**DESIGN AND CALIBRATION
OF AN ULTRA-HIGH
PRESSURE X-RAY CELL**

G. L. KULCINSKI

SEPTEMBER, 1967

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PRESSURE X-RAY CELL

By

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ABSTRACT

A device to generate pressures up to 50 kbars ($\sim 750,000$ psi) has been designed to function in the environment of a nuclear reactor. Its overall cylindrical dimensions are 2.4 cm diam and 3.3 cm long. It has been adapted to yield optical and X-ray information, before and after irradiation, on a sample under pressure. Calibration of this device, by noting the X-ray lattice parameter shift of NaCl and the optical absorption shift of TlBr, is described. It has been shown that the pressure on a powdered sample surrounded by a metallic washer is considerably lower than that predicted by a simple force/area ratio. It is also shown that the pressure gradients on a powdered sample contained between diamond anvils are reduced by surrounding it with a metallic washer.

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INTRODUCTION

This report describes the design and operation of a self-contained high pressure cell that can exert pressures to 50 kbars and higher on a sample under neutron irradiation. With this cell, the effects of neutron irradiation on phase transformations can be studied by varying the applied pressure.

In the past, effects of neutron irradiation on the physical properties of solids has been studied by varying such parameters as temperature, flux rate, total fluence, energy of the neutrons, composition of the solid and its previous mechanical history. When applied to nonirradiated solids, a parameter which has been shown to be equally, if not more, effective as those previously mentioned is pressure. The combined effects of ultra-high pressures (>10 kbars)* and irradiation have not been studied in the past because of the problems encountered in equipment design.

Normally, the experimental space available for irradiation studies is a 5 to 7 cm diam tube. A review of the literature reveals that the most popular and functional devices in high pressure research (i.e., cubic,⁽¹⁾ tetrahedral,⁽²⁾ and "belt"⁽³⁾ configurations) are too heavy and require too much space to be placed inside of a reactor. Bridgman anvil⁽⁴⁾ and piston-cylinder⁽⁵⁾ devices conform more readily to the required geometry. However, due to the unusually large separation between the actual pressure cell and the pressure generating equipment, considerable lengths of high pressure tubing are required. This

* 1 kbar = ~1000 atm

is undesirable not only from a safety standpoint, but it also requires that the hydraulic fluids used to transmit the pressure to the cell be subjected to considerable doses of ionizing radiation. Since hydraulic fluids are organic in nature and because organic substances are subject to severe degradation under irradiation, a question of reliability is raised. Indeed it has been found that under both pressure and irradiation, polymerization is considerably accelerated.^(6, 7, 8)

In order to solve the above problem, several self-contained high pressure cells are being designed and constructed. One of these is a Bridgman pressure cell that can exert pressures to 50 kbars and higher on a sample while requiring no external tubing or control leads. Furthermore, it is possible with this cell to obtain "in-situ" optical and X-ray analysis of the sample before and after irradiation. The design and operation of this cell is described below.

DESIGN OF CELL

Since pressure is merely an applied force divided by the area on which that force acts, one can obtain large pressures by applying a small force on an even smaller area. Figure 1 is a schematic of a diamond high pressure cell utilizing this principle, and Figure 2 is a photograph of a working model. It consists of two disc-spring-loaded, gem-cut diamonds which act simultaneously as pressure anvils, optical windows, and X-ray transparent windows. The culet faces of the diamonds are purposely ground to different diameters in order to minimize alignment problems. The alignment of the diamonds is accomplished by the set screws in the base of the cell and the faces are usually parallel to plus or minus two minutes of one degree. In some respects this cell incorporates features from devices designed by Lawson and Tang,⁽⁹⁾ Jamieson,⁽¹⁰⁾ Lippencott et al.,⁽¹¹⁾ and a previous cell developed by Kulcinski et al.⁽¹²⁾ The unique feature of this cell is its size and its capability to yield X-ray information on solids under pressure before and after irradiation.

