

# Materials characterisation service

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High-pressure is no longer a specialised tool. It has become vital to fields ranging from geophysics to optoelectronics, from polymers to the production of vaccines (see e.g. Dunstan and Scherrer, 1998). Take for example high-pressure biotechnology. This is an emerging technique initially applied for food processing and more recently in pharmaceutical and medical sciences. Here pressure enables enzymes to be stabilized and both their activity and specificity can be modulated as a function of pressure. High-pressure engineering of proteins may be used for enzyme-catalysed synthesis of fine chemicals, pharmaceuticals, and production of modified proteins of medical or pharmaceutical interest.

In essence, the strength of high-pressure studies rests in its ability to enable the tuning, in a controlled and reversible manner, of the volume of a sample and, therefore, the ensuing properties of that sample. The application of pressure can produce structural, electronic and other phase transitions, polymerisation of organic substances, and many other phenomena (Eremets 1996).

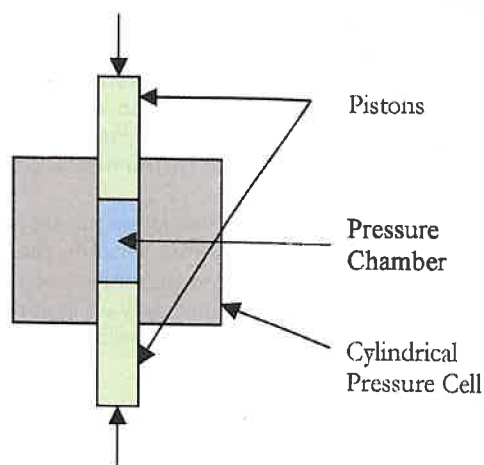
This expansion of fields benefiting from studies at high-pressures has inevitably led to the demystification of high-pressure techniques and equipment – a field notoriously dominated by experts. Thus, for example, the commercial development of miniature diamond anvil cells (which can generate pressures above 400 GPa or 400,000 ambient pressure) supplanted equipment weighing many tons, with essentially an instrument that could be held in the palm of the hand. These diamond anvil cells enable a variety of optical, x-rays and other measurements to be carried out at high and low temperatures and high magnetic fields in a safe and user-friendly fashion.

At easyLab we have commercially developed miniature piston-cylinder type cells. Although these cells are limited to the range of pressures up to above 3 GPa, they allow for much larger samples to be studied. Furthermore, our piston-cylinder cells make possible for different techniques to be used, such as electrical resistivity and magnetisation. In what follows we briefly discuss the technical aspects of achieving high-pressures (up to 3 GPa) with piston-cylinder cells. Then we present typical engineering and scientific results obtained with this type of equipment.

## Technical Details

Piston-cylinder high-pressure cells are also referred to as hydrostatic cells. This is because pressure homogeneity has been shown to be very good with these devices. Of course, hydrostatic conditions are also obtained in diamond anvil cells, but piston cylinder remains one of the most obvious methods for pressure generation.

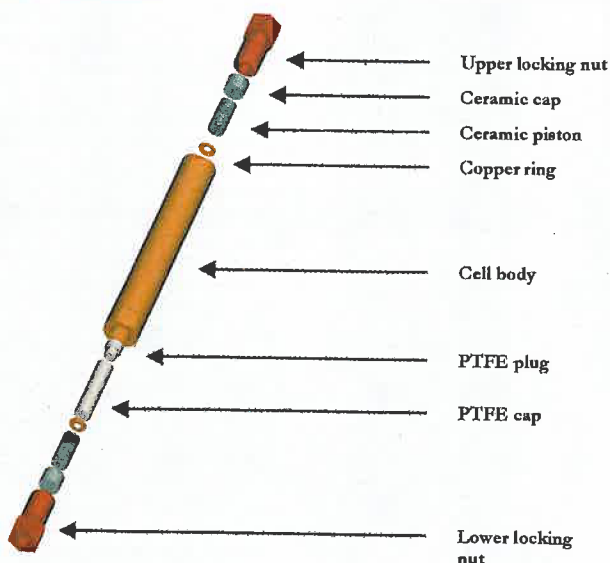
Fundamentally, a pressure-transmitting medium (within which the sample resides) is compressed, inside a relatively thick-walled cylinder, by pistons pushing in on either side (see Fig. 1). The main challenges faced in the optimisation of the performance of these cells are related to the choice of materials in general (but, in particular, for the cylinder and pistons), as well as, obtaining



