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Publisher: Taylor & Francis

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High Pressure Research: An International Journal

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/ghpr20>

Miniature diamond anvil cell for ^3He insert into quantum design physical property measurement system

K. V. Kamenev ^a, J. Sanchez-Benitez ^a & S. Tancharakorn ^a

^a Centre for Science at Extreme Conditions, School of Engineering and Electronics, University of Edinburgh, The Kings Buildings, Edinburgh, EH9 3JZ, UK

Version of record first published: 03 Apr 2007.

To cite this article: K. V. Kamenev, J. Sanchez-Benitez & S. Tancharakorn (2007): Miniature diamond anvil cell for ^3He insert into quantum design physical property measurement system, High Pressure Research: An International Journal, 27:1, 189-192

To link to this article: <http://dx.doi.org/10.1080/08957950601000054>

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Contributed Paper

Miniature diamond anvil cell for ^3He insert into quantum design physical property measurement system

K. V. KAMENEV*, J. SANCHEZ-BENITEZ and S. TANCHARAKORN

Centre for Science at Extreme Conditions, School of Engineering and Electronics,
The Kings Buildings, University of Edinburgh, Edinburgh EH9 3JZ, UK

(Received 15 June 2006; revised 10 July 2006; in final form 10 July 2006)

We report on the design of a miniature high-pressure cell designed to work at sub-Kelvin temperatures with ^3He insert into a commercially available physical property measurement system (PPMS) which is popular among researchers. The issue of severe space limitation around the ^3He sample platform has been overcome by using a new design for an opposed-anvil pressure cell, which we called a split-cylinder design. To our knowledge, this is the first pressure cell created for ^3He insert into PPMS and probably one of the smallest pressure cells ever made for resistivity measurements. The pressure cell itself has been built from non-magnetic Cu–Be alloy and used with diamond anvils generating pressures of 10 GPa. The cell has been used for electrical resistivity measurements using standard PPMS data acquisition system. In addition to the pressure cell, a special holder has been built to maintain the alignment of the cell during pressure applications.

Keywords: PPMS ^3He insert; Diamond anvil cell; Electrical resistivity measurements; Quantum design; sub-Kelvin temperatures

1. Introduction

The physical property measurement system (PPMS) from Quantum Design, USA [1] is the instrument based on a superconductive cryomagnet with various inserts for measuring physical properties of matter such as electrical resistivity, magnetic susceptibility, heat capacity, etc., at a wide range of temperatures and magnetic fields. Because of its user-friendliness and versatility, PPMS has become the instrument of choice for researchers worldwide. Recently in addition to magnetic field and low temperatures pressure was added as a variable. These are piston-cylinder cells with maximum pressure limit of up to 3 GPa [2, 3]. These pressure cells are used with the mainframe PPMS, which has the lowest temperature of 1.8 K. PPMS also has a low-temperature option which is based on ^3He refrigeration and capable of reaching the temperature of 0.35 K. We took the challenge to design a high-pressure cell that could be used with the ^3He insert for electrical resistivity measurements and was capable of reaching the pressure of 10 GPa.

*Corresponding author. Email: k.kamenev@ed.ac.uk

2. Design: challenges and solutions

The major problem was the size of the sample space available in the ^3He insert. It is a semi-cylinder of 22 mm in height with a radius of 13 mm. We have considered several options, trying to optimize the use of available space, and stopped on the design which we called a 'split-cylinder'. The pressure cell is shown in figure 1. Its body is made of a cylinder with a diameter of 26 mm, which is cut in half along its main axis and then one of these halves is cut in half again perpendicularly to this axis, producing the top and the bottom plates (figure 1). This proposed design is somewhat similar to the one proposed by Merrill and Bassett [4], but the shape of the cell, the geometry of the screws, alignment rods and the position of the holes for alignment had to be optimized with the dimensional restrictions in mind.

Three pins align the plates and allow them to slide vertically while remaining parallel to each other. One diamond anvil is glued to the bottom plate directly. The other anvil is glued to a short cylinder which is then inserted into the recess in the top plate. The anvil on this cylinder can then be aligned with the stationary anvil horizontally by positioning this short cylinder with three grub-screws, which are inserted at an angle of 120° to each other. Once the anvils are aligned the force can be applied by means of three M4 screws. The cell was made of non-magnetic BERYLCO-25 alloy. Bevelled diamonds with $800\ \mu\text{m}$ culet were used as anvils. In order to make resistivity measurements, in our pressure cell, we used fine gold wire for making electrical feed-throughs and laminated the stainless steel gasket with MgO powder to insulate the wire. Pressure was measured by using a standard Raman spectroscopy technique on a tiny ruby chip inside the hole in the gasket.

Taking into account the dimensions of the cell and in order to aid the alignment during loading we constructed a holding fixture, which is a tight fit around the cell and which guides the cell during loading (figure 2). It is worth emphasizing here the importance of high-precision machining of all parts in order to achieve the perfect alignment of the anvils. This has been achieved by using grinding and honing techniques.

