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New experimental perspectives for soft x-ray absorption spectroscopies at ultra-low temperatures below 50 mK and in high magnetic fields up to 7 T

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A new ultra-low temperature experiment including a superconducting vector magnet has been developed for soft x-ray absorption spectroscopy experiments at third generation synchrotron light sources. The sample is cooled below 50 mK by a cryogen free ³He-⁴He dilution refrigerator. At the same time, magnetic fields of up to ± 7 T in the horizontal direction and ± 0.5 T in the vertical direction can be applied by a superconducting vector magnet. The setup allows to study *ex situ* and *in situ* prepared samples, offered by an attached UHV preparation chamber with load lock. The transfer of the prepared samples between the preparation section and the dilution refrigerator is carried out under cryogenic temperatures. First commissioning studies have been carried out at the Variable Polarization XUV Beamline P04 at PETRA III and the influence of the incident photon beam to the sample temperature has been studied. Published by AIP Publishing. [<http://dx.doi.org/10.1063/1.4947516>]

I. INTRODUCTION

In the last decades, single-molecule magnets (SMMs) have attracted much attention due to their high potential in the rising field of molecular spintronics.^{1–3} For instance, prominent examples are the high-spin molecules Mn₁₂ and Fe₈.^{4,5} These type of magnets are of great interest for both future applications and fundamental research since they show classical as well as quantum behavior.⁶ Below a blocking temperature T_B , SMMs show remanent magnetization with thermal relaxation times defined by the temperature and the height of the magnetic energy barrier which separates the two magnetic states.¹ Because T_B can vary from several K down to some tens of mK,³ sufficient cooling techniques are required to slow down the relaxation and study possible new SMMs candidates to finally increase T_B . Up to now, most experimental studies have been performed in the bulk phase since the deposition of SMMs on surfaces is rather difficult and the molecules easily change their characteristic properties due to the hybridization of the electronic structure with the surface.^{7,8} Nevertheless, the deposition is highly recommended from a technical point of view.² Thus, changes of the electronic structure of the deposited molecules together with the simultaneously changed magnetic properties have to be studied systematically.

The x-ray absorption spectroscopy (XAS) and x-ray magnetic circular dichroism (XMCD) spectroscopy techniques are well established to investigate the electronic and magnetic properties of such dilute nanoscale materials in an element-

selective way.^{9,10} Besides the total magnetic moment, the XMCD technique allows to determine separately the local spin and orbital magnetic moments by magneto-optical sum rules.^{11,12} However, since these surface sensitive spectroscopy techniques average over an ensemble of nanoparticles, it is necessary to align the ensemble either by exchange interaction with a magnetic surface or by an external magnetic field parallel to the incoming photon beam. The field strength necessary to align the magnetic moments of the ensemble depends on the total magnetic moment, the magnetic anisotropy, as well as the temperature. While the magnetic moment and the anisotropy are intrinsic quantities of the system, the magnetic field together with the temperature is varied within the experiment and allows to study both quantities in detail.

In this article, we present a new experimental setup which offers temperatures down to 50 mK and magnetic fields up to 7 T while simultaneously performing x-ray absorption measurements (XAS, XMCD). Thus, the setup enables the study of the above described SMMs together with fundamental physics phenomena like the Kondo effect, superconductivity, or other correlation effects where low temperatures in the mK regime are essential.

This paper is structured as follows. In Section II, the design of the experimental station is described which is separated into the dilution refrigerator with vector magnet, the sample transfer, and the preparation section. In Section III, the cooling performance of the dilution refrigerator as well as the influence of a magnetic field sweep on the sample temperature is demonstrated. First commissioning data from the beamline P04 at PETRA III reveal how the photon heat load affects the sample temperature.

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FIG. 1. CAD drawing of the assembled experiment with a total height of about 3.5 m: (1) Dilution refrigerator section with coldfinger and sample holder. (2) Vector magnet (± 7 T horizontal, ± 0.5 T vertical) with UHV cross. (3) Slewing transfer system. (4) Preparation chamber. (5) Two-piece transportation and adjustment frame. The coordinate system is related to the vector magnet axis. Reprinted from Ref. 24.