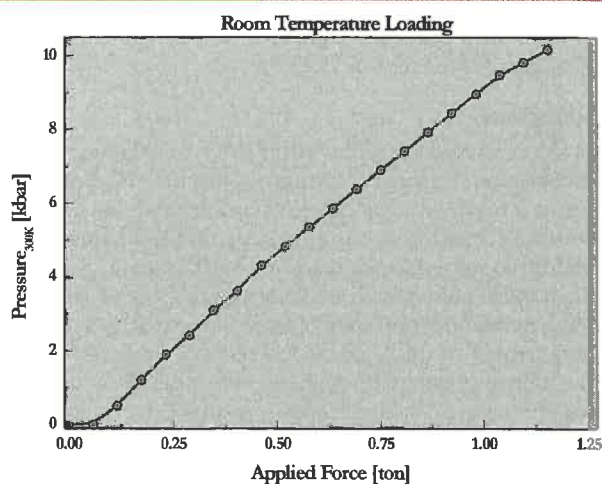
▲ Fig. 1: Basic principle of the piston-cylinder type pressure cell.

robust feedthroughs for wiring (if required). These must be allied, of course, to accurate design and precise engineering if such high-pressures are to be maintained whilst avoiding leaks.

In order to maximise working pressure one needs to look for materials with the highest tensile strengths, as well as, modifying the internal stress distribution of the cell. As far as materials are concerned, beryllium copper is one of the most widely used materials in the construction of cylinders for hydrostatic cells. It is relatively non-magnetic, and has an ultimate tensile strength of the order of 1.5 GPa. Matsumoto (2002) has compiled a useful list of other suitable materials. We have carefully chosen the materials for our cells depending on the maximum pressure to be achieved as well as the type of measurements that will be undertaken. Hence, for example, for our high-pressure cell for magnetisation, we have selected appropriate materials that, when combined in the cell, give a total low magnetic background. Figure 2 represents a 3D solid modelling exploded view and description of this cell.



▲ Fig. 2: 3D modelling exploded view of easyLab Mcell 10.



▲ Fig. 3: Typical loading curve of the easyLab Mcell 10 at room temperature.

Employing special techniques, such as, using short tapered plugs, using gaskets to support the plugs, etc, can modify internal stress distribution. However, in order to achieve the highest possible pressures with this construction, interference fit needs to be employed. Interference fit is achieved by the insertion of various cylindrical shells into each other. By force fitting an inner 'liner' inside an outer jacket, the zone close to the bore is put under compression, while the outer regions are put under tension. Hence a 'double-walled' piston-cylinder high-pressure cell that is pre-stressed in this way has a more favourable stress distribution when loaded than a conventional single-walled cylinder cell. This is because the outer regions carry a higher tensile load, thus pushing up its tensile limit. Thus, depending on the maximum pressure required for experimentation, our high-pressure cells employ these methods. Our cell for magnetisation is a single-walled cell, which achieves pressures in excess of 1 GPa. On the other hand our high-pressure cell for electrical measurements employs a double-walled construction.

The making of consistently reliable electrical feedthroughs is an art on its own. Although there are many known methods of introducing electrical leads into high-pressure cells, the most attractive involves the use of a tapered cone stopper (Eremets 1996). Wires are passed through the hole and glued into position by using, say, epoxy resin. Although this sounds trivial, extensive care needs to be applied when undertaking the preparation of such parts. The metal surfaces must be impeccably clean, dry and warm. The epoxy needs to be degassed prior to use. Any imperfections on the seal will translate into failure. We have developed our own techniques for obtaining reliable feedthroughs. Furthermore, our feedthroughs come already wired with the necessary (room and low temperature) resistive manometers.

### Typical Results

easyLab currently offers three different piston-cylinder pressure cell modules to the market:

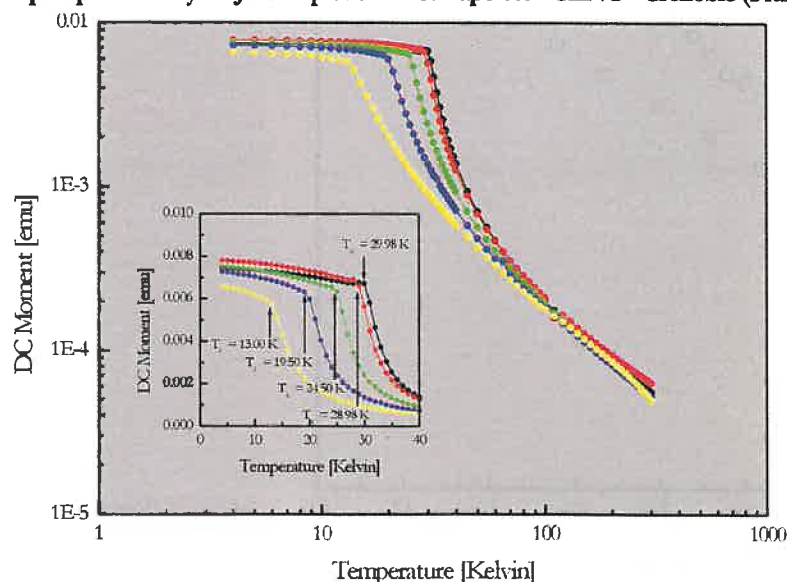
easyLab Mcell 10 is a high-pressure cell module that was specifically engineered for magnetometry under high-pressures up to above 1 GPa. It is fully compatible with commercially available squid magnetometers.