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FIGURE 1. Schematic of Diamond High Pressure X-Ray Cell

The pressure is applied in the following manner. The sample, foil or powder, is placed on one of the diamonds. An external press acts through the spring retainer to compress the disc springs, which in turn apply a compressive force to the upper diamond. The diamonds are held in their individual seats with plaster of paris. While the required pressure is maintained, the sleeve is screwed down until it engages the retainer. The external load is then released, leaving the threads to support the compressed disc springs. Since irradiation space is at a premium in most reactors, it is desirable to make the diameter of the cell as small as possible, while still able to deliver the required pressure. An important advantage of disc springs over conventional coil springs is their ability to deliver rather large loads

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FIGURE 2. Exploded and Assembled Views of Diamond High Pressure Cell

(~100 to 150 kg) for very small compressions (~0.04 cm). This would correspond to a 2500 to 3500 kg/cm spring constant for coil springs. Another important advantage of disc springs is that one can increase the load applied at the same deflection by placing the washers in series (i.e., on top of each other) or increase the deflection at constant load by stacking them in parallel (i.e., top to bottom, as illustrated in Figure 1). Hence one can design a spring arrangement for almost every conceivable load-deflection requirement. No such possibility exists with conventional coil springs. Besides allowing the cell to be essentially self contained, the springs have a relatively low potential energy storage. This means that in the event of failure, little damage will be done to the surrounding reactor components. Contrast this behavior with the failure of a gas or liquid system pressurized to 500 to 1000 psi.

The minimum diameter of disc spring which can deliver 100 to 150 kg at an acceptable stress in the spring is 2 cm. Adding appropriate clearance and wall thickness for the sleeve, the minimum diameter for the cell in Figure 1 is 2.4 cm. In the loaded condition the cells are 3.3 cm long.

The entire cell and disc springs are made of a Ti-6Al-4V alloy. This material has a very small absorption cross section for neutrons ($\sigma_a = 5.6$), reasonable hardness ($R_C 34$), and possesses satisfactory yield strength (~150,000 psi). The importance of the last two properties arises from mechanical design, and the first is important for the X-ray analysis.

X-ray film is quite sensitive to the photons emitted from a material undergoing radioactive decay. If the intensity of photons from the irradiated pressure cell is greater than the diffracted intensity of X-rays from the sample, the film will be fogged, making X-ray analysis very difficult, if not impossible. Hence it is required that the cell be made of materials which have low absorption cross sections or very short lived isotopes.

The major isotopes in the titanium alloy which emit high energy photons are ^{28}Al , ^{51}Ti , ^{52}V , all of which have half lives of less than 6 min. However, due to minute impurities in the alloy, it is necessary to wait a few days after removal before the radioactivity is reduced to the point where the cell can be handled. Very little activity can be expected from the plaster of paris or the diamonds unless they contain considerable impurities.

A disadvantage of this design is the extremely small sample size that must be used. To generate 50 kbars with 100 to 150 kg of force, the maximum area of the sample is $2 \times 10^{-3} \text{ cm}^2$. Figure 3 shows a magnified view of the maximum size sample that can be used. This particular sample was formed by subjecting ZrO_2 powder to 30 kbars. The octagon shape corresponds to the face of the small diamond and the edge view shows that an equilibrium thickness of 10^{-3} cm is attained at those pressures.

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FIGURE 3. ZrO_2 Sample After Compression to 30 kbars. The sample in the center is mounted on 0.3 mm glass fiber. Edgewise view (right) of sample that is 0.01 mm thick and compared to pinpoint (left).

The cell can be heated to 150 to 200 °C by inputs of 35 to 60 W to an electrical resistance heater wound into one of the pistons. However, in most irradiation runs the heater is not used.

The typical cell weighs 50 g, of which there are 45 g of titanium, 3 g of aluminum, and 2 g of vanadium. The diamonds weigh 0.06 g, the plaster of paris 0.05 g, and the samples usually less than 0.001 g.

