High resolution capacitance dilatometer for measuring thermal expansion and magnetostriction for use inside a PPMS- or cryogenic-system

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THE DILATOMETER

The great advantage of the new type of measuring cells is based on the unique combination of a patented driving technology, the powerful design and the high level of manufacturing quality.

KEY FEATURES AND ADVANTAGES:

+ Small dimensions (diameter 26 mm, width 20 mm, height 23 mm)

For the first time, a high-resolution dilatometer of such type is small enough to be used with the commercial PPMS (Physical Property Measurement System) of Quantum Design and can be mounted on a attocube rotator and fits inside the inner vacuum chamber of a dilution refrigerator (40 mm) and allows a rotation of 360 degrees.

+ Extremely high resolution $\Delta L/L$ in the region of 10^{-9} to 10^{-10}

The most sensitive method for detecting length changes of solids is the used capacitive dilatometry. The resolution of such a dilatometer is determined by the diameter of the two capacitor plates and by the parallelism between them: The cells produced by the new technology are only marginally wider than the size of the capacitor plates and therefore allows the use of maximal sized capacitor plates. To guarantee best parallelism between the plates we developed a patent-pended grinding device to polish the capacitor plates within its frames. Both these innovations produce an unprecedented resolution in a capacitive dilatometer of this compact size.

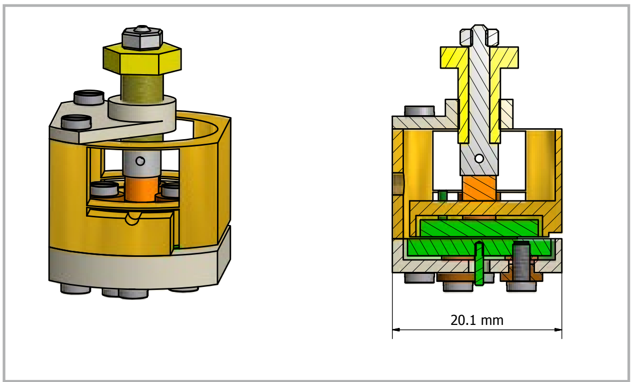

Fig. 1

+ Small weight (42 g) and High temperature stability

Due to the small cell mass and the low numbers of contact surfaces, one achieves a good thermal homogeneity and an excellent temperature stabilization of the cell. This provides a fast thermal relaxation time and a high sensitivity of the cell on temperature changes and therefore makes the cells ideally suited even for high temperature measurements. Therefore the dilatometers allow to reliably measure thermal expansion and magnetostriction from 300 K down to ultra-low temperature (less than 10 mK)

+ Suitable for very strong magnetic fields (current max. tested field: 30 T)

The dilatometers are made of copper beryllium. Due to the low Be content its thermal expansion and its thermal conductivity are nearly that of pure Cu, whereas its electrical conductivity is reduced by a factor of 30. As a consequence, the influence of eddy currents induced by time variation of the magnetic field is much smaller than in cells made from pure copper or silver.

+ Simple mounting of the sample

Due to the very small cell mass and the small numbers of contact surfaces, the thermal equilibrium in the cell and sample material can be reached very quickly upon cooling and heating. Therefore, a usually used complicated setup, where the sample is thermally isolated from the cell, is not necessary. Samples of less than 1 mm to 7 mm length can be measured.

+ Stable and suitable for long term application

The two capacitor plates are separated by electrically insulating pieces of vespel and 0.5 mm sapphire washers and are surrounded by guard rings to avoid stray electric fields. For both the lower and the upper capacitor plate, three BeCu screws are used to fix the plates to the guard rings. In contrast to gluing the plates, this makes the construction much more stable and suitable for long term applications.

NEW TYPE OF DILATOMETER

New innovative production method (milling and electrical discharge machining): Unprecedented resolution in a dilatometer of this compact size

DESIGN AND OPERATION

There's a comprehensive description of cell background calibrations and test measurements in R. Küchler et al., Rev. Sci. Instr. 83, 095102 (2012)

The construction of our dilatometer is based on the design of Pott and Schefzyk (J. Phys. E: Sci. Instrum. 16, 4456 (1983)). All parts (except some insulating spacers) are machined out of high-purity beryllium-copper. While the lower capacitor plate (6) is mounted to the fixed outer cell frame (3), the upper capacitor plate (5) is fixed to the movable part (1), which is held in the frame by two BeCu leaf springs (2). In this parallelogram suspension the upper plate can only move vertically. The sample is fixed by means of an adjustment screw (9) between the outer frame (3) and the movable part (1). In this construction, a length change of the sample (4) causes an equivalent displacement of the upper plate with respect to the lower, and therefore, a change in capacitance. By using the simple formula for a plate capacitor the length change can then be calculated. The adjustment screw (9) contains a piston (10), which is hung up on the head of the screw. This prevents the warping of the sample during positioning. A 30 degree window between the movable part (1) and the fixed outer frame (3) allows for an easy mounting of the sample (4), but is small enough to ensure a good mechanical stability of the cell. Samples of less than 1 mm to 7 mm length can be measured. The two capacitor plates are separated by electrically insulating 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 plate (5), three BeCu screws are used to fix the plates to the guard rings (7,8).

