Application Note

easyLab

Innovative Solutions for Materials Characterisation

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easyLab Mcell 10 – 10 kbar hydrostatic pressure cell for Quantum Design MPMS measurement platform

Introduction

This application note introduces the new pressure cell from easyLab, Mcell 10, which has been specifically engineered to enable end-users to perform SQUID magnetometer measurements under high-pressure up to 10 kbar and at low temperatures down to below 2K.
In particular it is explained how the pressure cell is assembled and interfaced with the MPMS. Pressure loading results are presented up to 11.5 kbar. Finally, measurements performed on a sample of MnSi are presented and compared with published data.

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easyLab Mcell 10 module contents

The high-pressure cell module Mcell 10 represents a complete package providing the end-users with all the necessary primary components of the pressure cell and the interfacing components to connect to the MPMS measurement probe.

Very importantly, the easyLab Mcell 10 module is also supplied with the purposely-designed desktop hydraulic press ram (Mpress) and all the necessary consumables to enable several measurement campaigns

- Mcell body (1) and locknuts (2) – low magnetic material
- Ceramic pistons (2) and piston caps (2)
- Tungsten Carbide pusher (1)
- Liquid transmitting medium (40ml)
- Vials for Transmitting medium (2)
- Syringe (1)
- PTFE caps (20) and plugs (20)
- Anti-extrusion Cu rings (40)
- Mounting platform (1)
- MPMS probe interfacing rod (1)
- Cell centring sample – Nickel spheres (2)
- Sn manometers (10 cm)
- 10 ton hydraulic press ram including:
  - Hand pump (1)
  - Hydraulic hose (1)
  - Hydraulic cylinder (1)
  - Oil pressure gauge (1)
  - Pressure cell interfacing socks (3)
  - Extraction pusher (1)
  - Pressure cell centering ring (1)
  - Loading and unloading dies (2)
- Locking spanner (1)
- User guide (1)
- Storage box (1)

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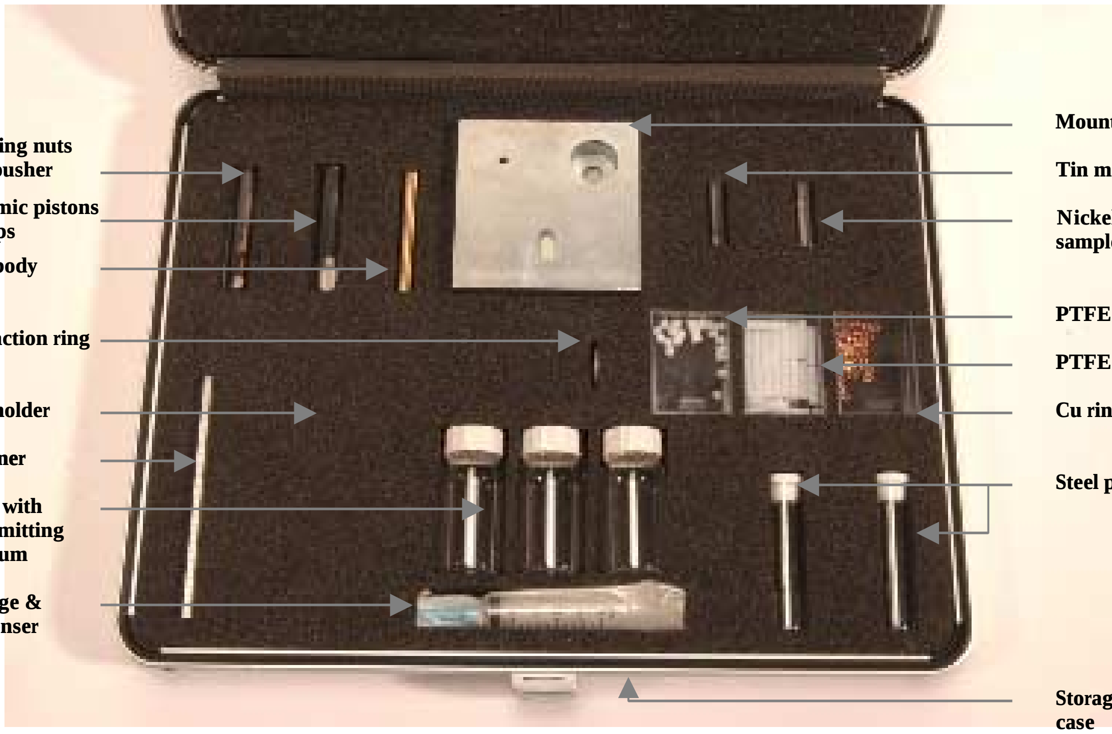