It has been found that powdered samples similar to those in Figure 3, while suitable for optical measurements, are unsatisfactory for in-cell X-ray analysis work. This is because of very large pressure gradients characteristic of this type of cell^(10, 11) and the small amount of material available in a sample extruded to equilibrium thickness. The first effect results in very broad X-ray lines, and the latter produces a very spotty Deby-Scherrer pattern. In order to compensate for this, a washer of relatively incompressible material is placed around the sample to prevent extrusion and thus maintain sample thickness (Figure 4).

The washer is usually made out of 0.018 cm thick molybdenum foil. Molybdenum is used because of its ability to flow under pressure and because of its short activation half life. The foil is placed between the diamonds and the pressure is raised to 20 to 30 kbars. The pressure is released and the foil removed. A 0.02 to 0.03 cm diam hole is then drilled into the indentation made by the small diamond (Figure 4). The diamonds are ~0.05 cm in diameter.

The material to be studied is placed in the hole in the foil by forming a pellet of the powder around the washer and then splitting the washer out of the pellet. Usually the pellet is formed by pressures less than 4 to 5 kbars. Any extra material clinging to the metal is removed and the washer is replaced on the small diamond exactly in the position from which it was removed. The cell is reassembled and is ready for X-ray analysis.

Schematic of Washer for Diamond High Pressure X-Ray Cell

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FIGURE 4. Schematic of Washer for Diamond High Pressure Cell

As can be seen in Figure 1, the base of the cell is designed so that diffracted X-rays up to 20 to 30° can be analyzed. In most studies, zirconium filtered molybdenum radiation is used to obtain a maximum of diffraction lines within the limited 2θ range.

After the sample is loaded and compressed, the cell is placed in a specially designed X-ray camera, which is shown in Figure 5. The camera is conventional except for the fact that the sample-to-film

distance can be varied by the thickness of spacers used. The camera is also designed so that the film may be removed and replaced without disrupting the sample position, and the sample may be removed and inserted without disrupting the film position.

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FIGURE 5. Schematic of X-Ray Camera for Diamond High Pressure Cell

The complete transparency of Type I diamonds in the visible region allows both transparent and reflected analysis of the materials under pressure between the diamond faces. Examples of this technique will be shown later in this report.

CALIBRATION

GENERAL CONSIDERATIONS

In the simplest case when using Bridgman anvils without washers, one could estimate the pressure on a sample by dividing the force applied by the area on which it is acting. This assumes that the pressure is uniformly distributed over the sample. However, it has been shown in the past⁽¹²⁻¹⁷⁾ that this method of estimating the pressure does not take into consideration the pressure gradients resulting from friction forces, both in the sample and in the equipment. As a result, the maximum pressure in the sample may be as much as two times the average pressure as determined by the force/area (F/A) ratio. Specifically, it has been shown that the pressure varies from the center of the sample to a higher^(15, 18, 19) or lower^(12, 13, 17, 20, 21) value near the edge, depending on whether deformable (metallic) or nondeformable (diamond) anvils are used respectively. This behavior is shown in Figure 6.

The introduction of a metallic washer around the sample tends to reduce the pressure gradients. It has been shown by Myers et al.⁽¹⁵⁾ that for sample diameter-to-thickness ratios of 15 or more, the pressure gradients are reduced to less than 10%. Jackson⁽²¹⁾ has treated this problem theoretically and has made the following predictions: 1) the pressure gradient will be reduced, and 2) the actual pressure experienced by the sample will depend on the ratio of its yield strength to that of the washer material.* While the mathematical analysis of this situation is very difficult, it appears that if the ratio of yield strength is greater than one, the pressure on the sample will be greater than the average applied pressure (as determined by F/A). On the other hand, if the sample has a lower yield strength than the washer (a most likely situation when metal washers are used), the pressure on the sample will be less than the F/A ratio.

An experimental example of the strong-sample-weak-washer combination is given by McWhan.⁽²²⁾ He has calibrated his apparatus

* This ratio will hereafter be referred to as k .