The next problem we needed to solve was the electrical and thermal coupling of the pressure cell to the ^3He insert into PPMS. To make the electrical connection to the socket on the ^3He

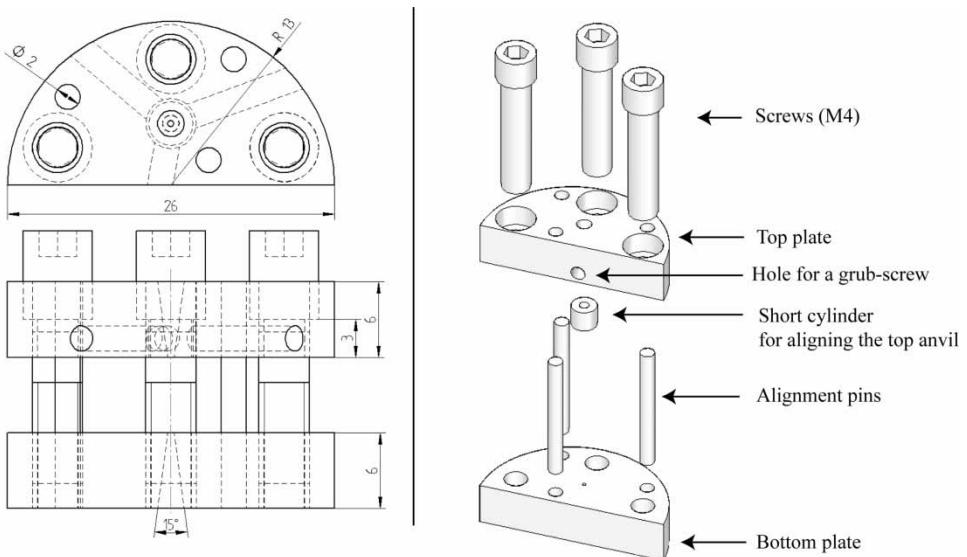


Figure 1. Drawing of the pressure cell with key dimensions and exploded view diagram.

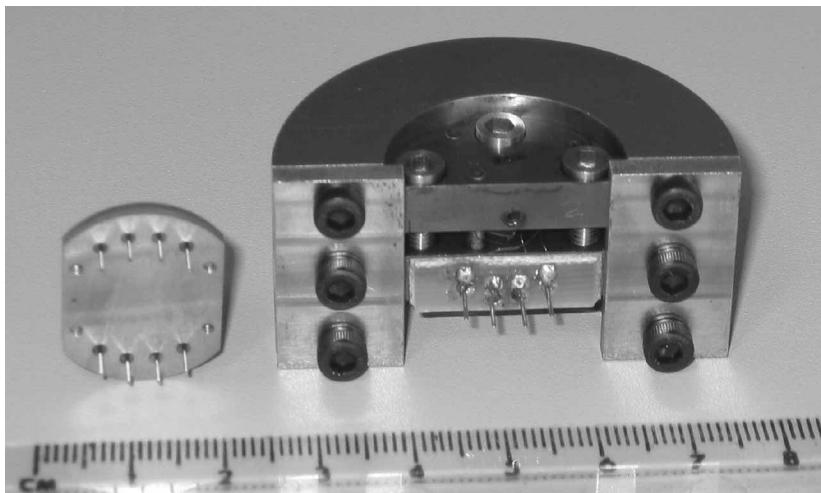


Figure 2. Pressure cell inside the holding fixture (right) and a standard ^3He PPMS puck (left).



Figure 3. The pressure cell on the ^3He platform.

insert, which is in ordinary use used for plugging sample pucks, we used a strip of a printed circuit board (PCB) with suitably drilled holes for the plug-pins and mounted on the bottom plate of the pressure cell (figure 2). We used the same gold-plated pins that are used in standard PPMS sample pucks (Kensington Electronics, USA), mounting them into the holes next to the conductive pads. The feed-through wires coming out of the hole in the gasket are soldered to these conductive pads. The PCB strip is then covered with a thin plastic film with holes for the pins to insulate the pads and wire and to avoid a short-circuit on contact with the ^3He platform. The cell could then be plugged into the ^3He socket (figure 3) and the standard PPMS data acquisition system could be used for making electrical resistivity measurements. Thermal coupling of the cell to the ^3He stage has been achieved through the top plate of the cell attached via a spacer to the ^3He sample platform by means of the platform screw. N-type grease is applied between the cell and the platform to enhance the thermal conductivity.

During the tests of the pressure cell with the ^3He insert, the PPMS reached the lowest temperature of 0.35 K. The temperature was measured by the thermometer on the ^3He platform. The change in pressure with temperature can be estimated from measurements on materials with known pressure dependence of the superconductive critical temperature.

Acknowledgements

We acknowledge the support from the Royal Society and from EPSRC (Grant EP/D503744/1). We also thank the Royal Thai Government for a studentship (ST).

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