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S. Klotz [a] , G. Hamel [a] & J. Frelat [b]

[a] Physique des Milieux Condensés, UMR 7602, Université P&M Curie, 4 Place Jussieu, 75252, Paris, France

[b] Modelisation en Mécanique, UMR 7607, Université P&M Curie, 4 Place Jussieu, 75252, Paris, France

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A NEW TYPE OF COMPACT LARGE-CAPACITY PRESS FOR NEUTRON AND X-RAY SCATTERING

S. KLOTZ[a,*], G. HAMEL[a] and J. FRELAT[b]

[a]*Physique des Milieux Condensés, UMR 7602, and* [b]*Modelisation en Mécanique, UMR 7607, Université P&M Curie, 4 Place Jussieu, 75252 Paris, France*

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We present a new type of compact hydraulic press of 200 t capacity and 60 kg mass provided with two large openings of 140° (equatorial) *x* 60° (azimuthal) around the sample area. This device has been designed and optimized using finite element calculations, and was built and recently successfully tested. A smaller version with 50 tonnes capacity and 8 kg mass is also available. This 'VX' type Paris–Edinburgh press is expected to have numerous applications in neutron and X-ray scattering whenever large sample volumes (typically 1–100 mm^3) are required, in particular for angle-dispersive powder neutron diffraction on reactor sources, single crystal neutron diffraction, and inelastic neutron and X-ray scattering.

Keywords: High pressure cell; Neutron scattering; Paris–Edinburgh press

There is a surprisingly wide field of applications for compact presses for the study of matter under high pressure in the 10 GPa range using samples of large volume, *i.e.* in the order of 1–100 mm^3. It is easy to show that such pressures require forces in the order of 100 t and hence presses with considerable size and mass. 'Compactness' is however a critical parameter for many applications, in particular for experiments on large-scale facilities such as neutron and synchrotron sources, which explains the efforts made in recent years for the development of small large-capacity presses.

By far the most successful of such devices is the Paris–Edinburgh (PE) press [1]. By careful design it was possible to build a hydraulic press with 250 t capacity and a mass of less than 60 kg to compress samples of 100 mm^3 to 10 GPa, or samples with 35 mm^3 to 25 GPa [2]. A useful figure of merit for 'compactness' is the product $K = V\,P^3/\mathrm{M}$, where V is the sample volume, P is the maximum pressure, and M is the mass of the press. A survey of pressure devices of all types (piston–cylinder, diamond and multi-anvil cells) shows that K is typically 10^2 GPa^3 mm^3/kg, and more or less independent of the technology involved. For the PE press K is up to 10^4 GPa^3 mm^3/kg. The PE cell was originally built for neutron powder diffraction on spallation sources, but soon found applications for inelastic neutron scattering on reactor sources [3], X-ray diffraction under high pressure–high temperature using synchrotron radiation [4], and inelastic X-ray scattering [5].

* Corresponding author. Fax: +33-1-4427-4469; E-mail: stk@pmc.jussieu.fr

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One of the disadvantages of the standard design of the PE cell is its limited angular aperture. The four tie rods restrict the equatorial aperture to $4 \times 70^\circ$, while the azimuthal window is less than $\pm 7^\circ$. This is non-critical for its original application, powder diffraction on the ISIS pulsed neutron source (Chilton, Didcot, UK) since the diffraction angle 2θ of the instruments involved (PEARL/HiPr and POLARIS) is fixed at 90°. For inelastic neutron scattering on triple axis spectrometers, however, the diffraction angle and sample (hence cell-) orientation have to be varied over a wide range of angles. Since both incident and diffracted beams are close to the equatorial plane, the four windows separated by the tie rods are a serious inconvenience. The same applies to angle-dispersive neutron powder scattering on continuous neutron sources, where a continuous coverage of 2θ between 20° and 140° is desirable, which is impossible with the traditional design. Finally, a large angular aperture is essential for single crystal neutron diffraction. Similar considerations apply to X-ray scattering applications of the PE cell [4, 5], though the restrictions in aperture are less critical since there is generally a larger flexibility in the choice of wavelength.

