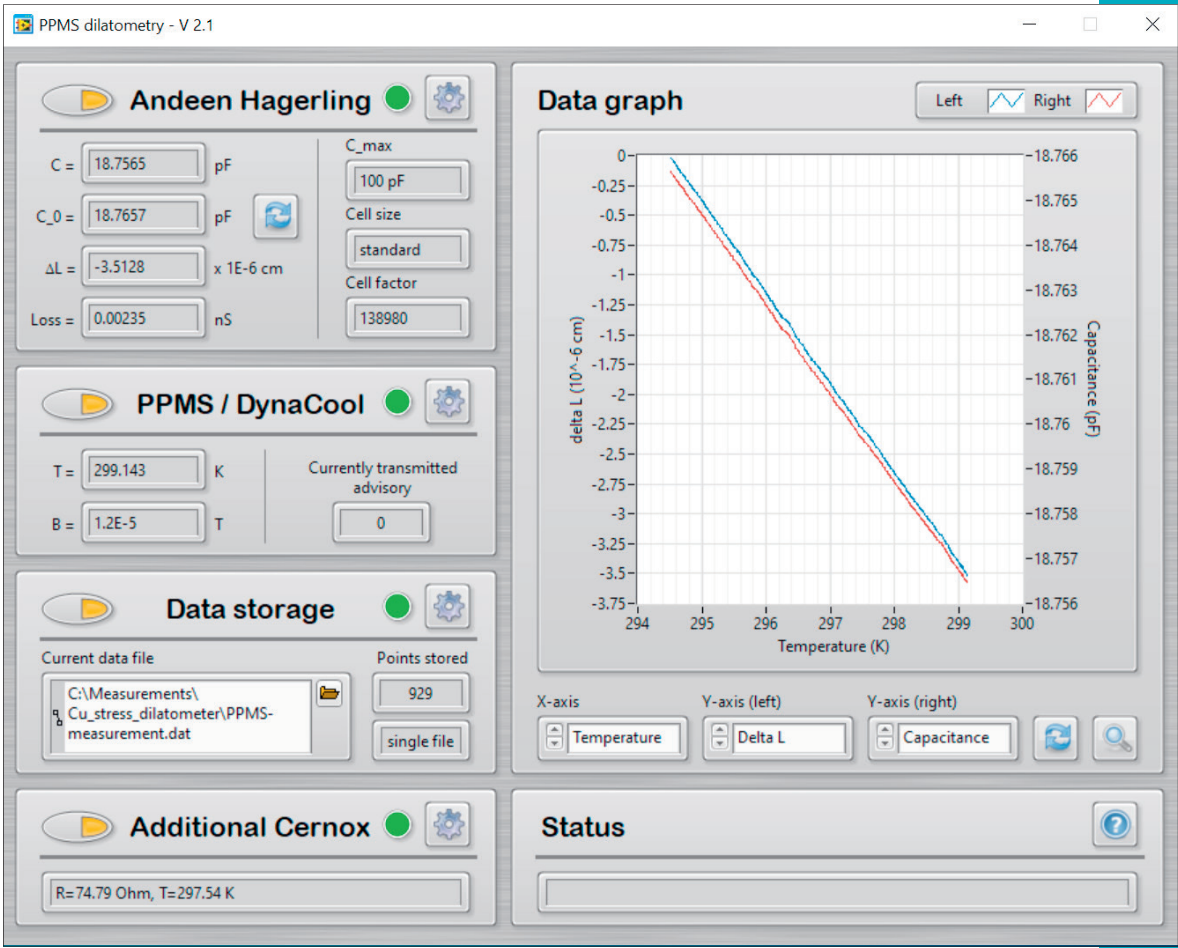
It is possible to select either a single data file or measurement sequences with multiple files. For the latter, you have to use MultiVu advisory to start and stop storing data. At first, the appropriate sequence has to be written with MultiVu. Note that each time two advisory have to be set. The first 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:

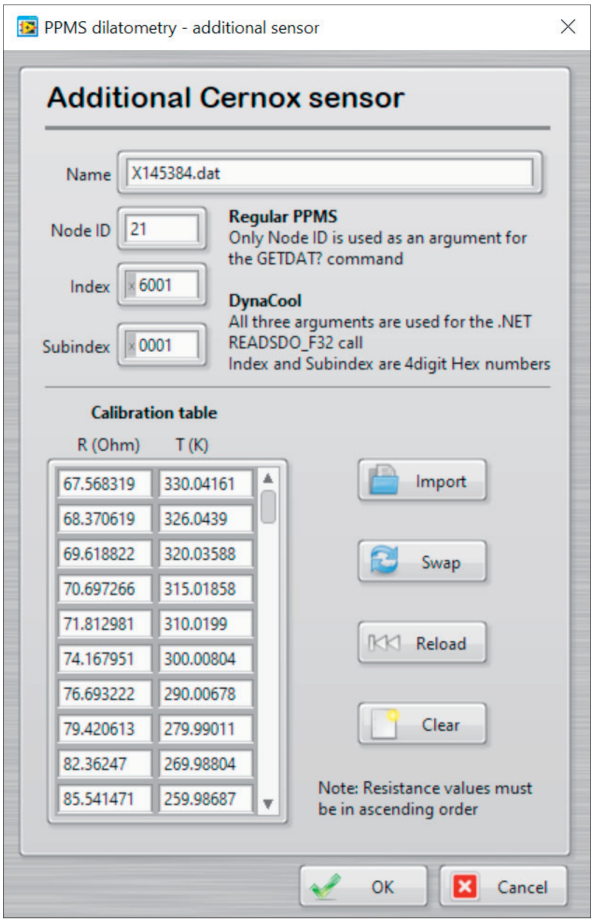
$$\Delta L = \varepsilon_0 \pi r^2 \frac{C - C_0}{C \cdot C_0} \; 1 - \left(\frac{C \cdot C_0}{C_{max}{}^2} \right)$$