A) Deformable Metallic Anvils
After Christensen⁽¹⁴⁾

B) Nondeformable Diamond Anvils
After Kulcinski⁽¹²⁾

FIGURE 6. Examples of Pressure Distributions on Samples
Contained Between Bridgman Anvils

by X-ray analysis of a sample surrounded by boron epoxy discs. In this case the average pressure on the sample is 1.5 to 2 times that determined by the F/A ratio.

There are no known calibration data on the reverse situation, although several groups are known to be using high-yield-strength metals as washers when studying low-yield-strength compounds and liquids under pressure.⁽²³⁻²⁵⁾ Since most of the work with the cell described in this paper will deal with the latter case, it was necessary to provide such data.

THEORY

Let us initially consider the case of a plastic material B surrounded by an elastic material A (Figure 7). Later we will allow A to become plastic and examine the way this effects the pressure to the sample. The axial stress on the sample (B) within the region $0 < r < c$ is given as⁽²¹⁾

$$\sigma_z(B) = [\sigma_{z(r=c)}(B)] \exp \left[2 \frac{f(c-r)}{h} \right] \quad (1)$$

where:

$\sigma_{z(r=c)}(B)$ is the axial stress on an increment of sample at $r = c$,

f is the coefficient of friction between the sample and the anvil,

h is the thickness of the washer.

Since the radial stress, σ_r , must be continuous between region A and B and from the yield criterion at $r = c$ one finds:

$$\sigma_{r=c}(A) = \sigma_{r=c}(B) = [\sigma_{z(r=c)}(B) - \sigma_0(B)] \quad (2)$$

where σ_0 = yield stress of sample.

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FIGURE 7. Geometrical Model for Theoretical Calculation of Pressure on a Sample (B) Surrounded by a Washer (a) Between Bridgman Anvils

By substituting the boundary condition of Equation (2) into the general equations for the radial and tangential stresses in the elastic region, $c < r < a$, one can solve for $\sigma_{Z(r=c)}^{(B)}$ in terms of the elastic stresses in (A). Such a substitution yields⁽²¹⁾

$$\sigma_r(A) = \frac{2f}{3h} \sigma_Z(A) \left[-r + \frac{c^3 - a^3}{c^2 - a^2} + \frac{a^2 c^2}{c^2 - a^2} \frac{(a - c)}{r^2} \right] + \left[\sigma_{Z(r=c)}^{(B)} - \sigma_o(B) \right] \cdot \left[\frac{c^2}{c^2 - a^2} - \frac{a^2 c^2}{(c^2 - a^2)r^2} \right] \quad (3)$$

and

$$\sigma_\theta(A) = \frac{2f}{3h} \sigma_Z(A) \left[r + \frac{c^3 - a^3}{c^2 - a^2} - \frac{a^2 c^2}{c^2 - a^2} \frac{(a - c)}{r^2} \right] + \left[\sigma_{Z(r=c)}^{(B)} - \sigma_o(B) \right] \cdot \left[\frac{c^2}{c^2 - a^2} + \frac{a^2 c^2}{(c^2 - a^2)r^2} \right] \quad (4)$$

Using the octahedral stress theory which states:

$$\sigma_o^2 = \sigma_1^2 + \sigma_2^2 + \sigma_3^2 - \sigma_1\sigma_2 - \sigma_2\sigma_3 - \sigma_3\sigma_1 \quad (5)$$

where $\sigma_1 = \sigma_r$

$\sigma_2 = \sigma_\theta$

$\sigma_3 = \sigma_Z$

σ_o = Yield in tension or compression,

and inserting the following values of σ_Z , σ_r , and σ_θ at $r = a$ (where yielding first takes the place in A)

$$\sigma_r(A) = \sigma_1 = 0 \quad (6)$$

$$\begin{aligned} \sigma_\theta(A) = \sigma_2 = \frac{2f}{3h} \sigma_o(A) & \left[a + \frac{c^3 - a^3}{c^2 - a^2} - \frac{c^2(a - c)}{c^2 - a^2} \right] \\ & + \left[\sigma_{Z(r=c)}(B) - \sigma_o(B) \right] \cdot \frac{2c^2}{c^2 - a^2} \end{aligned} \quad (7)$$