Figure 1: easyLab Mcell 10 module contents

easyLab Mcell 10 features and benefits

The Mcell 10 is a cylindrical hydrostatic pressure cell based on a single walled-design. The materials composing the cell have been carefully selected and tested to give the lowest magnetic background possible and yet maintaining high mechanical strength in order to withstand thermal and pressure cycles. The overall dimensions of the pressure cell have been optimised to give the maximum of sample space whilst still fitting in the sample space of the MPMS of Quantum Design. The concept of module has been developed to provide the novice as well as the advanced user with all the required tools to easily set-up, apply and release the pressure of the pressure cell.

easyLab Mcell 10 technical specifications

The Mcell 10 can achieve a maximum pressure of 10 kbar at room temperature and with a careful selection of the pressure-transmitting medium similar pressures can be achieved at low temperatures. The overall length of the pressure cell is 77 mm with an outer diameter of 8.5 mm. The available sample space is typically 1.9 mm diameter and 10 mm maximum long. The recommended maximum sample size is of 1.5 x 1.5 x 5 mm, which is within the acceptable limit to give a reasonable dipole response on the MPMS for most magnetic measurements.

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The total weight of the pressure cell assembled and ready to be mounted on the MPMS is 32 g, a weight that the MPMS actuator mechanism handles without any particular effort.

Although it does not make sense to provide any actual value of background susceptibility (as discussed later in this note) for the whole pressure cell, based on careful calibration and background pre-measurements, it is shown that measurements of samples with magnetic moments as low as 5.10^{-6} to 10^{-5} are possible.

easyLab Mcell 10 structure and main components

The figure below represents a 3D solid modelling exploded view and description of the Mcell 10.