4. Loss [nS] indicates the measured loss

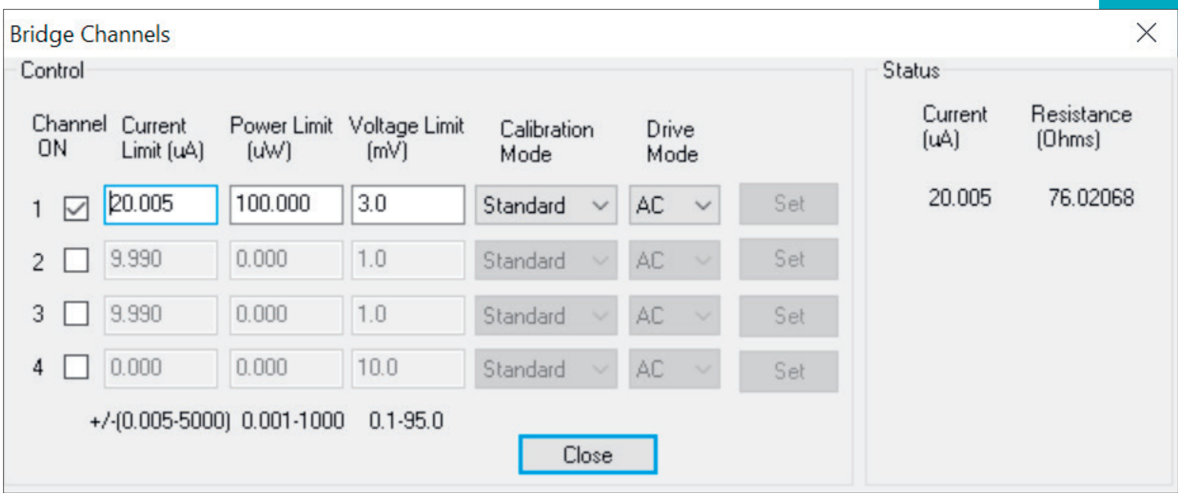
From PPMS:

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

ADDITIONAL CERNOX SENSOR:



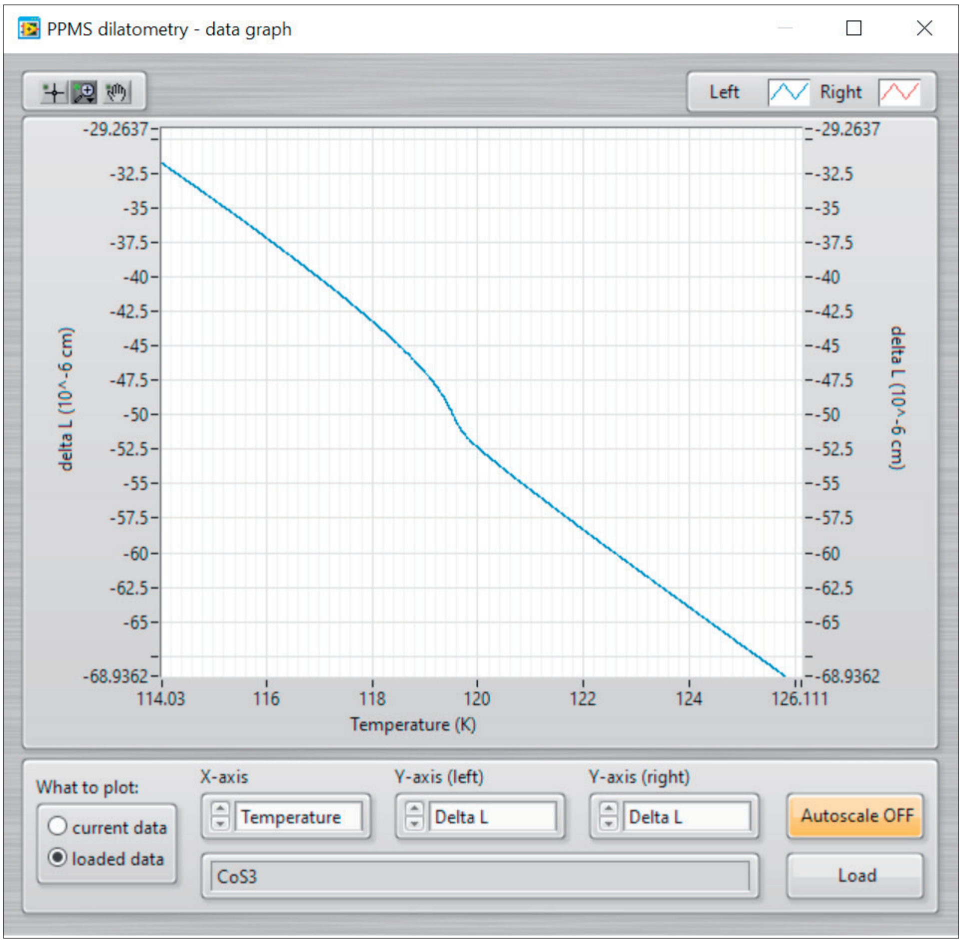
The standard- and stress-dilatometer probes contains an additional Cernox-resistance sensor, which is enclosed in the dilatometer holder, near the sample. The readout of this additional Cernox thermometer can be carried out by using the resistance bridge integrated in the Quantum Designs PPMS-system. 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:

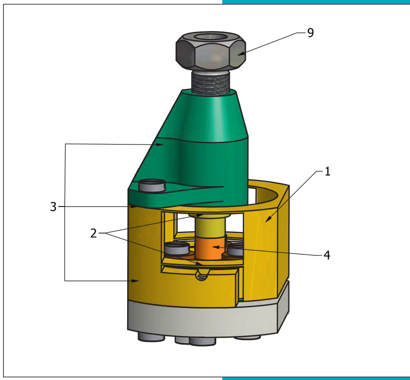
The best results can be obtained by sweeping up the temperature slowly with 0.3 K/min or comparable sweeping rates. A slow sweeping rate ensures that the temperature gradient within the dilatometer and the sample is small. Cooling down the PPMS-system is not working well at lower temperature using the stress-dilatometer probe.

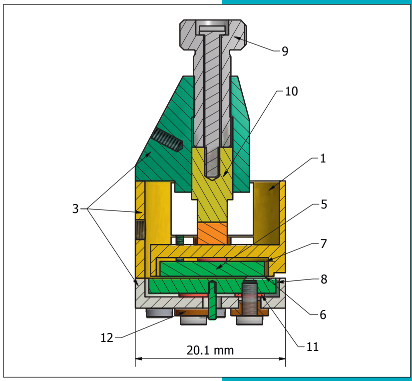
For the magnetic field sweeps, we suggest to use the lowest possible sweep rate.

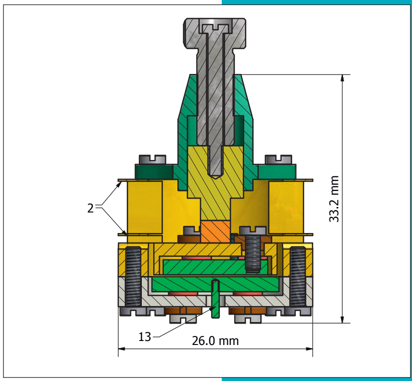
05

MEASURING PRINCIPLE

We have designed the uniaxial stress dilatometer based on our patented miniature dilatometer which operates on the principle of two parallel flat springs developed by Pott and Schefzyk. While the Pott-Schefzyk dilatometer was assembled from ten main parts, our innovation was to produce the corpus of the cell from a single piece of BeCu, using milling and spark erosion. New modifications allow to apply a substantial uniaxial stress. A schematic of the uniaxial stress dilatometer is shown in the figure. All parts (except some insulating spacers) were fabricated from high-purity beryllium copper to minimize eddy current heating during magnetic field sweeps. The main body (yellow part of Fig. 1) contains the mobile part (1), both springs (2), as well as the middle part of the housing (3). While the lower capacitor plate (6) is part of the housing (3), the upper capacitor plate (5) is fixed to the mobile part (1), which is held in the frame by two very thick 0.7 mm BeCu leaf springs (2). The sample is held between the piston (10) and the mobile part including the upper capacitor plate (5) and can be tensioned by the adjustment screw (9). A length change of the sample (4) causes an equivalent displacement of the upper capacitor plate with respect to the lower one and induces a change of the capacitance. Samples of less than 1 mm and up to 6 mm length can be measured. The two capacitor plates are electrically isolated by pieces of vespel (12) and 0.5 mm sapphire washers (11) and are surrounded by guard rings (7, 8) to avoid stray electric fields. For both the lower (6) and the upper capacitor plates (5), three BeCu screws are used to mount the plates to the guard rings (7, 8). Before assembling the dilatometer, the capacitor plates were polished within their frames. A uniform surface of the plates within their frames is most essential to achieve best parallel orientation of the plates. In its rest position, the capacitance of the dilatometer is about 6 pF, corresponding to a distance of 0.25 mm between the capacitor plates. After mounting the sample, the adjustment screw (9) is used to reduce this distance to about 0.07 mm, which corresponds to a capacitance of 20 pF. The absolute value of the capacitance is measured by a commercial capacitance measuring bridge (Andeen Hagerling 2550 A) with a resolution of 10^{-6} pF, which corresponds to a relative sensitivity $\Delta L/L \approx 10^{-8}$ for a sample length in between 1 and 6 mm. Our new design allows measurements under substantial uniaxial stress. Compared to the original “almost zero pressure” (AZP) cell (standard dilatometer), three fundamental changes were made. (a) Most