II. SYSTEM DESIGN

The main challenge in the design of the experiment lies in the combination of achieving sample temperatures in the mK regime as well as magnetic fields of several Tesla while simultaneously performing soft x-ray absorption measurements at a synchrotron beamline. In order to fulfill such conditions, the experiment is divided into five segments as presented in Fig. 1 and discussed in Secs. II A–II E.

A. Dilution refrigerator

The main part of the dilution refrigerator is given by the TritonUHV, a cryogen free ^3He - ^4He dilution refrigerator^{13,14} for UHV applications, designed and constructed by Oxford Instruments (OI).^{15–19} The Triton is subdivided into five different temperature levels which are thermally decoupled from each other as can be seen in Fig. 2. The main work against the outer room temperature thermal radiation is performed by the first stage of a pulsed tube refrigerator with remote motor (PTR, Cryomech PT-410 RM²⁰). It is connected to the first level plate (15) and cools the first level down to 45 K where a cooling capacity of 31.5 W is given. Similar to the first level, the second level is cooled by the PTR second stage (14), achieving a temperature of 4.2 K at 0.9 W. Below the second level, the inner circuit of the ^3He - ^4He dilution refrigerator is located, including the temperature levels three, four,

FIG. 2. Lateral cut through the dilution refrigerator and vector magnet: (1) Outer case. (2) Vacuum and superinsulation. (3) LN jacket with shields. (4) LHe jacket with shields. (5) Vertical field superconducting split-solenoid. (6) Horizontal field superconducting split-solenoid. (7) Valve to transfer manipulator. (8) Transfer baffles. (9) Beamline port with pinhole. (10) Coldfinger. (11) Mixing chamber plate. (12) 100 mK plate. (13) Still plate. (14) PTR 2nd stage. (15) PTR 1st stage. (16) Goniometer. (17) ^3He turbo pump. Reprinted from Ref. 24.

and five. In particular, these are the still plate (13) with a temperature around 1 K, the 100 mK plate (12) and the mixing chamber (11). The latter achieves temperatures down to below 50 mK and is electrically isolated from the rest of the system by incorporating insulating supports and inserting ceramic breaks in the heat exchangers and the pre-cool bypass of the refrigerator.¹⁸ Thus, the sample current can be measured via the mixing chamber plate, providing the opportunity for total electron yield measurements. From the top, the TritonUHV with the strong PTR cools and screens the sample in the center of the vector magnet while a combination of several insulation and cryogenic wet shield layers is used sideways. Behind the room temperature outer case, an insulating vacuum and several layers of superinsulation (2) reduce the heat load onto a liquid nitrogen (LN) jacket (3), serving as a 77 K radiation shield. The thermal heat load to the inner parts is further reduced by another insulation volume with a following 4 K shield, realized by a liquid helium (LHe) jacket (4). The latter also includes the vector magnet volume (compare also to Fig. 1). Inside the LHe jacket is the UHV volume of the TritonUHV. The TritonUHV is protected from the side by a 45 K shield between the PTR first and second stage, a 4 K shield from the PTR second stage to the mixing chamber plate and a 1 K shield from the still plate down to the entrance of the magnet. The latter one shields

FIG. 3. In graph (a), the temperatures of the five temperature levels (Fig. 2 11-15) measured by RuO₂ resistance sensors are shown for a typical cool down of the dilution refrigerator. Graph (b) shows the cool down at the transition position between the coldfinger and the mixing chamber with the ³He-⁴He mixture being pumped. The RuO₂ coldfinger sensor is radiation protected and was calibrated in the mK-regime to a nuclear orientation thermometer by Oxford Instruments. Reprinted from Ref. 24.

approximately 80% of the coldfinger to the 4 K radiation of the magnet section.