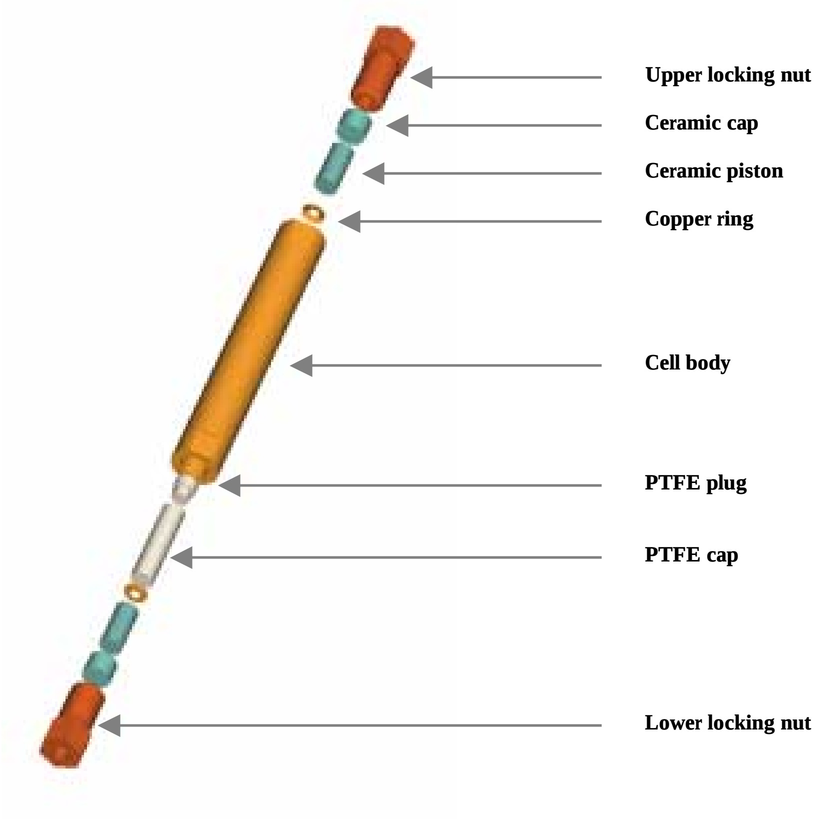

Figure 2: 3D modelling exploded view of the Mcell 10

Mcell 10 setting – up with the MPMS

In order to set up the pressure cell and mount it on the MPMS, we have engineered a range tools and accessories to facilitate these various steps. The setting up time is typically of around 30 minutes including sample mounting, pressure cell setting up, pressurisation and mounting on the MPMS. The pictures below represent these steps.

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a) Cell components on mounting platform
b) Cell after pressurisation
c) Cell mounted on MPMS probe
d) Cell in MPMS vacuum lock ready for loading


a)


b)


c)

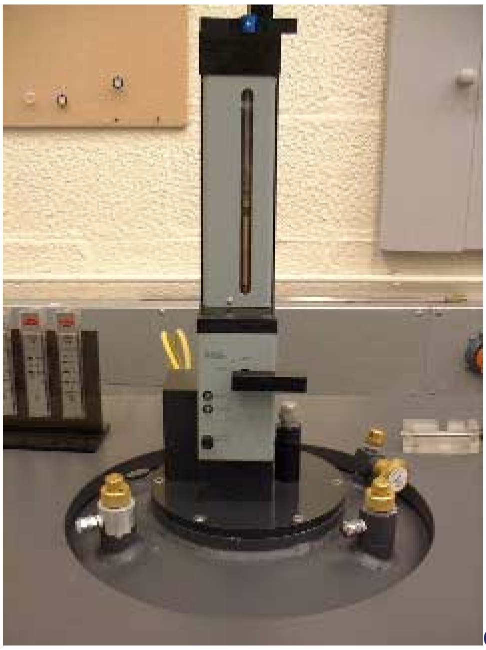
d)

Figure 3: Key steps of setting up the Mcell 10 on the MPMS

easyLab Mcell 10 Pressurisation

In order to apply pressure, a hydraulic pressure ram is required. Although any hydraulic press ram capable of generating at least 1.5 ton of force can be used, easyLab strongly recommend the use of our miniature desktop ram: Mpress. Indeed due to the relative small size of the cell it is essential to ensure the vertical alignment of all components during the pressurisation process. Otherwise, irreversible damages can be caused to the pressure cell. The Mpress includes key components ensuring the alignment of the cell during the pressurisation whilst maintaining a high degree of lateral safety protection for the end user.

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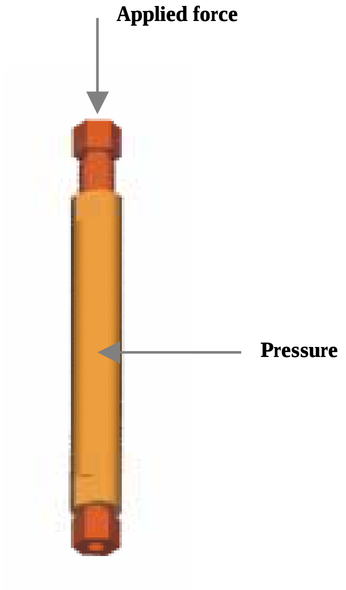
The way the cell is pressurised is by the application a known force on a tungsten carbide piston pushing onto the ceramic pistons. In turn, the PTFE cap, where the sample is sitting surrounded by the transmitting medium, has its volume reduced and hence the pressure increases.