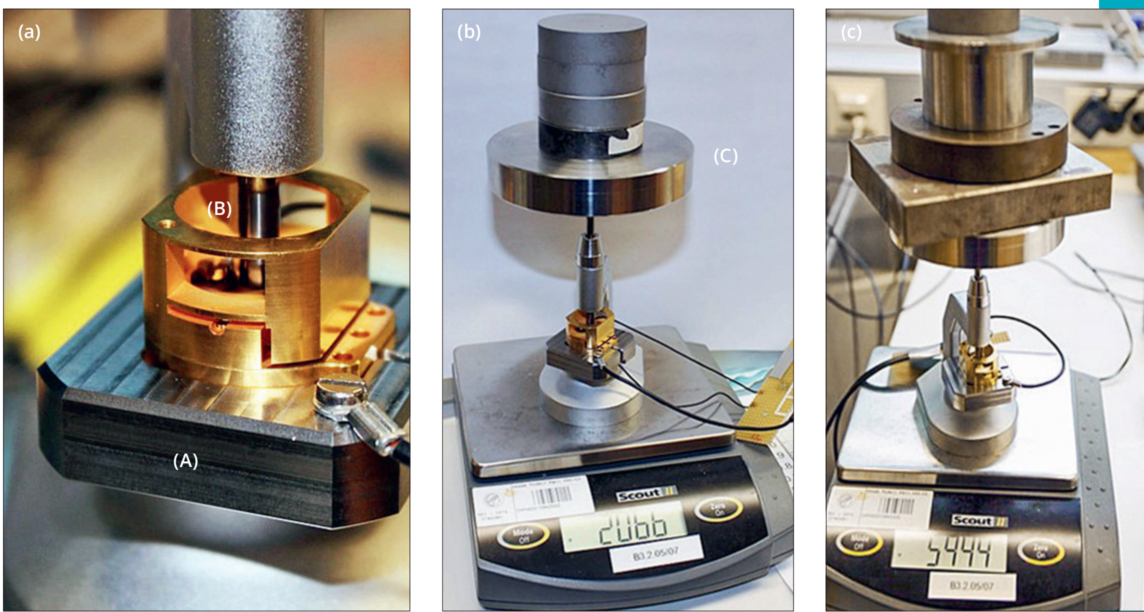
Uniaxial stress capacitive dilatometer in three-dimensional (upper), side cut-away (middle) and front cut-away (lower) views. The numbers illustrate:
(1) mobile part
(2) 0.7 mm thick Be–Cu flat springs
(3) housing
(4) sample
(5) upper capacitor plate
(6) lower capacitor plate
(7) upper guard ring
(8) lower guard ring
(9) adjustment screw
(10) piston
(11) sapphire washer
(12) insulating piece of vespel
(13) electrical connection.

important the thickness of the two leaf springs has been increased significantly from 0.25 mm to 0.7 mm. (b) The adjustment screw holder is bigger and more robust so that the entire construction can withstand the applied forces. (c) The cylindrical adjustment screw (9) contains an ovally shaped piston (10), which is fixed to the head of the screw. This prevents the piston from rotating during adjustment and thus avoids damage to the sample. Due to the substantial applied uniaxial stress, the adjustment screw can only be tightened by a wrench.