If the LN and LHe jackets are filled in advance, the overall cool down time takes about 24 h and is shown in Fig. 3. The cool down is started by switching on the PTR. Since the PTR is only in thermal contact with the first two levels, a bypass connected by heat exchangers to each level is used for cooling the lower three levels down from room temperature.¹⁵ Therefore, some of the warm ³He-⁴He mixture is used to cycle through the bypass. Under PTR cooling, the mixture is compressed with decreasing temperature until the whole mixture circulates through the bypass. After 18 h, the bypass pre-cooling has cooled the lower four levels (11-14) down to 10 K. Subsequent to the pre-cooling, the bypass is evacuated so that the temperature of the lower levels rises to approximately 30 K. In a next step, the condensing of the ³He is started in the condenser line of the TritonUHV by the use of an external compressor. The ³He is cooled by the second stage of the PTR, several heat exchangers, and an impedance down to 1 K before it enters the mixing chamber. After the condensing process is complete (around 23 h in Fig. 3(b)), the ³He turbo pump (17) and the still heater are turned on and the ³He-⁴He cooling is started.^{13,21}

The coldfinger is in thermal contact with the mixing chamber plate (Fig. 2) and has a length of 335 mm. It is made out of gold plated high-purity copper (gp-OFHC). A slotted design in the vertical field (*z*) direction was chosen to reduce heating by eddy currents induced by a sweep of the magnetic field. The temperature gradient along the coldfinger is less than 1 mK²² and thus, smaller than the resolution of the temperature measurement. A socket (gp-OFHC) is screwed to the lower end of the coldfinger which loads the sample holder by a right handed female thread made out of polytetrafluoroethylene (PTFE). Thus, the sample holder can be transferred from and to the dilution refrigerator. With the sample holder on the coldfinger, the mounted sample can be aligned to the beamline with a goniometer (16) designed and constructed by the Technische Universität München (TMU). The goniometer moves the TritonUHV as a whole and allows to align and rotate the sample along the *z*-axis (Fig. 1).

B. Vector magnet

Additionally to the mK temperatures, OI designed and implemented a superconducting 2D vector magnet to the experiment. The magnetic fields are generated by a split pair solenoid in the horizontal *x*-direction and a split pair solenoid in the vertical *z*-direction situated outside of the horizontal pair. So the horizontal split pair is nested within the split of the vertical split pair. The vector magnet is a wet magnet and embedded into the LHe jacket (Fig. 2). In this manner, the required LHe radiation shield is utilized effectively to cool the superconducting solenoids below their critical temperature. In order to minimize the heat load on the LHe bath, the magnet can be operated in persistent mode. The split pair solenoids surround a stainless steel UHV cross with an open bore of 42 mm in the *x*-, *y*-, and *z*-direction. In the *z*-direction, the cross is in contact with the UHV volume of the TritonUHV and on the opposite side to the sample transfer system. Up to the LHe bath, the magnet radiation shielding is identical with that of the TritonUHV. The only difference is given by the mandatory opening to the beamline, realized by a 3 mm pinhole, and two baffles needed to access the coldfinger for the sample transfer (Fig. 4). Both baffles are part of the LN and LHe radiation shields and are hold in a closed state by torsion springs. If a sample transfer is carried out, the baffles are pushed open by the transfer cryostat.

The open bore design of the magnet represents an upper limit to the desired maximum field at the center of the magnet of ± 7 T in the horizontal (*h*) and ± 0.5 T in the vertical direction (*v*). At the center position, the field homogeneity is specified to 0.8% (*h*) and 1.45% (*v*) over 10 mm diameter of spherical volume (DSV). The operating current of the solenoids is 16.16 A/T (*h*) and 158.39 A/T (*v*), resulting in maximum currents of 113.12 A and 79.20 A for full field performances. The designed nominal inductance is 17.38 H (*h*) and 2.17 H (*v*), corresponding to stored solenoid energies of 111.20 kJ (*h*) and 6.81 kJ (*v*) at maximum field strengths. The magnet was not designed for fast ramping performance because of the strong influence on the sample temperature (see Fig. 7 and discussion). Moreover, the ramp rates are limited due to the