The figure below shows a typical pressurisation curve at room temperature.



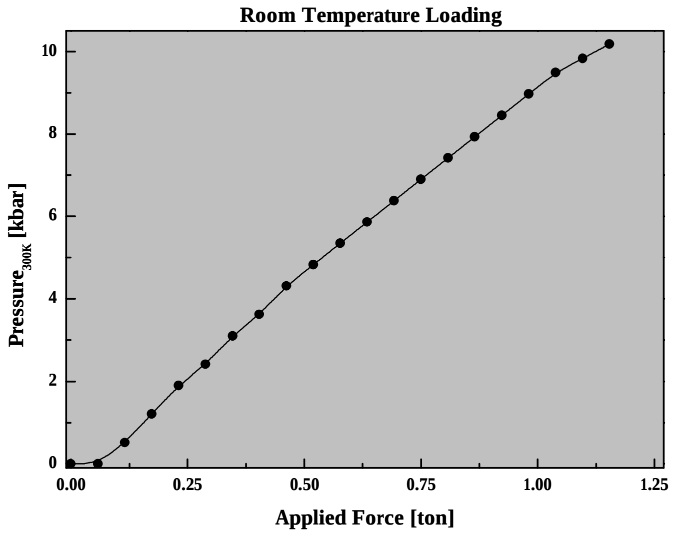


Figure 4: Typical loading curve of the Mcell 10 at room temperature

Once the pressure has been applied the pressure cell can be loaded onto the MPMS. However, due to the differential thermal contraction between the metallic components and the transmitting medium, one can expect some pressure variations when the system is cooled down. By a careful study and selection of the transmitting medium the Mcell 10 is only partially affected by this issue. The figure below represents the pressure profiles for various pressures as function of temperature.

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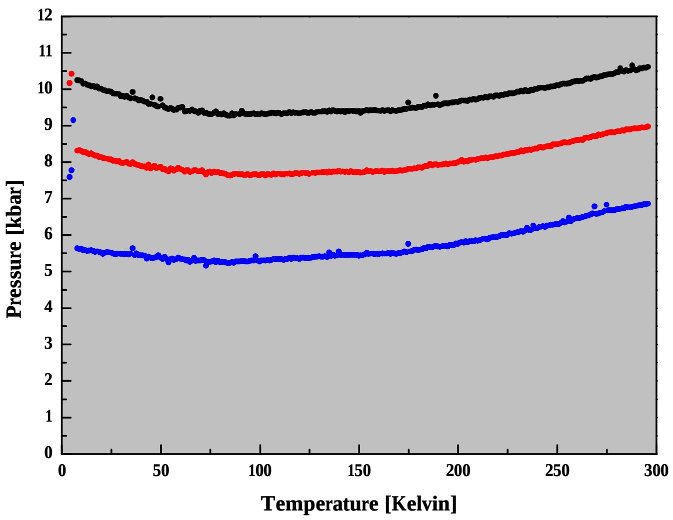

Figure 5: Pressure profile as function of temperature

In order to know the pressure at low temperature, two methods are possible:

1- Tin superconducting transition shift
2- Pressure calibration curve

In the case where the determination of the pressure needs to be very precise, it is preferable to use the first method. However, if the sample to be studied requires magnetic measurements below 4 K the second method should be chosen.

<u>1-Tin superconducting transition shift:</u>

One of the tin manometer samples supplied with the Mcell 10 is placed inside the PTFE caps with the sample to be measured. The value of the superconducting transition temperature, T_c [P], enables the estimate of the in-situ pressure as shown in the figures below.