06

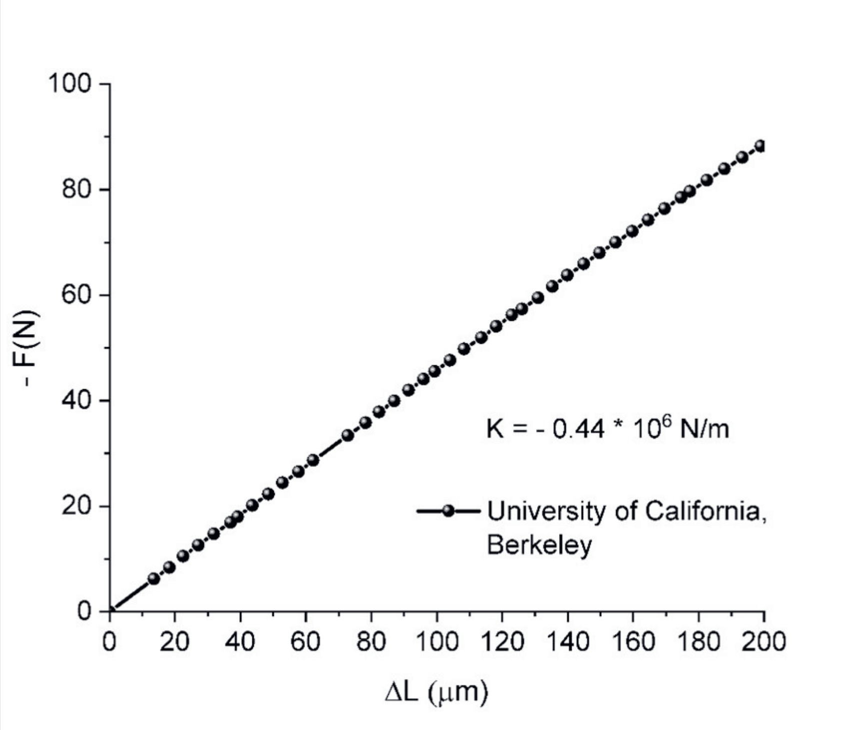
SPRING FORCE EXERTED ON THE SAMPLE

During measurements, the sample is clamped between a movable and a fixed plate by springs that exert an enormous force to the sample. The applied stress can be calculated by dividing the force by the sample cross section. By measuring crystal of rectangular shape with the same cross section at both ends, a uniform stress along the sample is guaranteed, which is an important advantage compared to piezostrain devices. **Therefore, we suggest cutting samples in that way that the sample shape becomes rectangular.**



The figure above shows photos of the setup used to determine the applied spring force. A dilatometer holder plate (A) is mounted on a tripod foot. The tripod contains a guide slot in which a pole (B) is inserted. By placing weights on the disk (C), significant force can be applied via the pole to the mobile part of the cell. The setup is placed on a scale to measure the entire load. The mobile part of the cell was forced down stepwise with weights up to 10 kg. The resulting length change ΔL was calculated from the measured capacitance using the equation for a plate capacitor (see Fig. 2 of Rev. Sci. Instrum. 87, 073903 (2016)).

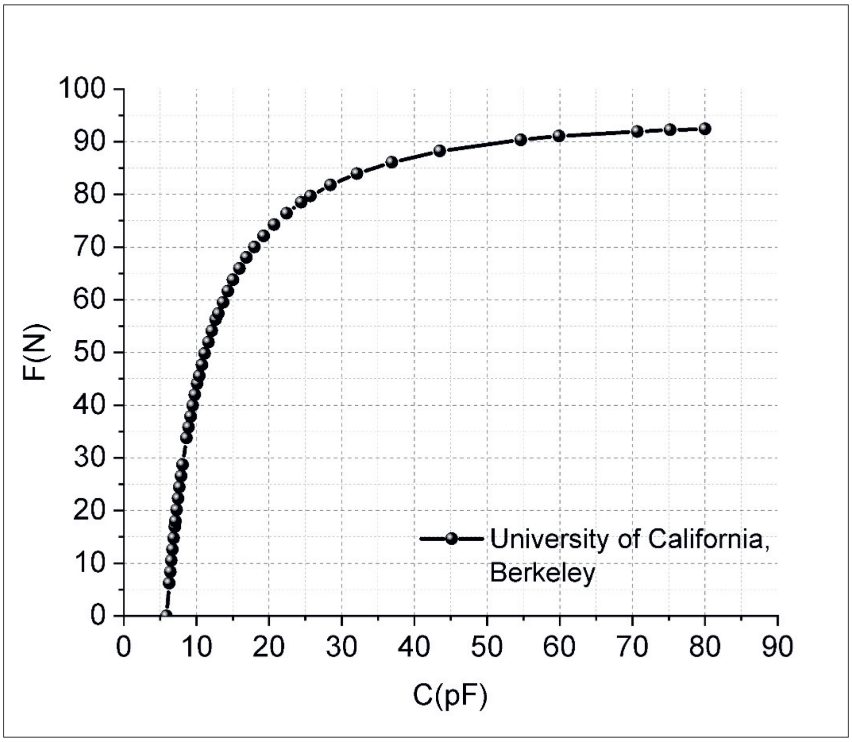
Next Figure shows the obtained linear relation between the length change and the applied force.



Relation between the applied spring force and the displacement of the upper capacitor plate from its rest position, 200 µm above the lower plate. Hooke's law F = k × ΔL with spring constant as quantified.

From this we derived a spring constant $k = -0.44 \times 10^{-6}\ Nm^{-1.}$ This observation of a linear relation is significant as it proves that the spring strain is within the elastic regime for the used springs of 0.7 mm thickness.

This means that the uniaxial stress dilatometer works over the whole range of operation from 10 N up to 75 N. Since the elastic modulus of copper alloys remains almost constant from room temperature to below 1 K, this value is applicable to the entire temperature range from 2 K up to 320 K. The determined relation between the force and capacitance is shown in the figure below.