In contrast to gluing the plates, this makes the construction much more stable and suitable for long term applications. Before mounting the dilatometer, the capacitor plates were polished within their frames. A uniform surface of the plates within their frames is necessary to achieve best parallel orientation of the plates. In its rest position the capacitance of the dilatometer is about 6 – 8 pF, corresponding to a distance of 0.25 – 0.20 mm between the capacitor plates. After mounting the sample, the adjustment screw is used to reduce this distance to 0.067 mm, which corresponds to a capacitance of about 20 pF. By careful construction of the capacitance measuring circuit (shielding, avoiding of ground loops etc.) the absolute value of the capacitance is measured with a commercially available capacitance bridge with a resolution of 10^{-6} pF, which corresponds to a relative sensitivity of $\Delta L / L$ $10^{-10} – 10^{-9}$.

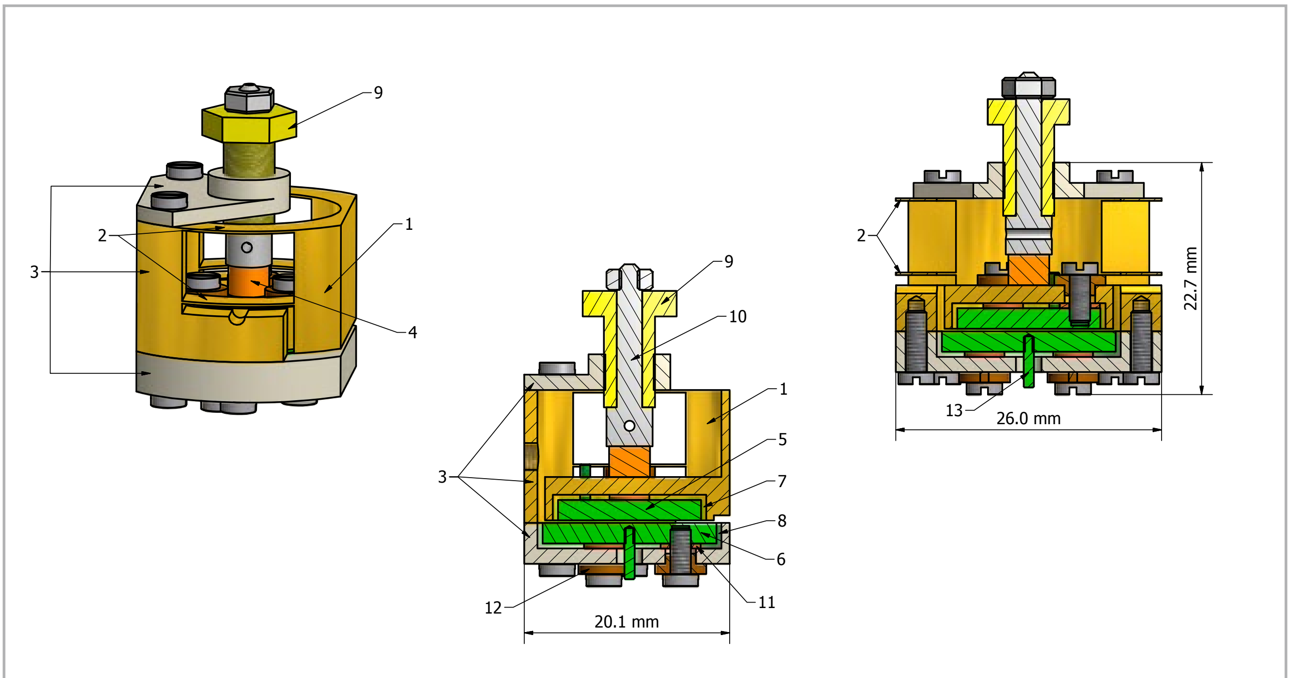

Fig. 2

INNOVATION

Makes it possible to extremely minimize the cell dimension, but the full width of the capacitor plates and therefore the resolution are completely preserved. Unprecedented resolution in a dilatometer of this compact size: By measuring at low-T the last digit of the capacitance measuring bridge is nearly stable. (See Fig. 3)