FIG. 4. CAD scheme of the sample transfer: (1) Coldfinger. (2) Socket with right handed female PTFE thread. (3) Sample seating area. (4) Sample holder back end with left handed female thread. (5) 4 K transfer baffle. (6) 77 K transfer baffle. (7) Sample holder front end with right handed male thread. (8) Transfer cryostat cold head (9) Transfer cryostat inner tube. (10) Transfer cryostat outer tube. (11) Ferrofluid rotary feedthrough with bellow.

present magnetic field of the other vector axis. The maximum ramp rates are stated to be 9.70 A/min (0.60 T/min) (h) and 31.68 A/min (0.20 T/min) (v) so that the magnetic field direction can be reversed within 23.3 and 5.0 min, respectively. A resistor cold diode provides quench protection of the magnet by dumping the stored energy in case of a loss of superconductivity. Additional protection is achieved by the magnet power supply which ramps down to zero amps and clamps the output if an increased voltage with falling current is detected.

C. Sample transfer

In order to investigate samples under *in situ* conditions and to load *ex situ* prepared samples, a UHV slewing transfer system was developed in close cooperation between Universität Hamburg (UHH) (transfer system) and OI (sample holder). With the transfer system, the samples can be transferred between the dilution refrigerator with magnet unit and the preparation chamber (compare Figs. 1, 2, and 4). Cryogenic temperatures are reached by a liquid helium flow cryostat which, for example, are important for samples with surface diffusion temperatures below room temperature, like deposited atoms and small molecules or clusters.

Fig. 4 shows a scheme of the sample transfer system. The sample holder (gp-OFHC) used to transfer the samples has a length of 210 mm and a diameter of 39 mm. A seating area of $22 \times 20 \text{ mm}^2$ ($w \times l$) is offered to fix the sample to the holder. On the coldfinger side of the sample holder, a right handed male thread (gp-OFHC) provides good thermal contact which is even enhanced by the designed conical contact areas on the socket and sample holder side. On the other side of the sample holder, a left handed female thread is used to transfer the holder in and out of the coldfinger.

The main component of the slewing transfer system is given by a LHe transfer cryostat. It transfers the sample holder by a left/right hand male/female thread combination. Therefore, the transfer cryostat cold head offers a left handed male thread to take up the sample holder. The sample is handed over from and to the transfer cryostat by a ferrofluid sealed vacuum rotary feedthrough. While the outer tube together with the cold head and the sample support of the transfer cryostat can be rotated with the attached handwheel by 360° , the inner cryostat tube stays fixed. In this way, the sample can be transferred literally by hand. A defined torque of 8 N m, applied by a torque spanner in the preparation chamber, is used to define

the force the user exerts on the transfer threads. In this way, the thread cannot be overwound and at the same time an adequate contact for a sufficient cooling is provided. High sensitivity for screwing the sample in and out of the particular threads is given by a short bellow, sitting right behind the rotary feedthrough. The bellow is supported by a surrounding of springs which compensate the compression of the bellow by the vacuum force. Hence, a linear translation about 20 mm along the cryostat axis can be performed easily by hand. Because the heat load to the transfer cryostat is transferred likewise to the dilution refrigerator upon a sample transfer, it is necessary to screen the cryostat as much as possible. Thus, an inner and an outer insulating vacuum screens the inner and the outer parts of the cryostat from the heat load of the room temperature outer case.

The transfer cryostat is mounted on a linear manipulator with a traveling distance of 500 mm. In particular, the manipulator consists of a slide which is ground into two rails and translated by a recirculating ball screw, driven by a stepping motor. An angle of 90° between the preparation manipulator and the coldfinger axis (see Fig. 1) was necessary to account for the given height of the beamline. Therefore, the cryostat manipulator needs to be rotated if a sample transfer takes place. Similar to the manipulator assembly, a slide on a curved rail is used to undergo the rotation. The movement is realized by a gear box which transmits the power of the used motor (Schneider Electric, model BRS3 3AD with 16 N m) with a gear ratio of 72:1 in order to overcome the vacuum force working against the rotation. Hence, it is possible to align the transfer cryostat on axis to the coldfinger as well as to the preparation chamber manipulator.