Spring force as function of working capacitance. The cell is typically operated in between 20 and 25 pF, corresponding to forces between 70 and 80 N.

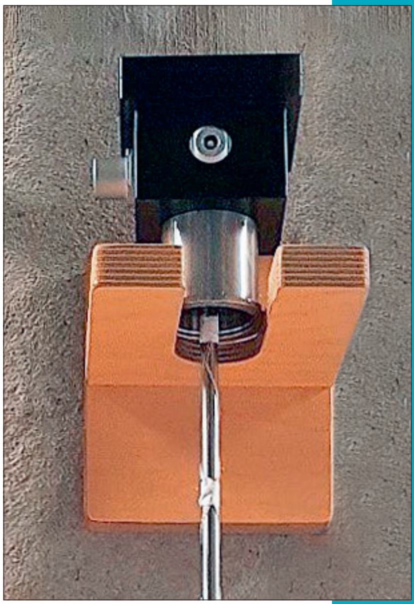
As the dilatometer is typical operated between 15 and 25 pF, a spring force of 60 to 80 N is obtained. This corresponds to a maximal uniaxial stress (p = F/A) of 0.80 and 3.2 kbar considering a cuboid sample with $(1\ mm)^2$ and $(0.5\ mm)^2$ cross section, respectively.

The PPMS-Dilatometry-Software allows reading all measured capacitances C (pF). The applied force and uniaxial stress can be determined by using the calibrated F (N) – C (pF) – curve.

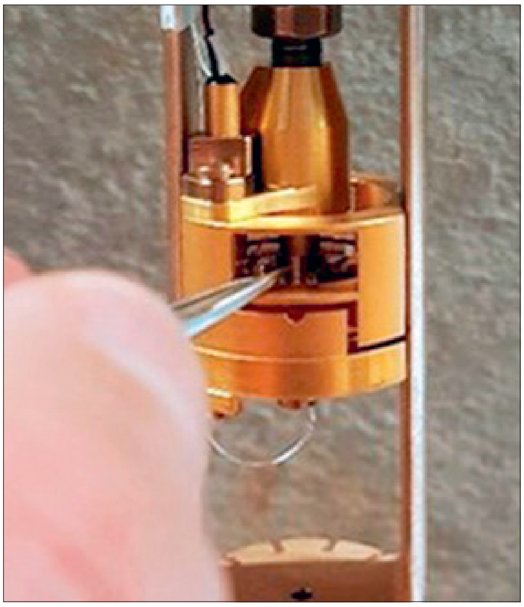
07

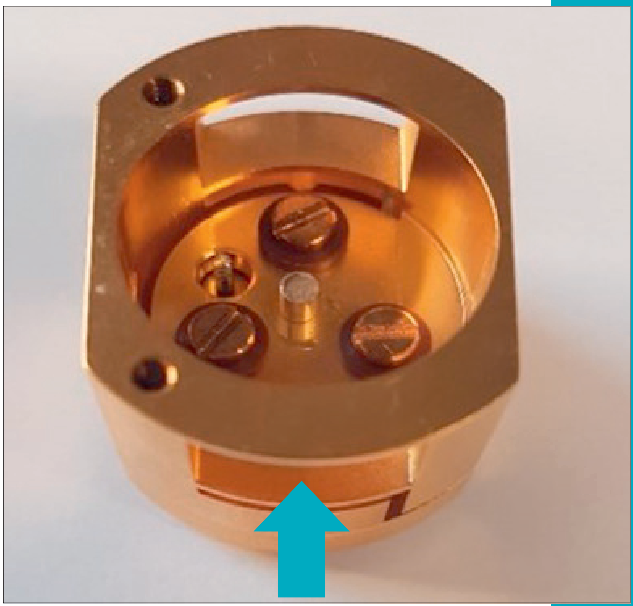
SAMPLE MOUNTING OF STRESS DILATOMETER

First, cut your sample in that way that the sample shape becomes rectangular.
Before you start sample mounting put your probe in a stable upright position. Then, 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 5.8 pF, consistent with the first data point in the F (N) vs. C (pF)-curve for F (N) = 0 (see the last chapter's last figure).

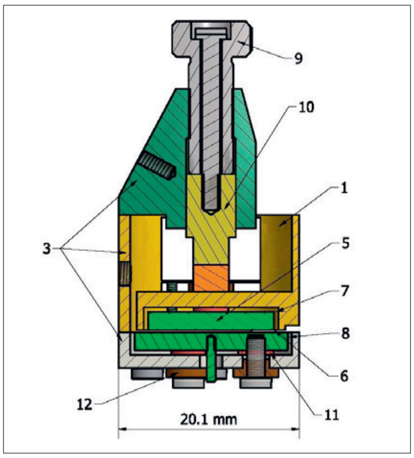


By means of a tweezer, the sample can be inserted into the dilatometer. To place the sample inside the dilatometer, choose the side where no screw is in the way obstructing access.