$$\sigma_Z = \sigma_3 \quad (8)$$

one finds that (see Appendix A)

$$\frac{\sigma_Z(B)_{ave}}{\sigma_T} = \left[\frac{1}{VU} (1 - t^2) + t^2 \right]^{-1} \quad (9)$$

where $\sigma_Z(B)_{ave}$ is the average axial stress in B

σ_T is the theoretical stress on the washer and sample = $\frac{\text{Load}}{\pi a^2}$

$$t = \frac{c}{a}$$

V is the factor which relates $\sigma_{Z(B)}^{ave}$ to $\sigma_{Z(r=c)(B)}$ and equals

$$\frac{1}{2} \left(\frac{h}{fc} \right) \cdot \left[\exp \left(\frac{2fc}{h} \right) - 1 \right]$$

U is the factor relating the $\sigma_{Z(r=c)(B)}$ to the yield stress of region A and equals

$$\frac{1}{K} + \frac{(1 - 2X\gamma_1) \pm [(1 + 2X\gamma_1)^2 + 4X\gamma_1(1 - X\gamma_1)]^{1/2}}{2A}$$

where $X = \frac{2tf}{3h}$

$$\gamma_1 = a \left(1 + \frac{2t^3 - t^2 - 1}{t^2 - 1} \right)$$

$$A = \frac{2t^2}{t^2 - 1}$$

For typical values of $f/h = 10$; $\sigma_Z(A) = \sigma_o$; $k = 8$; and $c/a = 0.5$ one finds that $\sigma_{Zave(B)}/\sigma_T \cong 0.50$. This says that a sample with a yield strength $1/8$ that of its containing washer will experience $\sim 1/2$ the theoretical pressure applied.

The situation in Equation 9 can be extended to a more realistic case under high pressures, namely, by allowing the washer material A to become plastic (see Appendix B). The ratio of actual to theoretical pressure then becomes;

$$\frac{\sigma_{Zave(B)}}{\sigma_T} = \left[\frac{Z}{VU} (1 - t^2) + t^2 \right]^{-1} \quad (10)$$

where Z relates $\sigma_{Zave(A)}$ to the yield strength of region A and is a function of a, c, h, and f.

$$Z = \frac{h}{2f} \frac{1}{a - c} \left[\exp \frac{2f}{h} (a - c) - 1 \right] \quad (11)$$

Substituting the same values used for the previous example one finds $\sigma_{Z \text{ ave}}(B)/\sigma_T \cong 0.45$, i.e., the pressure to the sample will be slightly less than 1/2 that determined theoretically.

While the above values are rather sensitive to f/h and k and are not to be taken as precise values, both indicate that a significant pressure reduction can occur when a weaker material is placed into a stronger washer, and both are compressed to high pressures.

EXPERIMENTAL TECHNIQUES

In order to test the validity of Equations (9) and (10), two methods of calibration were used:

1. The change in the lattice spacing of NaCl with applied load, and
2. The shift in the optical absorption edge of TlBr with applied load.

Both methods yield information about the absolute pressure experienced by a sample contained by a washer, and the second method reveals the magnitude of pressure gradients that may exist in the sample. Both of these methods yield continuous calibration data, and neither suffers from hysteresis effects as do most phase transitions.

The shift in the (200) spacing of NaCl was obtained from X-rays taken of the sample under various loads in the Diamond Cell. These changes were then compared to the equation of state for NaCl as derived by Decker.⁽²⁶⁾ Figure 8 gives the volume change and shift in the (200) line spacing at 25 °C as a function of pressure. The specimen-to-film distance was calibrated by X-rays taken of NaCl at 1 bar and assuming $d_{(200)} = 2.821 \text{ \AA}$. Exposure time depended on the amount of NaCl used (size of hole in washer) and typical values varied from 4 to 16 hours with Kodak no-screen film.