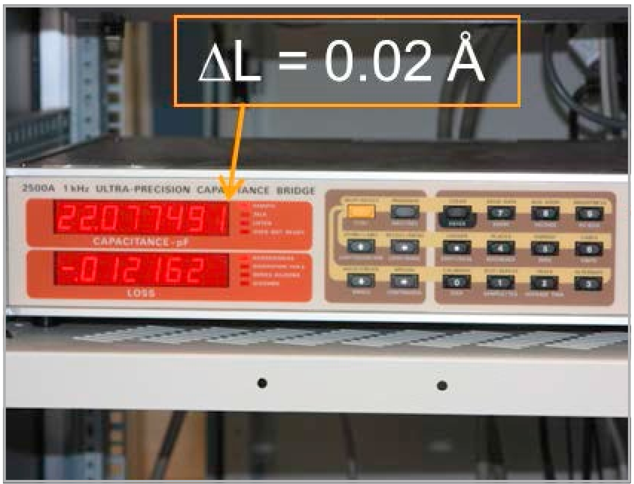

Fig. 3

The original prototype design of Ref. (J. Phys. E: Sci. Instrum. 16, 4456 (1983)) was assembled from ten main parts which determine the dimension of the cell. Our innovation is to produce the corpus of the cell, which originally consisted out of six different parts, from a single piece of BeCu, using milling and electrical discharge machining. The new main body (golden part of Fig. 2) now contains the whole movable part (1), both springs (2), as well as the middle part of the outer fixed cell frame (3). To include the springs in the main body, the BeCu block used for machining had to be annealed prior to processing for three hours at 600 K. This increases the hardness of the alloy by a factor of three and therefore enables the physical stability and elasticity of the springs, which are crucial for precisely plano-parallel vertical movement of the movable part with respect to the fixed outer cell frame. As shown in Fig. 2 the new manufacturing method makes it possible to extremely minimize the cell dimensions, which now allows the installation of the cell in extremely size limited sample chambers. Since our goal was to install the cell within an multi-functional insert of a commercial PPMS system and to rotate 360 degrees within a inner vacuum chamber of 40 mm, it was particularly important to reduce the width of the dilatometer. Using the new production method we achieved a width of only 20 mm with a cell diameter of 26 mm and an overall height of 22 mm. The new dilatometer has often great advantages compared to previous models. Although the whole mass of the cell could be reduced from more than 100 g to below 40 g, the full width of the capacitor plates and the resolution is completely preserved. Besides, due to the smaller cell mass and the significantly reduced numbers of contact surfaces, the thermal equilibrium in the cell material can be reached much faster upon cooling and heating. This makes the dilatometer suitable for measurements of thermal expansion at high temperature. Additionally, it is particularly suitable for magnetostrition measurements.

MADE OF CUBE: SUITABLE FOR HIGH MAGNETIC FIELDS!

The dilatometers are suitable for thermal expansion measurements in a broad temperature range from 300 K down to less than 10 mK, as well as for magnetostriction experiments in fields up to at least 30 T.

We have chosen a material with high thermal conductivity, insensitivity to high magnetic fields, and well-known thermal expansion characteristics in the whole temperature range. The possible high-field applications require a minimization of eddy currents in the metallic cell material. Usually eddy currents are induced by time variation of the magnetic field ($dB/dt \neq 0$) and cause magnetic moments. The induced moments would interact with the applied field and could produce a torque on the movable part of the cell, resulting in an unintentional displacement of the capacitor plate. This effect depends on the rate dB/dt and gives rise to an irrepressible noise level. One main requirement concerning the cell material is to minimize this effect. In addition, to avoid mechanical stress during temperature sweeps, the cell material needs to be very homogeneous. Furthermore, thermal equilibrium in the cell material has to be reached fast. The alloy copper beryllium with a beryllium concentration of 1.84 % meets all criteria most appropriate. Due to the low Be content its thermal expansion and its thermal conductivity are nearly that of pure Cu, whereas its electrical conductivity is reduced by a factor of 30. Therefore, the influence of eddy currents induced by dB/dt is much smaller than in cells made from pure copper or silver.