For these reasons we have investigated possibilities of increasing the aperture of the PE cell whilst keeping its essential characteristics (capacity, mass) as they are. To this end, it soon became clear that the design of the PE cell consisting of a ram, tie rods and top plate + breech as the principal elements—which is indeed a rather conventional approach in the design of presses—had to be abandoned. Instead, a cylindrical body was chosen in which the ram is inserted via an artillery or buttress thread (Fig. 1). The ram has cylindrical symmetry and a cross section with dimensions similar to those given by the standard PE ram. The separation of cell body and ram not only has practical advantages but appears to be essential. The most important innovation of the PE press is the particular shape of its cylinder (Patent Nr. 92 401074 7). Under load, radial deformations are limited to less than 0.03 mm at the level of the O-ring seal which prevents leaks and jamming of the piston. Such an 'auto-compensation' can only be achieved if the ram is supported at the lower circumference of the ram, which in the new design is most easily achieved by separating the ram from the body. In the device presented here, the deformations of the ram are much more complex, and are discussed in more detail below. The middle part of the press consists of two 'windows' covering 140° equatorial and 60° azimuthal (±30°) when viewed from the

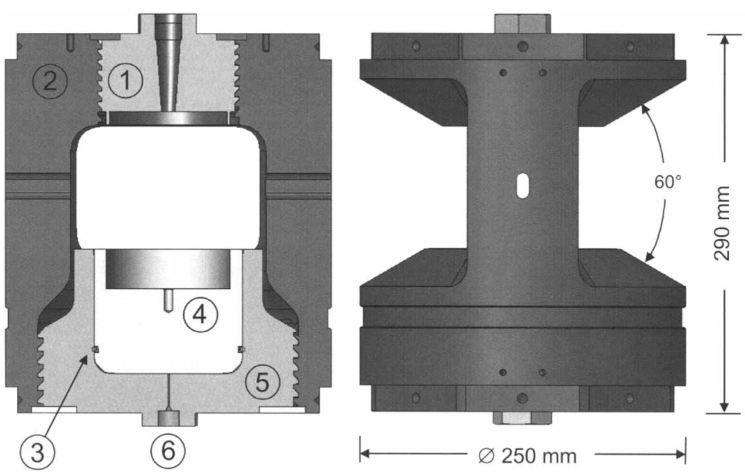

FIGURE 1 Cross section (left) and side view of the VX2 PE press (mass 60 kg, capacity 200 t). 1, breech; 2, body; 3, O-ring seal; 4, piston; 5, cylinder; 6, hydraulic fluid inlet. The azimuthal and equatorial apertures are 60° (as shown) and 140°, respectively.

position of the sample. The breech of 108 mm diameter is situated in the upper part and gives the flexibility to easily load anvil assemblies of various dimensions.

The use of finite element calculations was crucial in designing the cell since previous experience with the standard PE press could not readily be transferred to the new model. Extensive calculations on all parts of the press were carried out using the CAST3M software package [6]. A mesh covering the whole press was refined until convergence was reached. Second order finite elements were used, except at the interface of the artillery threads where linear finite elements had to be chosen. Some realistic approximations were made in order to model the interfaces given by the two artillery threads. These approximations are based on a unilateral contact problem, and the calculations were carried out by a special method which strictly respects the non-interpenetration of the bodies involved. The calculations rapidly revealed two inherent problems in such a ‘two-column’ design. These are the accumulation of stresses at the ‘corners’ of the two windows and the strongly non-isotropic radial deformation of the ram. The former problem can easily be dealt with by keeping the radius of the bend above 15 mm, whereas the latter is less trivial. In fact, under load the radius of the ram decreases by as much as ~ 0.15 mm in the direction between the two columns, whereas it increases by ~ 0.4 mm in direction of the windows. These values correspond to deformations at the upper edge of the ram, where they also can be accurately measured. In the vicinity of the O-ring seal, they become smaller, and below the seal all deformations are slightly positive. Deformations of 0.4 mm are approximately 10 times larger than the tolerance between piston and cylinder and therefore cannot be ignored. The finite element calculations indicated however, that by a judicious positioning of the O-ring, even such strongly non-isotropic deformations can be reduced to values below 0.05 mm. These considerations determined both the shape of the O-ring groove (not shown) and its position.