FIGURE 8. Volume Change and Lattice Parameter Change of NaCl as a Function of Pressure⁽²⁶⁾

The material used for this study was Mallinckrodt reagent-grade NaCl. Maximum stated impurities were less than 100 ppm. The NaCl was ground to a powder and only that material which passed through a 325 mesh screen was used. The powder was dried at $\sim 200^\circ\text{C}$ in a vacuum for a day and stored in a sealed container before use. It was placed into molybdenum washers in the manner described earlier. The sample was cycled several times to maximum pressure before taking data. This cycling allows all the "voids" to be filled and insures that the sample will attain good contact with its surrounding washer.

Thallium bromide is known to have an optical absorption edge that progresses to the red wave lengths as pressure increases⁽²⁷⁾ (see Figure 9). Thus, by noting the wave length of light at which the normally transparent TlBr becomes opaque, the pressure on the sample can be determined. In addition, by noting the wave length at which absorption begins in any part of the solid and the wave length at which the whole solid is opaque, the amount of pressure gradient experienced by a sample can be estimated.

FIGURE 9. Optical Absorption Edge of TlBr as a Function of Pressure⁽²⁷⁾

TlBr obtained from Electronic Space Products was dried in a vacuum and loaded into the gasket in the normal way. Maximum impurity levels were less than 10 ppm and the powder size used was that which passed through a 325 mesh screen.

The optical absorption edge was measured by use of a Zeiss Ultraphot II microscope. A gradation filter was used to vary the wavelength of light passing through the sample. The absorption edge was noted visually at 100X magnification. The sample was cycled to maximum pressure several times before calibration data was recorded.

The theoretical pressure (F/A ratio) was calculated from load deflection curves for the disc springs and the area of the smallest face diamond. The diamond face areas varied from 2.00 to $2.46 \pm 0.01 \times 10^{-3} \text{ cm}^2$ and were determined on a Kodak Contour Projector at 100X. The load deflection curves for the compression of the disc springs were determined at various strain rates and temperatures on a type TT-CL Instron machine. It was found that increasing the strain rate from 0.025 to 0.25 cm/min had no effect on the curves.

Figure 10a shows the load-deflection curves for three different sets of Ti-Al-V springs and two sets of steel springs as arranged in Figure 11. The differences in the slope of the titanium spring set #1 and that of sets #2 and 3 stem from the fact that these washer sets were machined at different times. Since the load at any deflection is proportional to the third power of the washer thickness (0.107 cm in Figure 11) normal machining tolerance can have a noticeable effect on the springs load characteristics. The variation between the titanium washers and steel set #1 is a result of the difference in elastic modulus, 2.07×10^6 bar for steel and 1.14×10^6 bar for Ti-6Al-4V. It will be noticed that the slope of the load-deflection curve for steel set #2 is almost four times that of steel set #1. The factor of two increase in load is due to stacking two washers in series, and the reduction in maximum total deflection is due to having only four stacks in parallel as opposed to seven in steel set #1.

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FIGURE 10. (a) Load Deflection Curves for Disc Spring Arrangements in Figure 11; (b) Theoretical Pressure Deflection Curves for Disc Spring Arrangement in Figure 11 and Designated Set of Diamond Anvils

(a) Stacking Sequence for
Ti-Al-V and Steel Disc
Springs Set #1

(b) Stacking Sequence for
Steel Disc Springs
Set #2

(c) Dimensions

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FIGURE 11. Various Disc Spring Arrangements for Diamond High Pressure Cell

It should be emphasized that the curves in Figure 10 are for the spring arrangements and dimensions in Figure 11, and one could get larger deflection with the same maximum load or larger load at the same deflection simply by placing the springs in series or parallel, or by altering the dimensions.

It was found necessary to cycle the spring arrangement at least seven to ten times before reproducible (less than 2% variation) curves

could be obtained. Relaxation of the springs in a clamped position was found to be less than 5% over a period of a week. Cycling helps to reduce relaxation to the point where it is no longer significant.

