

High-pressure cell for a SQUID magnetometer with a plug for *in situ* pressure measurements

This article has been downloaded from IOPscience. Please scroll down to see the full text article.

2008 J. Phys.: Conf. Ser. 121 122001

(<http://iopscience.iop.org/1742-6596/121/12/122001>)

View [the table of contents for this issue](#), or go to the [journal homepage](#) for more

Download details:

IP Address: 129.215.196.1

The article was downloaded on 09/02/2013 at 14:58

Please note that [terms and conditions apply](#).

High-pressure cell for a SQUID magnetometer with a plug for *in situ* pressure measurements

J Sanchez-Benitez, S Tancharakorn, MK Hutchison, KV Kamenev¹
Centre for Science at Extreme Conditions and School of Engineering & Electronics,
University of Edinburgh, Mayfield Road, Edinburgh EH9 3JZ, UK

E-mail: K.Kamenev@ed.ac.uk

Abstract. We describe the design of a miniature high-pressure cell built for a Magnetic Property Measurement System based on the Superconducting Quantum Interference Device technology magnetometer. The key feature of the cell is the plug for *in situ* pressure measurements with feed-through wires connected to a manganin pressure sensor, which has a known pressure dependence of electrical resistivity. By monitoring pressure continuously during magnetisation measurements in the range of operational temperatures the true pressure can always be established.

1. Introduction

Magnetic Property Measurement System (MPMS) from Quantum Design (USA) is one of the most popular commercially available magnetometers utilising Superconducting Quantum Interference Device (SQUID) technology [1]. The MPMS can resolve magnetic moment changes as small as 10^{-8} emu over a wide range of temperatures and magnetic fields. Despite the major constraint of the instrument - the diameter of the inner bore of the MPMS is only 9 mm - there has been several pressure cells of the piston-cylinder type built for MPMS and pressure can now also be added as a variable for sensitive magnetisation measurements [2-8].

One of the problems however that needs to be resolved when the pressure cell is designed is that of the pressure measurement. There are several methods of doing this in the existing designs. However, all of them rely on a measurement of pressure at one particular temperature and then the pressure is *estimated* at any other temperature based on a certain assumption. This temperature correction is needed because of the pressure change on cooling or heating caused by a difference in thermal expansion of the pressure cell and the pressure-transmitting medium (normally, oil). In this work we take on the challenge to design the pressure cell in which pressure can be *measured* at any given temperature.

In the existing pressure cells for SQUID magnetometer the most frequently used method is by using a superconducting (SC) "manometer" like lead, tin or indium [9]. In these materials T_c is calibrated as a function of pressure and so by measuring the SC transition from a tiny speck of Pb, Sn or In one can accurately measure the pressure inside the pressure cell. The limitations of these method are that (i) it provides the pressure value for the temperature of the SC transition (typically at around 3-7 K); (ii) it produces a strong magnetic signal, which screens the sample and often makes it impossible to do an accurate measurements of the sample's magnetic susceptibility at temperatures

¹ To whom any correspondence should be addressed.

below the SC transition. The alternative method for measuring pressures is by either measuring elongation of the cell [9, 10] or by deriving the pressure from the load on the piston if a hydraulic press is used for applying pressure. This provides the pressure value at room temperature and eliminates the need for an SC "manometer" if measurements are to be done at very low temperatures. Combining the two methods described above it is possible to establish the absolute pressure change between the ambient and low (around SC transition) temperatures [10] which results from the difference in the thermal expansion coefficients of the pressure transmitting medium and the material of the pressure cell. However, determining the pressure in the vast temperature region between 300 K and about 3-7 K can be difficult. For simplicity it is often assumed that pressure changes linearly with pressure and the correction for the pressure is made based on this assumption. We show below that there is a deviation from linear dependence and using this approach can introduce a large error into the data analysis.

In large piston-cylinder cells there are two common ways for measuring pressure continuously as temperature is varied. One of them is with calibrated strain-gauges [11]. However implementing this method in the SQUID pressure cell design will be difficult as the strain-gauge will need to be positioned at the point of the maximum deformation which is in the middle of the cylindrical body of the cell, i.e. close to the sample position inside the cell, and thus will interfere with the magnetic signal from the sample. The other method is to measure pressure by measuring the resistance of a manganin coil located inside the pressure cell. Manganin is an alloy of 86% copper, 12% manganese, and 2% nickel. It has been developed by Edward Weston as the material with a very low temperature coefficient of resistance. Manganin also has its electrical resistivity calibrated as a function of pressure at any given temperature. There are several ways to calculate pressure from its resistivity [12, 13] of which the simplest one is the following:

$$R(P) = R(P_{\text{amb}}) (1 + \alpha P)$$

where $R(P)$ and $R(P_{\text{amb}})$ are resistivities of the manganin at pressure P and ambient pressures respectively, $\alpha = 0.002465 \text{ kbar}^{-1}$ and P is pressure in kbar.