HISTORY

Why development of such type of dilatometer

Over the last 10 years, the volume thermal expansion coeffcient has been established as one of the preferred quantities to investigate quantum critical phenomena. That is just one of the many reasons why high resolution thermal expansion measurements become more and more important. While measurements of the specific heat are standard experiments in most physics laboratories, thermal expansion studies are still less common due to technical difficulties. The rising interest in studying thermal expansion, not only at low temperatures, calls for an easy-to-use dilatometer with sub-angstrom resolution. The most sensitive method for detecting length changes of solids is capacitive dilatometry, which was first described by White in 1961. The only major disadvantage of their design is the big size of the dilatometer, which is necessary for a high resolution. We are now able to produce an extremely compact and miniaturized dilatometer constructed from a Be-Cu alloy using electrical discharge machining. Our innovative production method allows us to minimize the overall frame size of the cell without reducing the sample space or the diameter of the capacitor plates by the same amount, which preserves an extremely high sensitivity. Furthermore, the space saving design of the cell body leaves enough room for an integrated thermometer, so that the sample temperature can be monitored very precisely. This ensures an easy calibration and a very good reproducibility of the results obtained with our measurement setup. The most important novelty of our work was to reduce the cell dimensions enough to integrate the dilatometer into commercial measurement systems like the PPMS by Quantum Design. Due to the very small probe space available and thermal stability issues, no thermal expansion and magnetostriction insert with a comparable sensitivity has yet been developed for these common laboratory setups. Besides, our dilatometer can be used universally for thermal expansion and for magnetostriction experiments. The magnetostriction $\Delta L(B)$, i.e. the length change of a sample induced by an applied magnetic field, is a thermodynamic quantity that provides important information about field induced phase transitions of matter at constant temperature and pressure. Our miniaturized high resolution dilatometer can even be mounted on a rotator and still fits inside the inner vacuum chamber of a dilution refrigerator. The one-axis rotation set-up allows a rotation of 360 degrees. This makes it now possible to study the angle dependence of quantum oscillations or the anisotropy of the order parameter of a superconductor.

WORKING ENVIRONMENT

Our dilatometers can be used in a wide temperature range. A low temperature limit is not known, the dilatometers have been tested down to ultra-low temperature (less than 10 mK). The upper temperature limit is determined by the thermal capability of the insulating pieces of vespel and the used coaxial cables. Up to know, we tested the dilatometers only slightly above room temperature.

The measurement quality depends on the sample material, the electronic and cryogenic peripheral equipment (shielding, avoiding of ground loops, mechanical vibrations, ...).

To achieve best possible results the dilatometer has to be operated in a slowly flowing inert gas atmosphere, where the dielectric constant of the medium does not change with temperature (e.g. Helium, Nitrogen, clean and dry air, vacuum). Our dilatometers have been tested in vacuum, in PPMS-systems in Helium-atmosphere with pressure below 1 mBar and in Helium exchange gas cryostats. The operation in flowing gas inserts or direct liquid gases (Helium) is not recommended.

Additionally, it has to be mentioned that vibrations caused by pumps connected to your cryogenic equipment can induce an irrepressible noise level to your capacitance measurement.

Of particular importance is also the correct cabling of the two capacitor plates. It is essential to shield the thin coaxial cables from the measuring bridge down to the plates. Feed troughs and connectors have to be shielded, too. A good way is to use feed troughs made of plastic.

APPLICATIONS

The dilatometers can be used with several cryogenic refrigerator systems down to ultra-low temperature (less than 10 mK) and in magnetic fields to at least 30 T. We have successfully tested and operated dilatometers of this new type with the commonly used physical property measurement system (PPMS) by Quantum Design, as well as with two other cryogenic refrigerator systems (Dilution refrigerator and exchange gas cryostat). For all these systems we additionally offer the matching accessories for suitable mounting.

PPMS (PHYSICAL PROPERTY MEASUREMENT SYSTEM)

The dilatometer can be used within the multifunctional chamber of a commercial PPMS (Physical Property Measurement System) of Quantum Design (Thermal expansion and magnetostriction between T = 2 K and T = 300 K)

Two options:

Option one: (a) Dilatometer mounted within your own multifunctional chamber (QD-P450A)

Fig. 4

The multi-functional PPMS insert is thermally coupled to the annular region at the bottom, where heaters warm the helium gas to the correct temperature, via a pin connector. We offer gold plated thermal anchors fixed on a copper block (1). The block can be mounted just above the cell so that the thermal anchors touch the inner chamber of the PPMS cooling channel. This further improves the thermal coupling of the cell. Additional anchors (2) can be mounted above the insert at several points of the measurement probe to reduce the temperature successively from the top of the cryostat down to the cell. To reduce the heat leak caused by the coaxial cables, they have to be wrapped around the measurement probe and the anchors. The temperature can be measured by a commercial Cernox resistance thermometer (4), which has to be enclosed in the dilatometer holder (3), near the sample.

PPMS-attachment: 1 × gold plated thermal anchors fixed on a copper block (1), 3 × additional anchors (2), 2 × dilatometer holder (3)