easyLab Pcell 15 and Pcell 30: these modules enable transport measurements up to 1.5 and 3 GPa, respectively. Although they were engineered to seamlessly integrate to Quantum Design's Physical Properties Measurement System platform, these modules may also be used in dilution refrigerators,  $^3\text{He}$  and  $^4\text{He}$  cryostats, as well as cryo-coolers or any other cryogenic platform

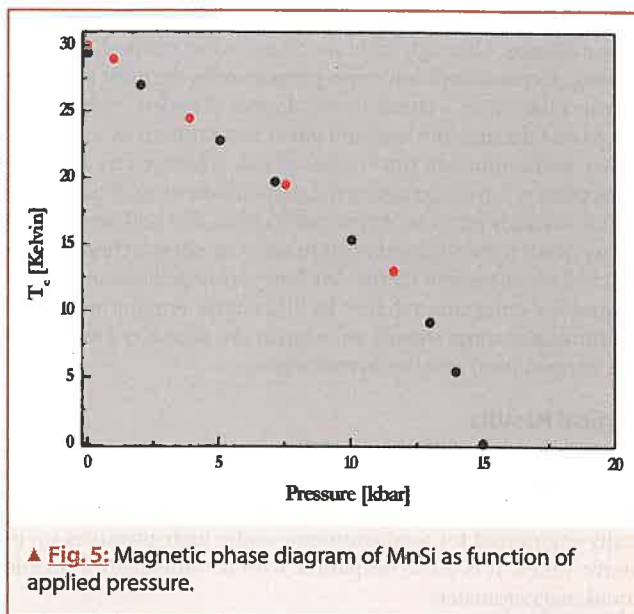
The concept behind these modules is to provide the novice as well as the advanced user with all the required tools to easily set-up, apply and release the pressure of the cell. We have engineered a range tools and accessories to facilitate these various steps. Thus, for example, the setting up time of the easyLab Mcell 10 is typically of around 30 minutes, including sample mounting, pressure cell setting up, pressurisation and mounting on the squid magnetometer. Figure 3 shows a typical pressurisation curve at room temperature for this cell.

As an example of the application and use of this high-pressure cell module we have measured the DC magnetisation of the itinerant ferromagnet MnSi in a squid magnetometer from Quantum

Measurements in collaboration with Dr M. Lees - University of Warwick (U.K.)  
Samples provided by Dr J. Flouquet & Dr G. Lapertot - CENG - Grenoble (France)



◀ Fig. 4: DC magnetisation of MnSi measured at 200 Oe for pressures 0, 0.99, 3.88, 7.56 & 11.63 kbar.



Design, model MPMS-XL5.5. This work was carried out in collaboration with Prof. D. McPaul and Dr M. Lees from the University of Warwick (U.K.). The samples of MnSi were kindly provided to us by Dr J. Flouquet and Dr G. Lapertot from the CEN.G in Grenoble (France).

The graph in figure 4 shows our data. Pressure was regularly increased from ambient pressure to 11.6 kbar. The pressure effect is clearly observed in the data where the magnetic transition temperature is greatly reduced under pressure from 30 K to 13 K.

Based on these results, the magnetic phase diagram as function of pressure of MnSi can be compared with the published results, as shown in figure 5. 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. (1995).

Naturally we have also undertaken the same exercise with the modules of the Pcell range. Thus, for example, figure 6 shows the loading curve of the easyLab Pcell 30.

**Conclusions**

easyLab Ltd is based in Egham, Surrey (UK) at the Enterprise Hub, Royal Holloway, University of London. The prime objective of the company is to provide innovative solutions for materials characterisation. We do this by offering a ‘materials characterisation’ service for industrial and academic clients and by developing, in-house or through collaborations with academics, equipment which extends the current boundaries of measurement into extreme conditions.

In this paper we discussed the first series of products launched by the company: hydrostatic high-pressure modules for cryogenic and high magnetic field use. High-pressure is no longer a specialised tool. It has become vital to fields ranging from geophysics to optoelectronics, from polymers to the production of vaccines. Here we concentrated on the study of new materials, in particular, magnetism and superconductivity. We described some technical aspect of achieving high pressures (up to 3 GPa) and also presented some typical results that can be obtained with our modules, which demonstrate some of their capabilities. Please visit [www.easy-Lab.co.uk](http://www.easy-Lab.co.uk) for further details.

**References**

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