Manganin pressure sensors are normally produced as small coils and introduced into pressure cells by means of an electrical plug. The design of the plug is typically based on a reversed Amagat cone [14]. The conical window itself can be made of steels, alloys or other metals in which case the wires are electrically insulated from the plug by use of pyrophyllite or resin epoxy [15, 16]. In some cases the Amagat window is made exclusively of pyrophyllite or epoxy [17 - 20]. However, all of the above plugs have been produced for rather large pressure cells. The bore of a SQUID pressure cell is typically 2.5 - 3.0 mm in diameter and the outer diameter is restricted by the 9 mm bore of the magnetometer. Introducing a plug with electrical feed-throughs into such a small bore was the first major challenge we needed to overcome in the design process.

2. Design

The conventional design of a feed-through plug for large-volume pressure cells typically consists of two parts - the plug itself and a backing screw used to push the plug into the bore and engage the seals. The advantage of using this 2-piece method is that as the screw turns the plug is engaged into the translational movement only, thus avoiding the twisting action at the neck of the plug once the metal seal is engaged. However, our estimates have shown that the space restrictions would make it impossible to implement the 2-piece design for a plug with feed-throughs in the case of a miniature pressure cell for the SQUID magnetometer. The reason for this is that it would be difficult to pull out the plug out of the pressure cell during the disassembly because it is virtually impossible to reach to it and to grip it firmly inside the hole in the cylinder of the cell which is less than 6 mm in diameter.

We therefore decided to use a 1-piece design in which the plug and the screw are made as a single part. The design of the plug required careful calculations based on the strength of materials involved in order to avoid the above mentioned problem of the neck of the plug being effectively "welded" to the bore of the cell once the metal seals are engaged. The drawing of the plug is shown in Fig. 1.