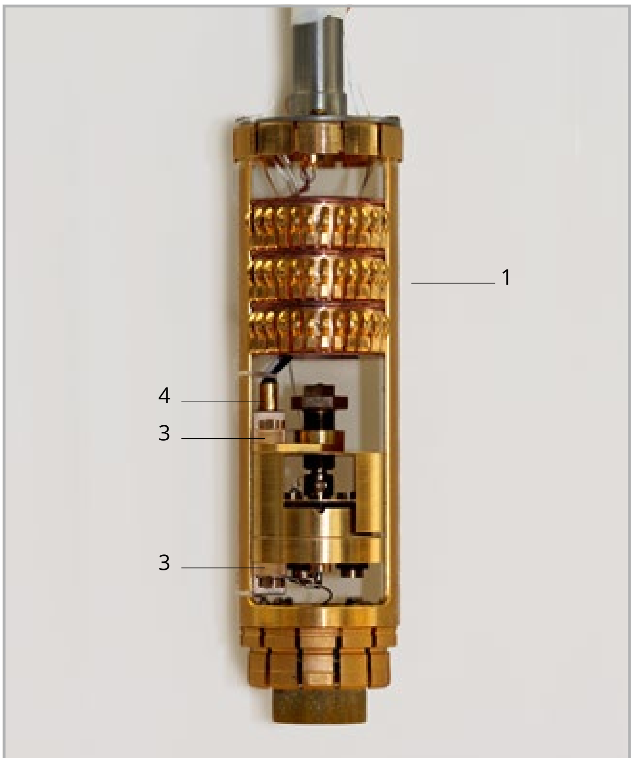

Fig. 5

What else is needed to perform PPMS dilatometry measurements within your own multi functional insert

(a) Capacitance bridge. We suggest to use the Andeen Hagerling 2550 A (1 kHz) or its predecessor model AH 2500 A (1 kH), or at least a Ultra-Precision Capacitance Bridge. By measuring at low-T we reach the limit of the performance, that means the last digit of the capacitance measuring bridge is already stable at constant temperature.

(b) Commercial (Cernox) resistance thermometer to monitor the temperature. The resistance thermometer can be enclosed in the dilatometer holder (3), near the sample. Twisted Pair Beryllium Cooper Loom (CMRdirect) for cabling.

(c) A pair of ultra thin coaxial cables and suitable connectors (We use Fischer connectors (DEE 102 A021-600 and DBEE 105 A093-80)). To reduce the heat leak caused by the coaxial cables, they have to be wrapped around the thermal anchors and the measurement probe.

(d) We offer **software for FREE**. The readout of the dilatometry data can be done with a Labview program and the temperature and field sweeps can be controlled comfortably with a Multi View program. You **don't** need a additional resistance bridge, you just have to connect the Fischer connector (DBEE 105 A093-80) with your PPMS-system. By means of our software you then can use its integrated resistance bridge

Easy Calibration. You find a detailed description in Rev. Sci. Instrum. 83, 095102 (2012). **Note:** The cell effect depends on the measured sample length. Since the cell effect will linearly increase with the sample length one has to do the background calibration for at least two samples (for instance 1 mm and 4 mm). Then one can easily calibrate the cell background for any sample length taking into account the linear dependency.

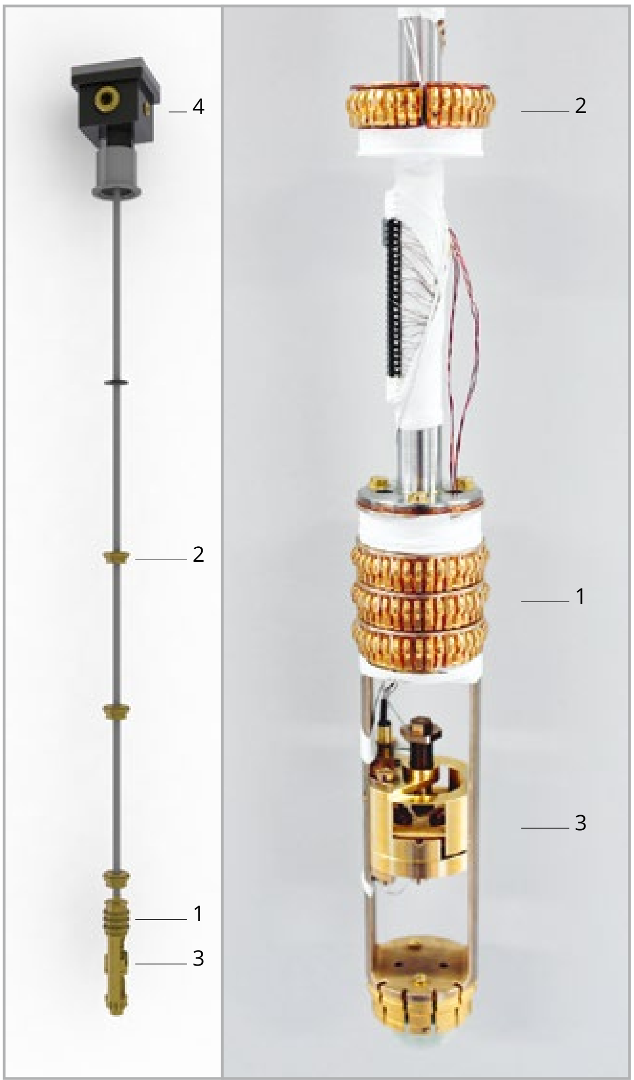

Fig. 6

Option two: (b) Dilatometer already mounted and cabled within a special dilatometry PPMS insert. This type of insert is designed and developed for best possible dilatometry measurements and can be used in PPMS systems.