D. Preparation section

The preparation chamber was designed and constructed by UHH and is of cylindrical shape with an inner diameter of 270 mm and a length of 213 mm. The cylinder is mounted on a vertical plate with a CF DN-63 opening to the transfer section. The chamber is designed to offer the necessary equipment needed for *in situ* sample preparation and characterization under UHV conditions together with the desired sample transfer system. Therefore, a combination of a 4-axis manipulator with LHe flux cryostat (x, y, z, θ) mounted on a differential pumped CF DN-300 rotary flange (Ω) is used to access the whole chamber volume. By a rotation of the rotary flange, the

manipulator travels on a circular path so that the sample can be moved in front of each specific CF port. In order to prepare and characterize the sample surface, a sputter gun, an evaporator and a low energy electron diffraction (LEED) system are mounted. Unoccupied ports provide space for additional equipment. Other CF ports on the rotary flange are exclusively used for the sample transfer.

Via a load lock with magnetic transfer rod, the sample is brought into the preparation chamber. This is realized by handing the sample over to the transfer cryostat. There, the sample holder is tightened precisely with 8 N m, defined by a torque spanner used on the appropriate feedthrough. Afterwards, the sample holder is handed back either to the preparation cryostat or to a passive sample docking station. The latter can be used to position a second sample in the preparation chamber. Because the sample holder is always pointing in the same direction, the magnetic rod, the cold head of the cryostat and the passive docking station are similarly equipped with a right handed female PTFE thread, similar to the coldfinger in Fig. 4.

E. Transportation and beamline alignment

The whole experiment was designed by UHH as a mobile setup which is, due to its extreme height and weight of about 3.5 m and 3.2 ton, very ambitious. For transportation, the experiment can be split into two parts. In Fig. 1 (5), the upper and lower parts are indicated by the labeling. The splitting takes place between the lower side of the vector magnet and the sample transfer section. Both sides are equipped with UHV valves, which are connected by a bellow to preserve the UHV conditions in both sections. The small volume between the valves can be pumped separately. After the small volume has been vented, the upper part with the dilution refrigerator and the vector magnet can be moved up by three parallel driven linear actuators. The actuators are constructed in a similar fashion to the transfer system and offer a total traveling distance of 250 mm. The full traveling distance can be accessed if the upper part of the experiment is decoupled from the lower, otherwise the movement is limited to ± 10 mm due to protection of the connecting bellow. After the dilution refrigerator

and the vector magnet have been moved up, both parts are lifted onto support wagons for transportation to the synchrotron beamline.

After the experiment has been reassembled at the beamline site, it is necessary to align the center axis of the vector magnet with the photon beam. Thus, the height of the vector magnet (z -direction) can be adjusted within the described ± 10 mm. The accuracy of the z -component is 0.02 mm and can be swept continuously. In the xy -plane, the experiment can be adjusted by shifting a second plate with the experiment on top against the base plate. The second plate has a linear traveling distance of ± 30 mm in the x - and y -direction each with a precision in the range of 0.1 mm. The position for all three axis is readout by linear encoders. In addition to the linear translations, the azimuthal angle between the two plates can be varied up to $\pm 3^\circ$.

III. PERFORMANCE OF THE DILUTION REFRIGERATOR AND THE VECTOR MAGNET

A. Cooling performance

Systematic cooling power studies were performed in order to determine the heat load to the sample. Thus, a well-defined heat load was applied to the last cooling level by a heater placed close to the mixing chamber. Fig. 5(a) shows the temperature measured at the crossover point to the coldfinger in dependence of the applied still heater power. The lower three curves with 50, 70, and 100 μW represent studies performed during the commissioning phase of the experiment. It is easy to notice the two minima around 9 and 35 mW which seem to be a characteristic signature of the setup. Fig. 5(b) shows the appropriate temperatures of both minima for different heat loads collected in the commissioning phase of the experiment. Both curves diverge if the heat load is enhanced meaning that the temperature difference between the minima of Fig. 5(a) increases. This characteristic temperature difference only depends on the systems additional heat load (e.g., the mixing chamber heater) and is independent of the achieved base temperature. The latter simply shifts the cooling power curves in Fig. 5(b) to higher or lower temperatures,