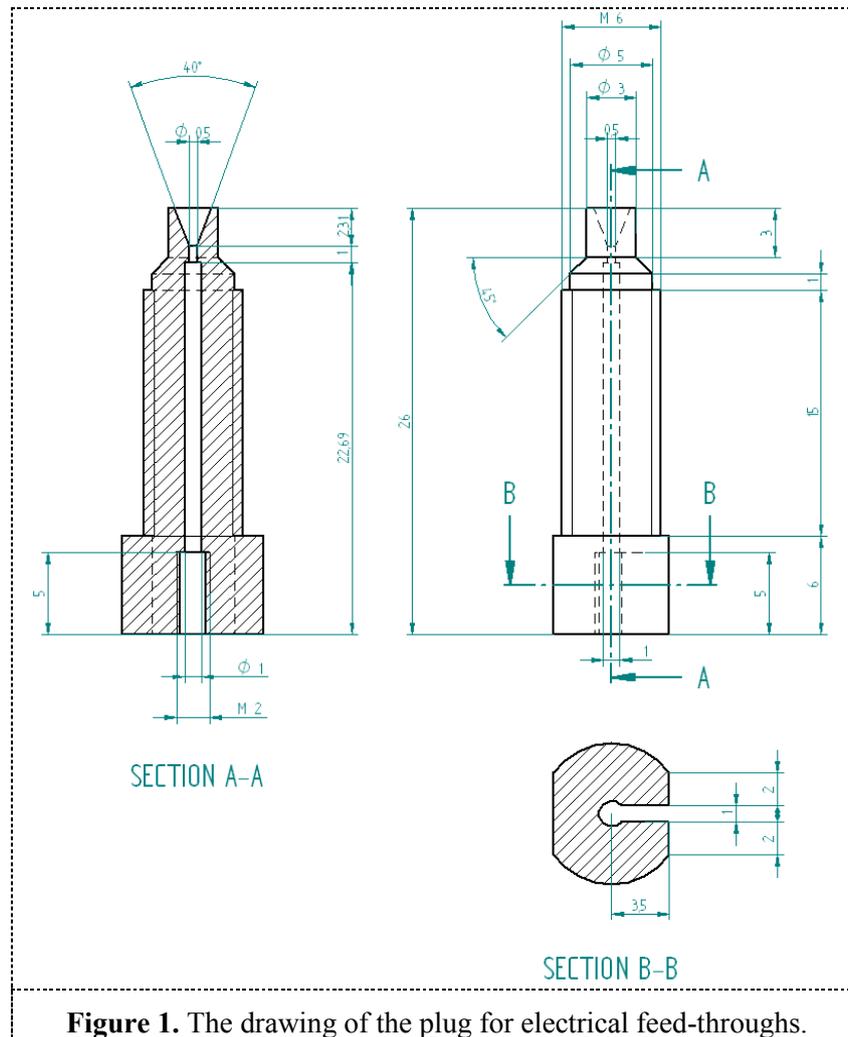


Figure 1. The drawing of the plug for electrical feed-throughs.

The plug is made of BERYLCO-25 alloy (NGK Berylco U.K. Ltd). The feed-throughs are made of up to 20 enamelled copper wires of 0.1 mm diameter. Although only 4 wires are required for the resistivity measurement we have found having spare wires useful in case of any possible breakages. The wires are introduced into the pressure cell through a narrow neck of 0.5 mm in diameter, which at the top end opens up into a conical hole for a reversed Amagat cone and at the bottom end becomes a 1.0 mm diameter channel for taking the wires out. Once the wires are introduced into the plug, the conical neck is sealed with Stycast 2850 FT epoxy (Emerson & Cuming Inc.) with catalyst 24 LV, as this has proved to be the most reliable at low temperatures. The epoxy is allowed to go down the channel under the force of gravity without any extra suction force at the opposite end of the plug. This has been found to give the most uniform distribution of the epoxy without air bubbles trapped in it. The external end of the plug has a 5 mm long and 1 mm wide slot cut to take the wires out through the side of the plug and an M2 thread is made inside the channel to hold the pressure cell (once assembled) on the MPMS sample rod. A copper seal is put on the 3 mm neck to provide the seal between the cylinder of the pressure cell and the plug.

The cross-sectional view of the assembled pressure cell is shown in Fig. 2. The pressure is applied in a hydraulic press and is generated in the oil pressure-transmitting medium by a piston at the end opposite to the plug. The pusher shown in Fig. 2 is removed once the pressure is applied. The oil used in our experiments is Daphne 7373 (IDEMITSU-ILS) and the maximum pressure that can be achieved in this pressure cell is 10 kbar. The copper wires terminate on a 4-pin plug that connects to a 4-pin

socket assembled on an adapter connected to the standard MPMS sample rod as shown in Fig. 3. The rod is hollow and sealed at the opposite end to eliminate any leak of air into the sample chamber.