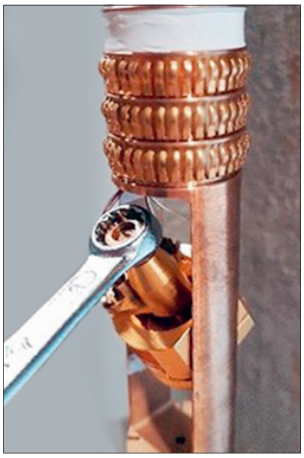


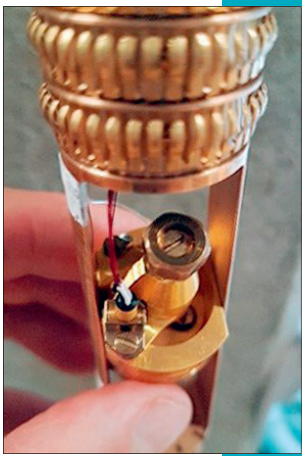


Then, the adjustment-screw can carefully be tightened using your fingers until the sample is clamped. That is when you will notice a small increase in the measured capacitance.



The cylindrical adjustment screw (9) contains an ovally shaped piston (10), which is fixed to the head of the screw. This prevents the piston from rotating during adjustment and thus avoids damage to the sample. To apply further uniaxial stress, the adjustment screw has to be tightened using a wrench.

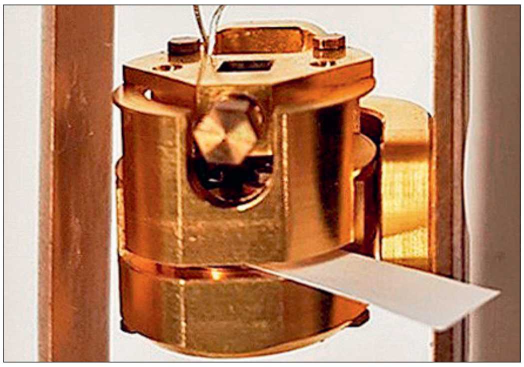




In case there is not enough space to use a wrench, carefully loosen the screws which holds the dilatometer in place. Then slightly rotate the dilatometer. Make sure that the screw is tightened again eventually.

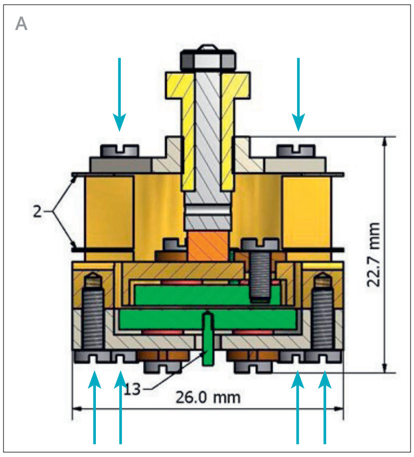
08

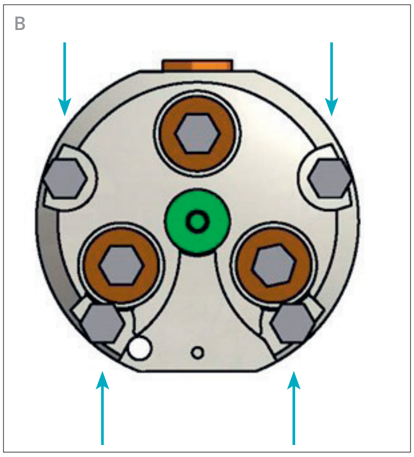
CLEANING THE DILATOMETER

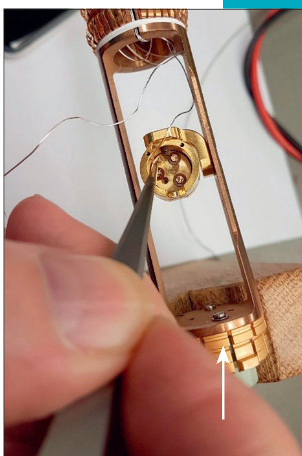


Sample particles or dirt may get in between the two capacitor plates and can thus create a short circuit. In such a case, the dilatometer has to be cleaned. First, try to remove the sample particles by cleaning the space between the two capacitor plates using a piece of paper. The paper can be soaked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

If this method does not work, unsolder and unscrew the dilatometer. Loosen the two upper screws (A) and remove the cover including the adjustment-screw. Then unsolder the coax-wires from their electrical connection (13). Unscrew the four outer bottom-screws (B), but not the three inner screws, which are insulated from the dilatometer body by vespel parts and hold the capacitor plate.

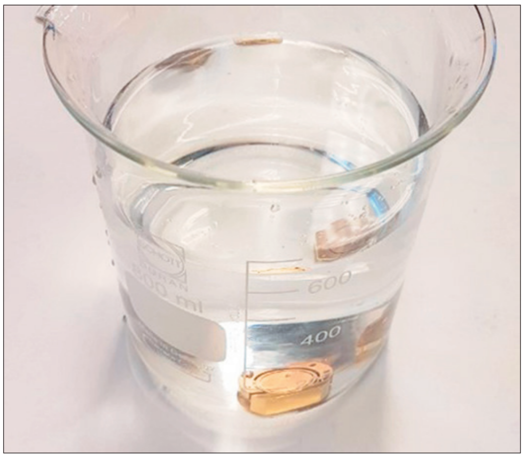






Caution: During unsoldering, please hold the coax-wires using a tweezer, so that heat is realeased via the tweezer and not via the coax-wires, which might otherwise get damaged.