The dilatometry PPMS insert is designed and developed for best possible dilatometry measurements and can be used in PPMS systems. The insert is thermally coupled to the annular region at the bottom, where heaters warm the helium gas to the correct temperature, via a pin connector. The large chamber, which contains the dilatometer (3), is signicantly larger than the chamber of the multi-functional probe. This makes the sample mounting especially easy and most convenient. The insert contains very effective gold plated thermal anchors fixed on a copper block (1), with a extra large-surface work platform. The block is mounted just above the cell so that the thermal anchors 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). For this reason the extra large-surface work platform of the thermal anchors are mounted at this level to effectively improve the thermal coupling of the cell. Additional anchors (2) are mounted above the insert at several points of the measurement probe to reduce the temperature successively from the top of the cryostat down to the cell. To reduce the heat leak caused by the ultra thin coaxial cables and the 12 twisted Pair Beryllium Cooper Loom, they were wrapped around the measurement probe and the anchors. The temperature can be measured by a Cernox resistance thermometer, which is enclosed in the dilatometer holder, near the sample. The head of the probe (4) contains three hermetic sealed Fischer cable connectors (2 × DEE 102 A021-600 for coax cable and 1 × 12 pin DBEE 105 A093-80 for thermometer and other use) The dilatometry PPMS insert package includes also **software for FREE**. The readout of the dilatometry data can be done with a Labview program and the temperature and field sweeps can be controlled comfortably with a Multi View program. You **don't** need a additional resistance bridge, you just have to connect the Fischer connector (DBEE 105 A093-80) with your PPMS-system. By means of our software you then can use its integrated resistance bridge.

What else is needed to perform PPMS dilatometry measurements within our special PPMS insert.

Just a capacitance bridge. We suggest to use the Andeen Hagerling 2550 A (1 kHz) or its predecessor model AH 2500 A (1 kH), or at least a Ultra-Precision Capacitance Bridge. By measuring at low-T we reach the limit of the performance, that means the last digit of the capacitance measuring bridge is already stable at constant temperature.

Easy Calibration. You find a detailed description in Rev. Sci. Instrum. 83, 095102 (2012). **Note:** The cell effect depends on the measured sample length. Since the cell effect will linearly increase with the sample length one has to do the background calibration for at least two samples (for instance 1 mm and 4 mm). Then one can easily calibrate the cell background for any sample length taking into account the linear dependency.

The dilatometers can also be used with several cryogenic refrigerator systems down to ultra-low temperature (less than 10 mK) and in magnetic fields to at least 30 T, with a very high resolution of 0.02 Å. We have successfully tested and operated dilatometers of this new type with two cryogenic refrigerator systems (Dilution refrigerator and exchange gas cryostat). For all these systems we also offer the matching accessories for suitable mounting.

DILUTION REFRIGERATOR

The dilatometer can be used within a Kelvinox dilution refrigerator between T ≤ 7 mK and T = 10 K

Two options:

Option one: (a) Low eddy current dilatometer holder

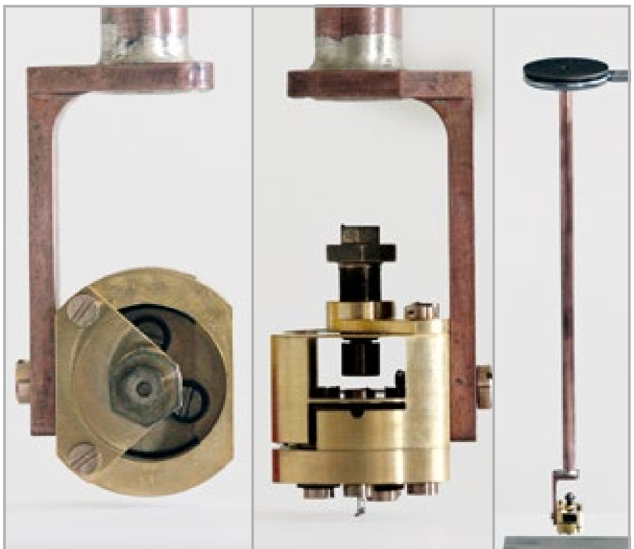

Fig. 7

The low eddy current dilatometer holder is a longitudinally slotted copper tube (thinwalled open section) extension from the mixing chamber to the field center of the magnet. It is specially designed to minimize eddy currents when changing the magnetic field of the magnet. The dilatometer holder allows aligning the dilatometer parallel and perpendicular to the magnetic field. The Dilatometer holder can be manufactured according to the cryoequipment of the purchaser. The price is depending on the complexity of your cryoequipment. The exact price can be offered at the time when you specify the attachments of your cryostat.