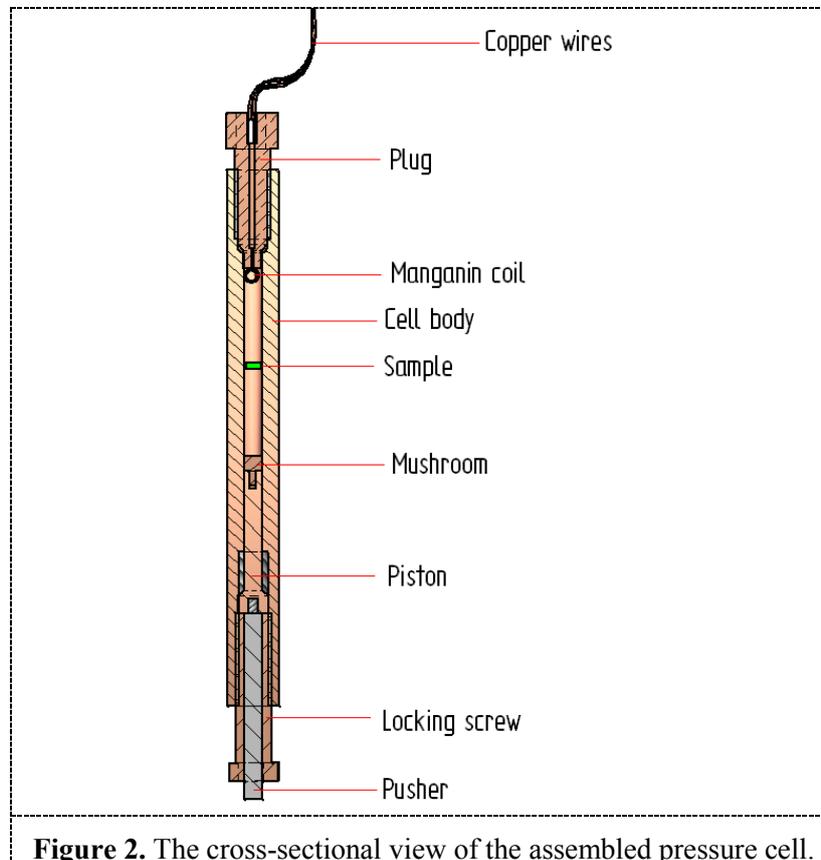


Figure 2. The cross-sectional view of the assembled pressure cell.



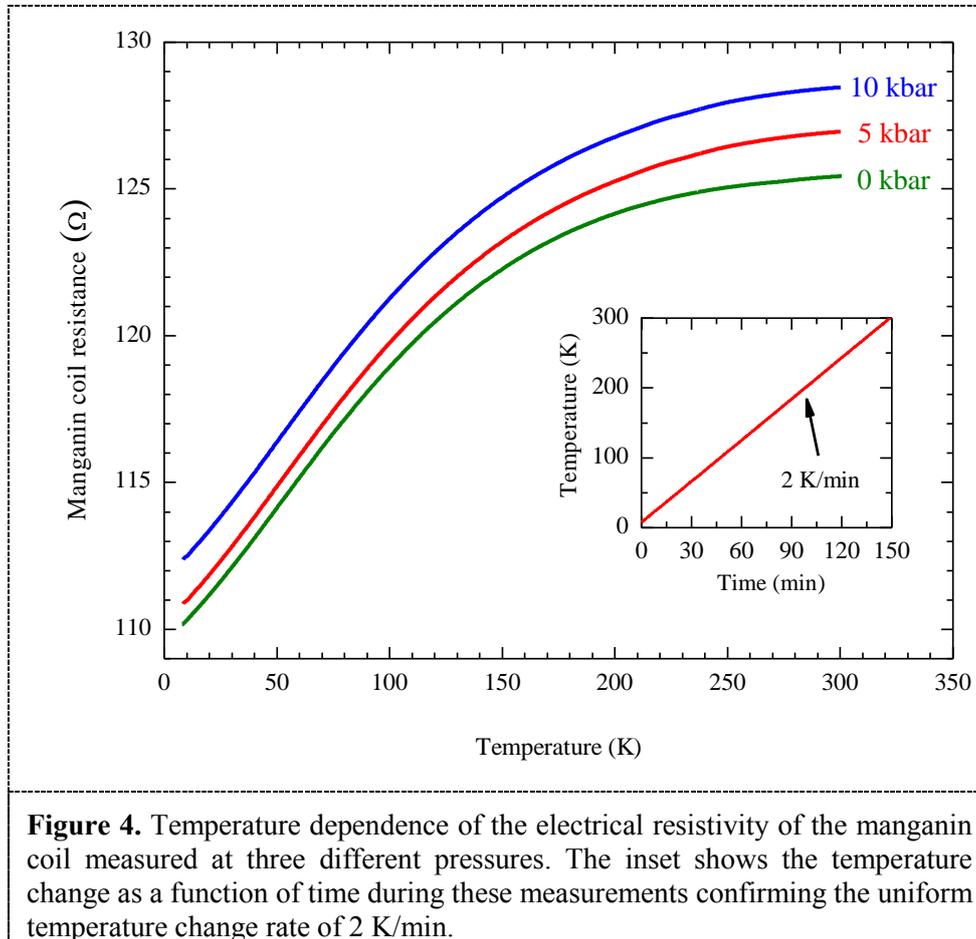
Figure 3. Assembled pressure cell attached to the MPMS sample rod through an adapter with a 4-pin socket mounted on it.

3. Tests & Measurements

There are several ways in which the data acquisition can be arranged and the resistivity data collected from the manganin pressure sensor. One of them is through the use of RP013 resistivity option for MPMS provided by Quantum Design. It allows to incorporate the resistivity measurements through the MultiVu software that runs the magnetisation measurements in the MPMS and write the resistivity data into the same data file as the rest of the acquired data such as magnetisation, temperature, magnetic field, etc. The alternative way is to use any data acquisition software for collecting the manganin resistivity data and to run it in parallel with MultiVu. This would produce two data files which can then be combined in one set of data based on the time column which can be included into both files.