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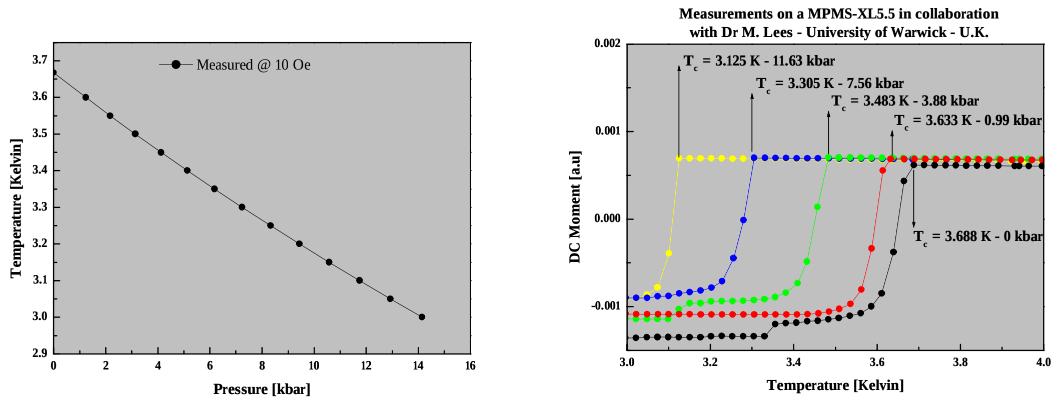

Figure 6: Pressure calibration based on the shift of the tin's superconducting transition temperature

The superconducting transitions presented on the right hand side panel have been measured on an MPMS-XL5.5 in collaboration with Dr Martin Lees of the University of Warwick. The figure below represents a typical screenshot of MultiVu of such transition.

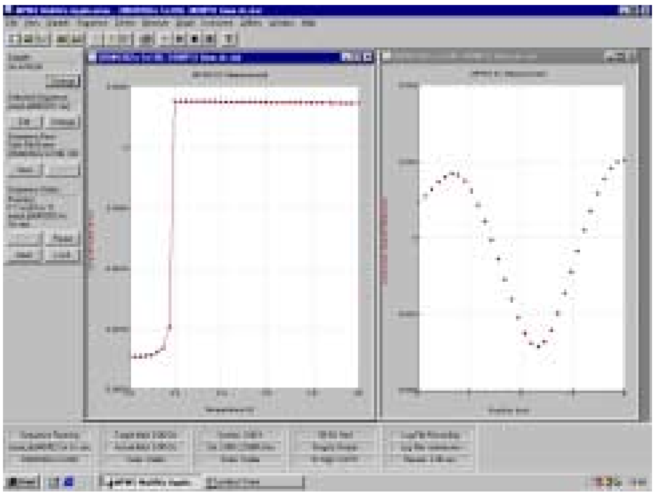

Figure 7: Screenshot of the tin transition – The right hand side panel represents the dipole response in the superconducting state at 3 K

<u>2-Pressure calibration curve method</u>

For some samples, the diamagnetic signal generated by the presence of the superconducting Sn sample might not be acceptable. In such a cases, the pressure needs to be estimated from the typical curve for pressure versus load for the Mcell 10. We studied for our Mcell 10, the relationship between the low temperature pressure and the applied force at 300 K. The results are presented in the figure below.

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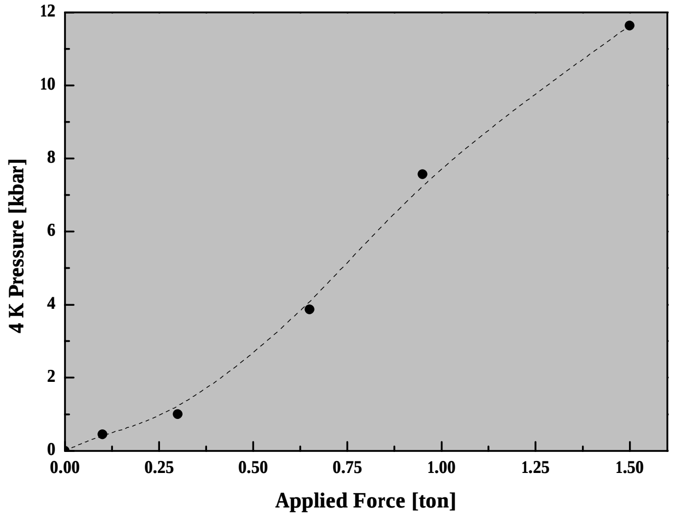