Now, the gap between the capacitor plates and its surrounding guard rings can be cleaned using a very thin brass foil. If, after the cleaning, you should still get a short circuit in between the plate and the cell body, some very small particles of dirt might be hidden below the capacitor plate. The dilatometer part then has to be cleaned in an ultrasonic cleaner.



Carefully place the corpus or the lower dilatometer part at the bottom of a large glass (The capacitor plates should face upwards) and cautiously fill the glass with Isopropanol. Put the full glass in ultrasonic cleaner and leave the dilatometer parts to be cleaned for 30 minutes. Make sure that, during cleaning, the glass cannot tip over, which might damage the polished capacitor plates. Finally, gently rinse the cleaned parts in calcium- and salt-free water and let the parts dry off completely.

09

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}:

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

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

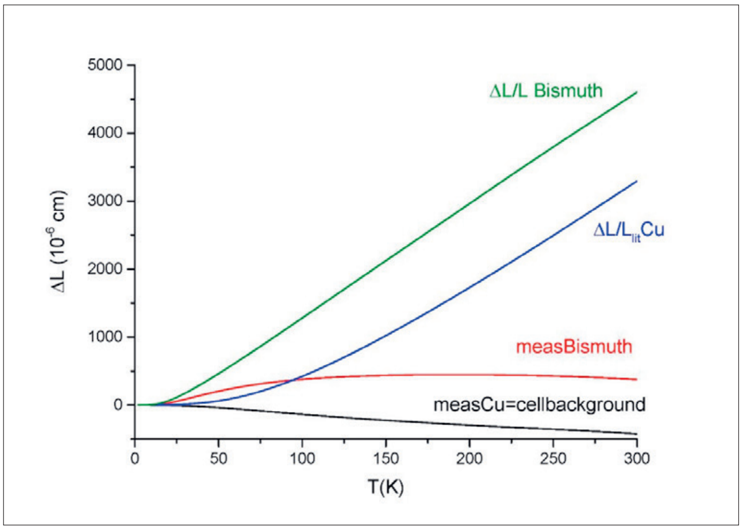
The relative length change of the sample normalized to its room temperature length L_0 is therefore given by:

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

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 3 mm large bismuth single crystal measurement. Note that the copper sample must have the same length as the investigated sample:

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

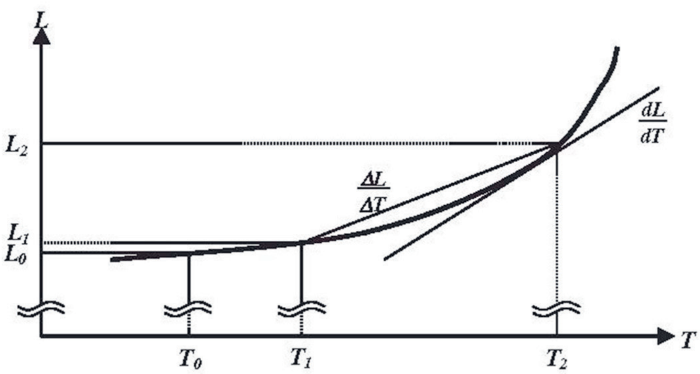


The cell background of the dilatometer is linear with the measured copper sample length. Therefore, only a small (1 mm) and a large (4 mm) copper sample have to be measured for the background subtraction. Then, all other sample length between them can be calculated. This is explained in detail in the appendix.

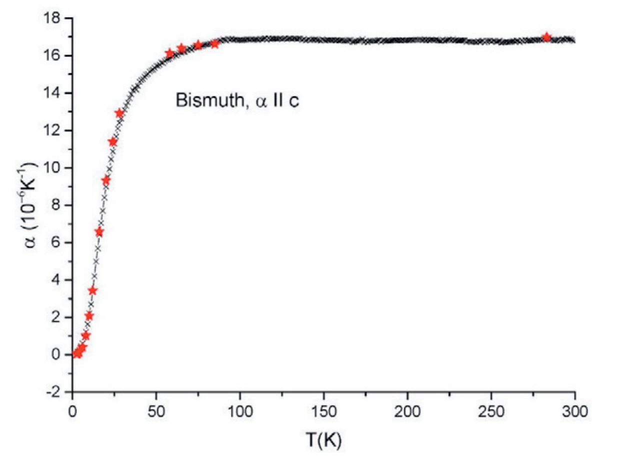
From the resulting $\frac{\Delta L^{Bismuth}}{L_0}$-data the thermal expansion coefficient

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

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



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



Red points show literature data

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