We have measured the resistivity of the manganin sensor in a pressure cell assembled with a sample mounting table and a sample inside as a function of temperature in the MPMS at a number of pressures. Because of the limited space available for this paper we will report the results of the

pressure dependence of the magnetic properties of the studied material elsewhere. The purpose of the test was to establish how the pressure actually changes inside the pressure cell at temperatures between 300 K and 2 K. The manganin coil resistance measured as a function of temperature at starting pressures (as applied at room temperature) of 0, 5 and 10 kbar is shown in Figure 4.



The measurements were performed at a number of temperature change rates and the optimum value was found to be 2 K/min, which is sufficient for the pressure cell to remain in thermal equilibrium with the environment during heating/cooling runs. The data were then treated using the equation quoted above and the results of the analysis for the initial pressure of 5 kbar is presented in Fig. 5. The solid line in Fig. 5(a) shows the actual pressure inside the pressure cell as measured by manganin gauge. The overall drop in pressure between 300 K and 2 K is just under 2.5 kbar. This has been observed for the Daphne 7373 oil before [6,10], however from the shape of the line it is clear that the linear approximation for the pressure change (dashed line in Fig. 5(a)) is inadequate and can result in an error of up to 0.6 kbar in pressure determination (Fig. 5(b)).

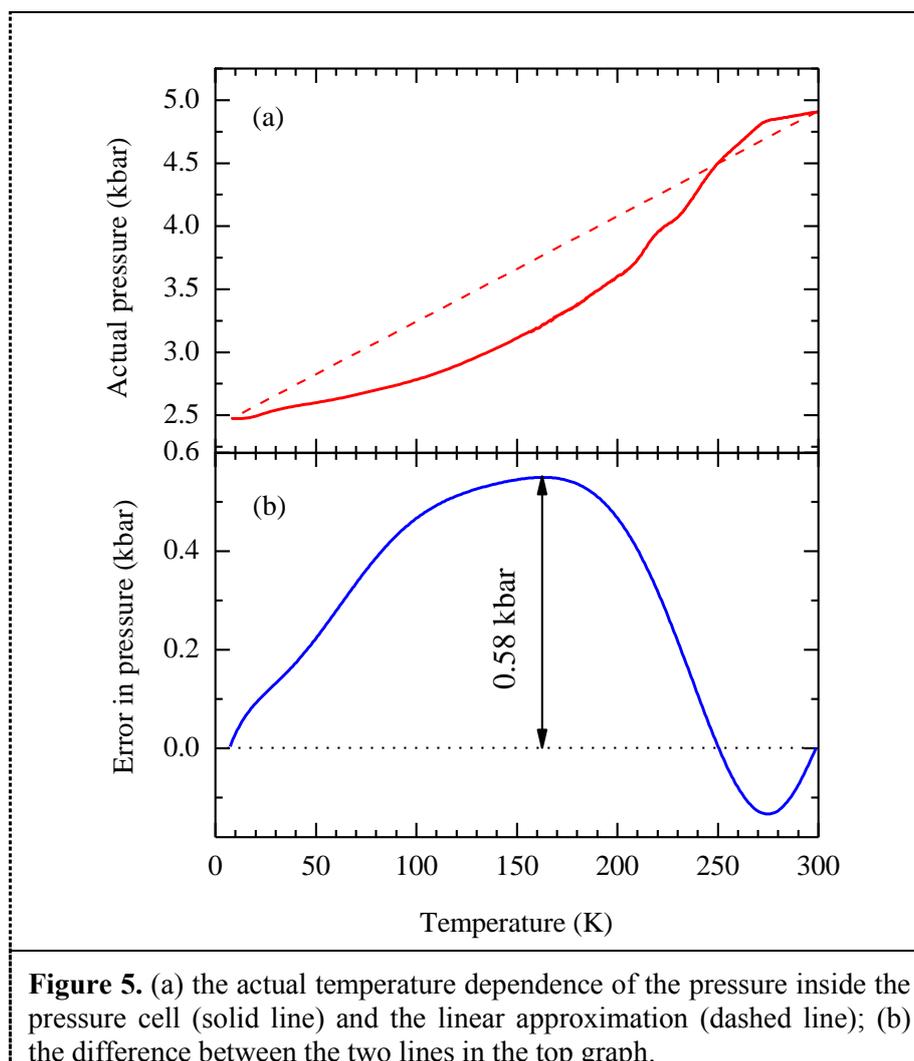


Figure 5. (a) the actual temperature dependence of the pressure inside the pressure cell (solid line) and the linear approximation (dashed line); (b) the difference between the two lines in the top graph.