Figure 8: Low temperature calibration for the Mcell 10

From the applied force at room temperature, the pressure at 4K can thus be estimated. It has to be noticed that this method is less precise than the previous one.

easyLab Mcell 10 Background

One of the difficulties faced when measuring the magnetisation of a sample inside the Mcell 10 is that the signal detected by the SQUID is in fact the superposition of the magnetic trace of the cell and the sample. The situation is identical to a measurement of a sample mounted on a sample holder, whereas:

$$V_{Measured} = V_{Sample} + V_{Mcell10}$$

Consequently what is needed to estimate the magnetisation of the sample is:

$$V_{Sample} = V_{Measured} - V_{Mcell10} = V_{Measured} - V_{Background}$$

However, one of the challenges is that the signal associated with the pressure cell is a complex signal far from being the usual dipole response as the SQUID detection coils 'see' the pressure cell travelling through the coils.

The Automated Background Subtraction (ABS) feature of the MPMS MultiVu needs to be used to record the background raw data signals associated of the pressure cell empty. When the measurements with the sample inside the pressure cell are performed, then the option 'Subtract Background' should be selected.

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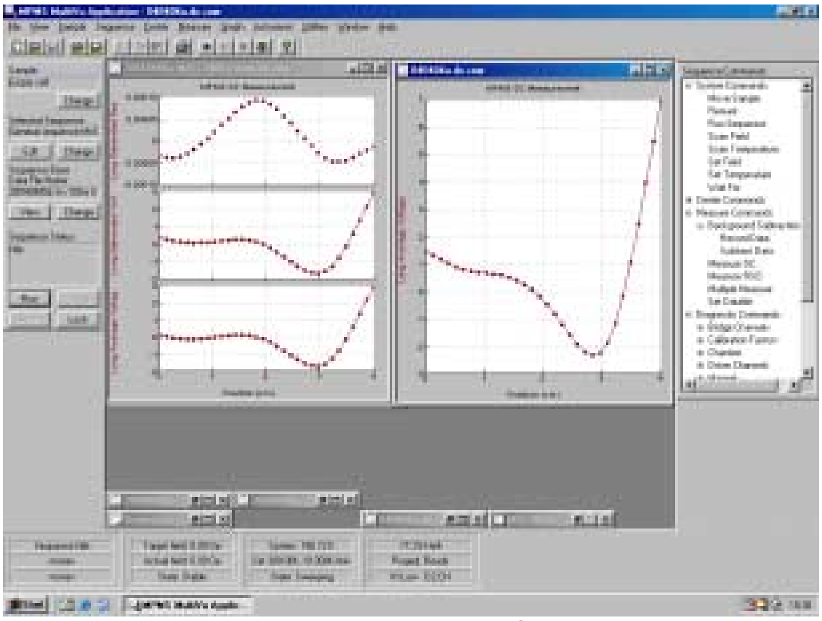

Screenshot of the background subtraction:

On the left hand panel are shown from bottom to top:

- Averaged measured signal
- Detrended measured signal
- Signal after background subtraction

The right hand side panel shows the raw recorded background signal.

Figure 9: Illustration of the background subtraction

<u>Note</u>: For more information on the effects of background subtraction on sample measurements and background subtraction techniques, see Quantum Design application notes 1014-201 and 1014-213.