FIG. 5. (a) Measured temperature versus the applied still power for certain mixing chamber heat loads and a Ti sample at the L_2 -absorption edge and an open exit slit. Note that the shift to higher temperatures for the Ti sample is due to a changed setup with respect to the commissioning phase of the experiment. (b) Mapped cooling power of both temperature minima at 9 and 35 mW proportional to T^2 . Reprinted from Ref. 24.

leaving the area in between unchanged. Hence, the mapped minima temperature differences can be used to determine the heat load to the sample by measuring the sample temperature in dependence of the still power (Fig. 5(a), full circles).

B. Commissioning at the beamline P04 at PETRA III

The influence of the photon beam on the sample temperature is crucial for future experiments with the setup. For example, the sample could undergo a possible phase transition by photon beam heating. Thus, commissioning studies were performed at the P04 soft x-ray beamline at the third-generation synchrotron light source PETRA III at DESY in Hamburg.²³ For the studies, a high purity Ti sheet (99.995) was mounted on the sample holder to perform x-ray absorption spectroscopy (XAS) measurements in the total electron yield mode. For all measurements, the storage ring PETRA III was running in the 480 bunch mode with a total electron current of 100 mA.

In order to estimate the maximum photon heat load to the Ti sample, the beamline monochromator was set to a fixed energy of 460 eV, according to the Ti $L_{2,3}$ -absorption edge. Additionally, the P04 exit slit was set fully open. The upper curve of Fig. 5(a) (full circles) shows the temperature depending on the applied still power for such conditions. From this dependency and the temperature difference of the curve minima, the maximum photon heat load to the Ti sample is estimated to 60 μ W according to Fig. 5(b). This corresponds to approximately 8×10^{11} photons/s and fits well to the beamline performance at that time.

In typical absorption experiments, the monochromator energy is scanned across the absorption edge which generally results in a change of the photon flux of the beamline. Therefore, several XAS long term scans were performed at the Ti $L_{2,3}$ -absorption edges. The exit slit was varied in each measurement in order to change the photon flux systematically. A typical long term scan running for 68 min is illustrated in Fig. 6 for an exit slit setting of 100 μ m. The long measurement time was chosen to suppress the uncertainties in the temperature measurement as far as possible. In the graph, the Ti $L_{2,3}$ -edge and the simultaneously recorded I_0 gold mesh XAS signals are both plotted versus the incident photon energy together with the corresponding sample temperature with respect to the measurement time. The I_0 gold mesh was placed right behind the last beamline valve in front of the experiment and serves as a measure of the photon flux. The measured base temperature lies around 46.5 ± 0.2 mK and stays constant over the first 16 min although the photon flux increases as can be seen from the gold mesh signal. Afterwards, the temperature increases linearly with the photon flux to a maximum of 48.0 ± 0.2 mK after 48 min of measurement time. With the decreasing transmission of the beamline at higher photon energies, the temperature falls down to 46.8 ± 0.2 mK at 68 min. The measurement shows that for a certain threshold, the applied heat load overcomes the cooling power of the dilution refrigerator so that the sample temperature increases. The maximum heat load of the presented measurement can be estimated by the open exit slit value if a linear dependence between the

FIG. 6. The upper graph shows the Ti $L_{2,3}$ -edge together with the gold mesh XAS signal which reflects the photon flux of the beamline. In the lower graph, the corresponding temperature of the Ti sample is presented over the measurement period. For this measurement, the exit slit was set to 100 μ m. Reprinted from Ref. 24.

exit slit width and the number of photons at the experiment is assumed. Thus, the approximated maximum heat load to the sample is around 10 μ W. This is also in accordance with the observed unchanged sample temperature at an exit slit width of 50 μ m or less.