It is hard to interpret the shape of the P(T) curve but it is clear that some features in it are due to the pressure transmitting fluid changing its state from liquid to solid. Other features are due to changes in the rest of the sample environment such as for example the thermal expansion of various parts of the pressure cell assembly, e.g. sample mounting table, piston, seals or the sample itself in the end. These changes can be specific to each individual measurement and thus the use of the *in situ* measurement of pressure can greatly improve the accuracy of the data especially in cases when the material studied is sensitive to pressure or when a fluid with unknown properties is used as pressure-transmitting medium.

We should also comment on the change of the resistivity of the manganin wire in magnetic field due to the magnetoresistance effect. It has been found recently that the magnetoresistance of the manganin is very low (+0.69% in the field of 10 T at temperature of 4 K) [21] and can in most cases be neglected. Alternatively, a correction can be made to take this change into account in calculating the actual pressure inside the pressure cell.

In conclusion, we have designed a pressure cell with a plug for *in situ* pressure measurement which has been tested to pressures of 10 kbar. It has been shown that the pressure inside the pressure cell has a complicated temperature dependence due to the thermal expansion coefficients of the materials used in the construction of the cell and that of the sample itself. This paper aims to provide sufficient information for the researchers interested in building and using the plug and the pressure cell.

4. References

- [1] Quantum Design, web-site: <http://www.qdusa.com>
- [2] Reich S and Godin T 1996 *Meas. Sci. Technol.* **7** 1079
- [3] Diederichs J, Gangopadhyay A K and Schilling J S 1996 *Phys. Rev. B* **54** R9662
- [4] Uwatoko Y, Hotta T, Matsuoka E, Mori H, Oki T, Sarrao J L, Thompson J D and Mori N 1998 *Rev. High Pressure. Sci. Technol.* **7** 1508
- [5] Kamishima K, Hagiwara M and Yoshida H 2001 *Rev. Sci. Instrum.* **72** 1472
- [6] Kamarád J, Machátová Z and Arnold Z 2004 *Rev. Sci. Instrum.* **75** 5022
- [7] Mcell 10 pressure cell manufactured by easyLab Technologies; web-site: <http://www.easylab.co.uk/mcell.html>
- [8] Uwatoko Y, Fujiwara T, Hedo M, Tomioka F and Umehara I 2005 *J. Phys.: Condens. Matter* **17** S1011
- [9] Eiling A and Schilling J S 1981 *J. Phys. F: Metal Phys.* **11** 623-39
- [10] Kamenev K V, Tancharakorn S, Robertson N and Harrison A 2006 *Rev. Sci. Instrum.* **77** 073905
- [11] Kamarád J, Kamenev K V and Arnold Z 1996 *High Pressure in Science and Technology*, ed W A Trzeciakowski (Singapore: World Scientific) pp. 51–53
- [12] Andersson O E and Sundqvist B 1997 *Rev. Sci. Instrum.* **68** 1344
- [13] Atanov Yu A and Ivanova E M, Translated from 1971 *Izmeritel'naya Tekhnika* **2** pp. 46–48
- [14] Bridgman P W 1931 *The Physics of High Pressure* (London: G. Bell and Sons, Ltd.)
- [15] Downs J L and Payne R T 1969 *Rev. Sci. Instrum.* **40** 1278
- [16] Schirber J E and Shanfeldt D W 1968 *Rev. Sci. Instrum.* **39** 270
- [17] Hammons B E 1971 *Rev. Sci. Instrum.* **42** 1889
- [18] Lazarre F 1953 *J. Phys. Radium* **14** 213
- [19] Dugdale J S and Hulbert J A 1958 *Can. J. Phys.* **35** 472
- [20] Petrova A E and Stishov S M 2006 *Instr. and Exp. Techniques* **49** 592
- [21] Abrecht M, Adare A and Ekinb J W 2007 *Rev. Sci. Instrum.* **78** 046104

Acknowledgments

This work was supported by EPSRC Grants EP/E06471X/1 & EP/D503744/1. We also thank the Royal Thai Government for a studentship (ST).