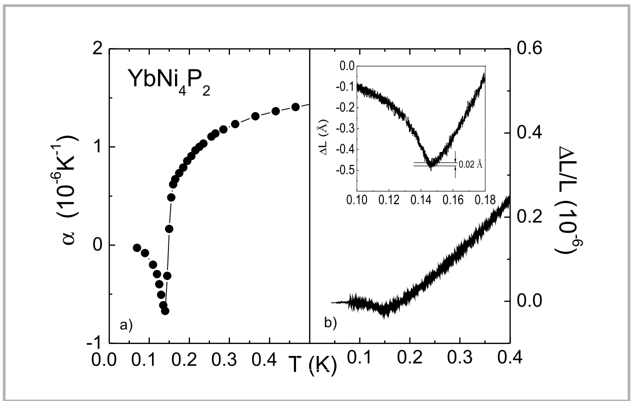

Fig. 8

To give an impression of the sensitivity of our dilatometer, in the picture is shown the relative length change and the low-temperature thermal expansion coefficient of an $YbNi_4P_2$ single crystal with a length of L = 1 mm, which is a ferromagnet below T_C = 0.15 K. For this measurement the cell was mounted on a cold finger of a dilution refrigerator, using the eddy current dilatometer holder. A sharp λ-type anomaly observed in the thermal expansion coeffcient α (T) at T_C indicates the second-order phase transition. The width of this anomaly is only about 20 mK, which is also observed in the specific heat. It demonstrates that, due to the tiny cell size, thermal equilibrium for both the cell and the sample is reached very quickly and a complicated setup, where the sample is thermally isolated from the cell, is not necessary. The inset shows the extraordinary sensitivity of our dilatometer with a very high resolution of 0.02 Å. For more details see: A. Steppke, R. Küchler, et al., Science, 339 (6122) 933-936 (2013).

Option two: (b) Low eddy current dilatometer holder for attocube rotator ANRv101

The dilatometer has also been designed for the use in magnetic field as high as at least 30 T. Due to its small size it can be mounted on a rotator and still fits inside the inner vacuum chamber (38 mm diameter) of a Kelvinox dilution refrigerator. The dilatometer can be rotated around a horizontal rotation axis using a piezoelectric rotator provided by Attocube Systems.

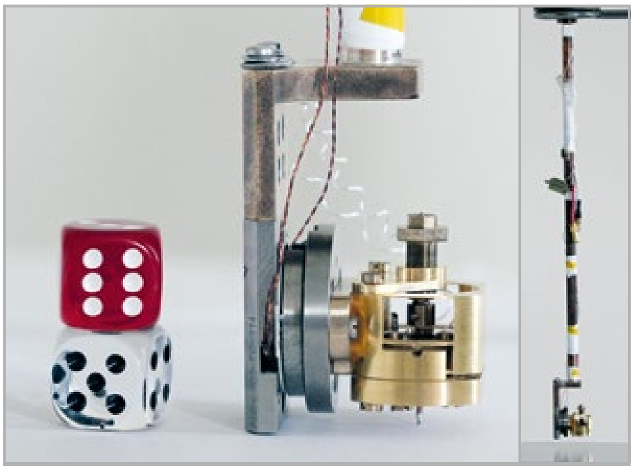

Fig. 9

The one-axis rotation set-up allows a rotation of 360 degrees. The attocube rotator is made of non-magnetic materials, which allows also the operation in very strong magnetic fields (current max. tested field: 31 T). The rotator angle can be measured by a rotary potentiometer with an accuracy of about 0.05 degrees. The low eddy current dilatometer holder is a longitudinally slotted copper tube extension from the mixing chamber to the rotator base plate. It is specially designed to minimize eddy currents when changing the magnetic field of the magnet. A additional offered adapter plate can be used for mounting the dilatometer on the attocube rotator ANRv101 base plate. The Dilatometer holder can be manufactured according to the cryoequipment of the purchaser. The price is depending on the complexity of your cryoequipment. The exact price can be offered at the time when you specify the attachments of your cryostat.