Figure 2 shows a picture of the 200 t press (VX2) next to a smaller version with 50 t (VX1). Like all standard PE type presses, they are made of high tensile steel 819AW from Aubert and Duval, hardened to 35 HRC (yield strength: 14 GPa, tensile strength: 18 GPa,

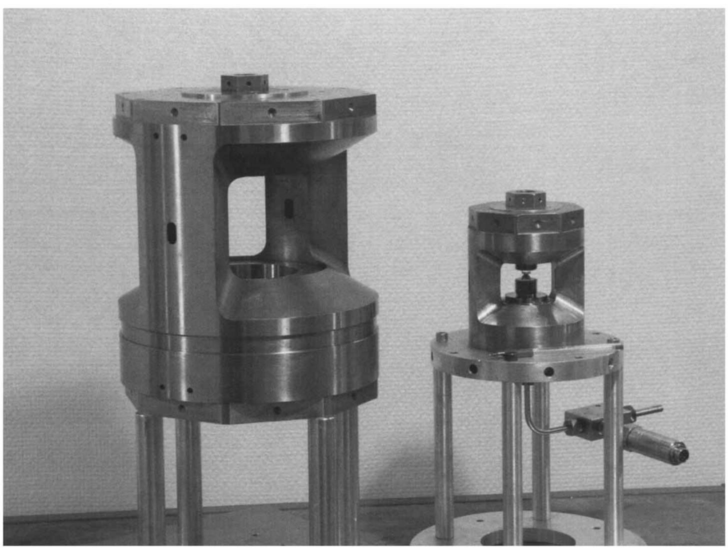

FIGURE 2 Picture of presses VX2 (left, mass 60 kg, capacity 200 t) and VX1 (right, mass 8 kg, capacity 50 t) on their stands. The VX1 is loaded with spherical sapphire anvils; the high pressure valve and the manometer are attached below.

Poisson's ratio: 0.3, Young's modulus: 200 GPa). Both presses were built by MG63 (Vergongheon, France) and tested at ambient temperature to their full capacity. The small version has already been used successfully in several single crystal neutron scattering experiments at the SXD station of the ISIS facility. In the following discussion, they are referred to as 'VX' type PE cells.

The advent of such 'panoramic view' type cells opens new opportunities in neutron and X-ray scattering. The most obvious application is angular-dispersive neutron scattering on reactor sources. Diffraction facilities on continuous neutron sources were previously not able to benefit from the capabilities of the PE cell, due to the limited angular aperture as discussed above. However, recent experiments at the D20 diffractometer of the Institut Laue Langevin (Grenoble, France) [7] and HRPT of the Paul Scherrer Institute (Villigen, Switzerland) [8] showed clearly that even in a scattering geometry where the incident and diffracted beams are in the plane of the gasket, refineable spectra of samples at 10 GPa can be obtained in a reasonable amount of time. Such powder diffractometers are usually equipped with 'banana' detectors spanning more than 100° in 2θ; hence the VX type presses will provide an ideal geometry for such measurements. Note that for this purpose the cell is equipped with holes in the middle of the two columns for the incoming and outgoing neutrons allowing 2θ scans between 20° and 160°. The benefit for high pressure inelastic neutron scattering on triple axis spectrometers is equally evident, since scans in Q space require both the diffraction angle and the sample orientation to be continuously changed. Future single crystal neutron diffraction measurements will also be able to exploit the capabilities of VX type cells, probably using versions with smaller capacities below 50 t similar to the one shown in Figure 2. Compared to other clamped cells with a 'panoramic' view which have been used for neutron scattering [9–11], the crucial advantage of the VX type presses is the possibility to change pressure *in situ* and in a controlled way. This is for example crucial if single crystals of molecular solids have to be grown in the cell. Also, the availability of large gemstones, such as synthetic diamond and Moissanite, for use as anvil material [12, 13] requires presses with large capacities which eventually exceed the capabilities of clamped devices.

There are several obvious extensions of the design shown in Figures 1 and 2. From our previous experience with the PE cell for low temperature measurements [14] it seems clear that a cryogenic version of the VX type cells is feasible. For this purpose, the only modification is the use of a piston with a Bridgman unsupported-area seal allowing the use of helium as the hydraulic fluid. Another evident modification is the replacement of the current ram as shown in Figure 1 by a model, which provides an axial hole, as in the standard V2 and V4 type PE cells. Such types of presses would allow transmission measurements and should be particularly useful for experiments which need optical access using transparent anvils.

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