C. Sweeping the horizontal magnetic field

Fig. 7 presents the influence on the mixing chamber temperature by the heat load induced by eddy currents under a magnetic field sweep. In particular, the temperature changes at the mixing chamber and the still during discrete horizontal field sweeps are shown. Between the sweeps, a typical XAS measurement was performed similar to Fig. 6. In Fig. 7(a), a sweep in the horizontal field direction between ± 1 T is presented. In this measurement, the exit slit was set to 100 μ m and the gold mesh was inside the beam path. The base temperature without field change during the measurements is around 48 mK. By selecting the maximum ramp rate, the field reversal took about 3.4 min. This time perfectly matches the rising slope of the mixing chamber temperature. In total, the temperature increases to about 94 mK before the dilution refrigerator overcomes the induced heat and cools the sample back to base temperature over a time period of 19 min. Hence, the overall sweep time is clearly dominated by the latter. Additionally to the influence on the temperature of the mixing chamber level, a temperature change is detected at the still. There, a small decrease of about 1 mK can be seen which can

FIG. 7. Graph (a) shows the temperature dependence of the mixing chamber and the still of two horizontal field sweeps between ± 1 T. In graph (b), the temperature of both levels is shown for a sweep between ± 7 T and a sweep down to 0 T. Reprinted from Ref. 24.

be explained by a perturbation of the still heater performance under the changing magnetic field. The slightly decreased heater output power influences the cooling of the mixing chamber in the order of 1–2 mK.

Fig. 7(b) shows the temperature dependence for high field sweeps. While the first sweep represents a magnetic field reversal between ± 7 T, the second shows the magnet ramping down from 7 T to 0 T. The base temperature is about 51 mK with the exit slit width set to 50 μm and the gold mesh moved out (compare to Fig. 7(a): 48 mK, gold mesh in, exit slit 100 μm). The influence of the mesh to the photon heat load by photon absorption can be seen nicely after the second sweep. There, the beamline valve is closed and the base temperature drops down to 45 mK. However, the field reversal from -7 to $+7$ T takes 23.3 min which is longer than the rising slope of the mixing chamber temperature. The maximum temperature of about 117 mK is measured at a magnetic field of 3.5 T, 6 min before the sweep has finished. Afterwards the dilution refrigerator needs 27.5 min to cool back down to base temperature. Hence, the overall sweep time lies around 51 min and is almost evenly distributed between the time of ramping the field and cooling back to base temperature. The still temperature is affected during the 7 T field sweep in the same order as in the 1 T sweep. A maximum mixing chamber temperature of 111 mK is measured for the second sweep from 7 T to 0 T. The rising slope of the mixing chamber has the same time scale as the sweep time and takes about 11.7 min. The mixing chamber is back at base temperature after a total time of 25 min.

From the performed measurements, it is easy to see that appropriate ramp rates have to be selected, depending on the samples investigated. For example, because for the highest ramp rates in the horizontal field direction the sample temperatures can be more than doubled, it might not be possible to study certain magnetic systems by field-cooled (FC) and zero-field-cooled (ZFC) analysis. In this case, slower ramping rates can effectively suppress the strong temperature increase but only under the high cost of expensive photon beamtime. This is an important point and has to be considered in the beamtime preparation.

During the commissioning time a massive coldfinger was mounted instead of the slotted designed. Therefore, the change of the mixing chamber temperature under a sweep of the

vertical magnetic field direction could only be tested under preliminary conditions. However, the maximum temperature for a field sweep between 0 T and 0.5 T was 88 mK. For a parallel field reversal between ± 7 T and ± 0.5 T the mixing chamber reached 138 mK.

IV. SUMMARY

We have built a new transportable experiment which provides sample temperatures down to 50 mK and magnetic fields up to ± 7 T, applicable at third generation synchrotron beamlines. A preparation chamber with load lock together with a cryogenic sample transfer system offer the possibility to investigate *ex-* as well as *in situ* prepared samples under UHV conditions. The cooling performance of the experiment with and without magnetic field was studied in detail. Commissioning studies at the P04 beamline at PETRA III have demonstrated that the photon heat load on the sample can be suppressed effectively if the photon flux is decreased by the exit slit of the beamline.

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