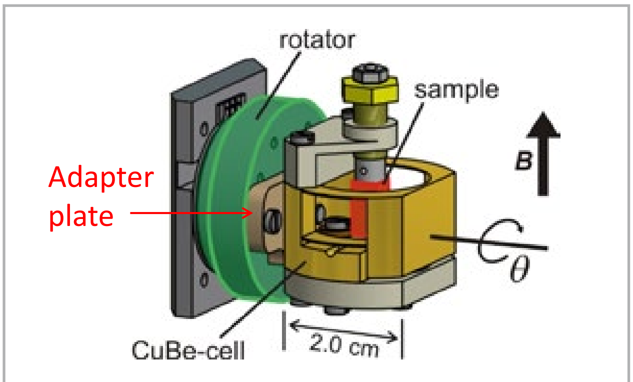

Fig. 10

To give an example of the exceptional sensitivity of our miniature dilatometer we show measurements of the quantum oscillations in the magnetostriction of bismuth. The figure shows the magnetostriction measured at 0.025 and 4.2 K along the high-symmetry crystalline axis for fields up to 10 T. At low temperature, the oscillatory phenomena, driven by the de Haas-van Alphen effect becomes clearly visible at magnetic fields as small as 0.2 T. In the field range ≤ 2 T more than twenty full periods were identified (see Inset of Fig. 11). The de Haas-van Alphen period was found to be 0.15 T^{-1}, in good agreement with the results obtained from other methods. This period corresponds to the maximum cross section of the hole ellipsoid of the Fermi surface. Due to the great sensitivity of the new dilatometer, displacements as small as 0.02 Å have been resolved.

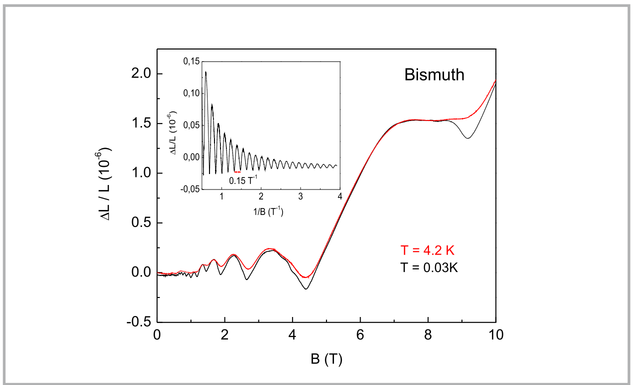

Fig. 11

To measure the angle-dependent Landau Level spectrum for electrons and holes, we measured the magnetostriction at various tilt angles Θ of the field in the trigonal-binary plane. The gure shows the relative length change between 0 and 18 T, for -10° ≤ Θ ≤ +10°. Oscillatory features of the Landau Level crossing are already visible in the raw ΔL/L data, as indicated by the red (holes) and blue (electrons) arrows. Most notably, we also observe anomalies at high fields far beyond the hole quantum limit of ~ 9 T, highlighted by the large blue arrows. The inset shows a surface plot of the magnetostriction coeffcient -λ derived from these measurements, with clearly discernible resonances. This is the first experiment where even the smaller peaks associated with electron LLs (blue) in bismuth can be resolved in a magnetostriction measurement. For more details a publication will follow soon.

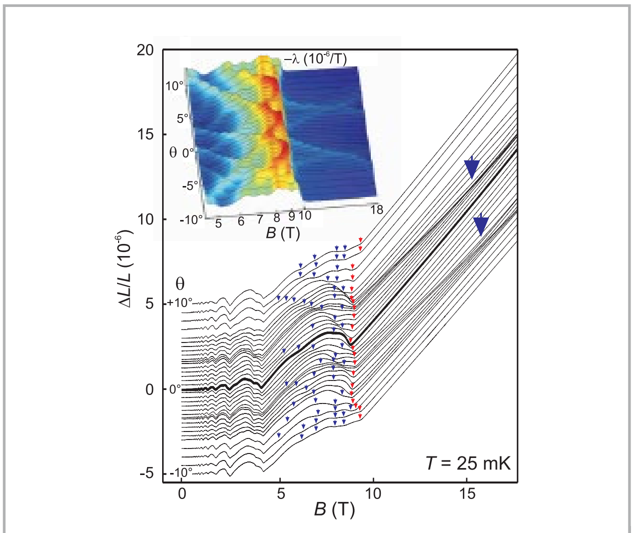

Fig. 12

What else is needed to perform low-T dilatometry measurements

(a) Capacitance bridge. We suggest to use the Andeen Hagerling 2550 A (1 kHz) or its predecessor model AH 2500 A (1 kH), or at least a Ultra-Precision Capacitance Bridge. By measuring at low-T we reach the limit of the performance, that means the last digit of the capacitance measuring bridge is already stable at constant temperature.

(b) Commercial (RuO_2) resistance thermometer to monitor the temperature. The resistance thermometer can be mounted on the dilatometer holder, near the sample. Resistance Bridge. We made good experience with the Lake Shore 370 AC

(c) A pair of ultra thin coaxial cables and suitable connectors. The two capacitor plates have to be connected by coaxial cables to the measuring bridge. To avoid ground loops, the cell has to be insulated from the rest of the insert by coaxial cable feed-throughs made of plastic.

(d) We offer **software for FREE**. The readout of the dilatometry data can be done with the offered Labview program.

Easy Calibration. At low temperature $T \leq 5$ K the cell background is extremely small due to the very small phonon contribution.