Examples of measurements performed using the easyLab Mcell 10 with an MPMS-XL5.5

This work has been performed in collaboration with Prof. D. McPaul and Dr M. Lees from the University of Warwick (U.K.). The samples of MnSi have been kindly provided by Dr J. Flouquet & Dr G. Lapertot from the CEN.G in Grenoble (France).

The background of the Mcell 10 has been measured at 200 Oe across the whole temperature range. The sample signal has been obtained from the method explained in the previous section. Pressure has been regularly increased from ambient pressure to 11.6 kbar. The pressure effect is clearly observed in the figure below where the magnetic transition temperature is greatly reduced under pressure from 30 K to 13 K.

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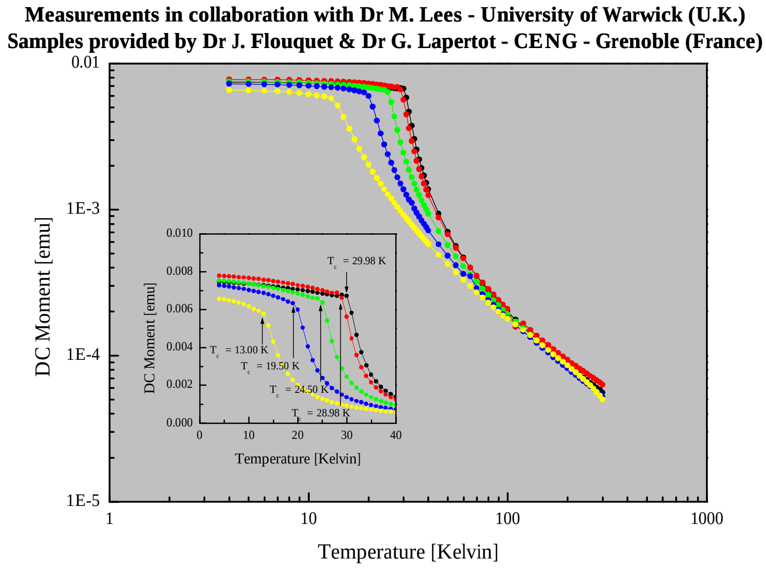

Figure 10: DC magnetisation of MnSi measured at 200 Oe for pressures 0, 0.99, 3.88, 7.56 & 11.63 kbar.

Based on these results, the magnetic phase diagram as function of pressure of MnSi can be compared with published results as shown in the figure below. The red points represent the results obtained with the easyLab Mcell 10, whereas the black points are previous results published by C. Thessieu *et al.*, Solid State Communications, 95, 707, (1995)

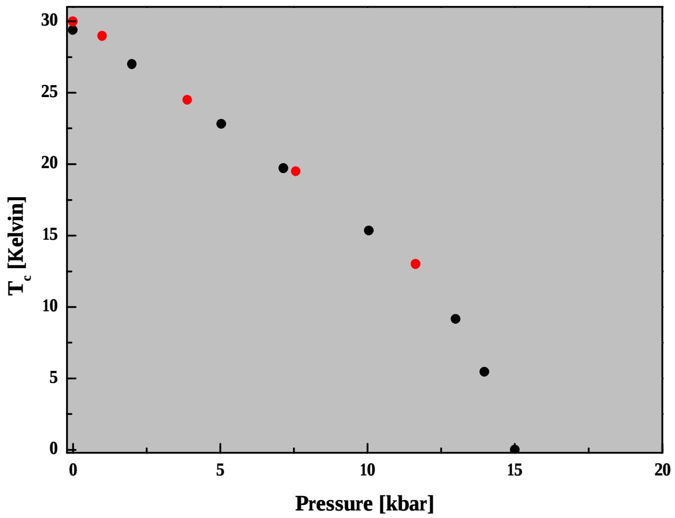

Figure 11: Magnetic phase diagram of MnSi as function of applied pressure

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