Figure 10b combines the load-deflection curves in Figure 10a with the areas of the anvil diamonds to yield typical theoretical calibration curves for various combinations of springs and diamond sets. Again, these curves are characteristic only of the arrangement in Figure 11, and virtually any slope could be obtained by proper design of the disc springs.

The effect of temperature on the load characteristics of the titanium springs was investigated and is shown in Figure 12. A 280 °C increase in temperature increases the load at 2.54 mm deflection by 40% to 112 kg. The temperature coefficient, while not truly linear, could be approximated by 0.09%/°C below 200 °C.

FIGURE 12. Effect of Temperature on the Load-Deflection Characteristics of Ti-6Al-4V Disc Springs as Arranged in Figure 11a

The washer material used in all cases around the samples was 0.018 cm thick molybdenum foil prepared in the manner described earlier. Hole sizes varying from 0.0158 to 0.0428 cm were drilled into the diamond impression with a high speed micro-drill press. The diameters of the holes were measured with a micrometer eyepiece on the Zeiss Ultraphot Microscope.

RESULTS AND DISCUSSION

Table I shows the results obtained from X-ray analysis, and Table II lists results from TlBr absorption edge studies. In the case of NaCl, c/a^* ratios varied from ~ 0.35 to ~ 0.83 and for TlBr from 0.31 to 0.88. Theoretical pressures ranged from 10 to 106 kbars by simply rearranging the disc springs. The accuracy of both methods of calibration is assumed to be ± 1 kbar below 20 kbars and ± 2 kbars > 20 kbars.

There are two general trends revealed by both tables:

- There is a significant departure from theoretically predicted pressures at the higher loads, approaching 0.2 to 0.5 σ_T .
- There is a definite relationship between the size of the hole in the washer and the fraction of theoretical pressure on the sample. Specifically, the larger value t has, the lower the pressure to the sample.

These two trends are amply illustrated by plotting all of the data on a load-versus-pressure curve such as in Figure 13. Figure 14 shows the same data normalized to the theoretical pressure, and again the departure is quite evident.

Photographs of the optical absorption in TlBr with and without surrounding washers are shown in Figure 15. It is obvious that the addition of a metallic washer around the sample considerably reduces the pressure gradients. Without washers, pressure gradients of as much as 40 to 50 kbars are found, whereas with washers, the gradients are reduced to less than 10 to 15 kbars.

* c/a is the ratio of the hole diameter to the diameter of washer.

TABLE I. Summary of NaCl X-Ray Data Used For Calibration
of Diamond X-Ray Cell-21 °C

Load, kg	c/a ^(a)	Lattice Parameter, Å ± 0.004	Pressure, kbars $\sigma_{Z \text{ ave}}^{(B)}$	Theoretical Pressure, kbars σ_T	$\sigma_{Z \text{ ave}}^{(B)}$ σ_T
23	0.35	2.789	19	10	0.90
	0.535	2.804	5	10	0.50
	0.682	2.799	6	10	0.60
43	0.530	2.778	12	20	0.60
	0.682	2.789	9	20	0.45
68	0.369	2.742	23	30	0.77
	0.535	2.768	15	30	0.50
	0.775	2.778	12	30	0.40
86	0.403	2.738	25	37	0.68
	0.825	2.772	73	37	0.35
93	0.369	2.736	26	40	0.65
	0.535	2.760	17	40	0.42
	0.775	2.763	16	40	0.40
129	0.403	2.715	33	56	0.59
	0.470	2.755	19	56	0.34
163	0.403	2.711	35	73	0.48
173	0.403	2.714	34	75	0.45
	0.470	2.730	28	75	0.37
186	0.403	2.700	42	80	0.53
250	0.403	2.696	44	111	0.39

(a) See Figure 7 for definition.

TABLE II. Summary of TlBr Optical Absorption Data
Used For Calibration of Diamond X-Ray Cell at 21 °C

<u>Load, kg</u>	<u>c/a^(a)</u>	<u>Absorption Edge, Å</u>	<u>Pressure, kbars $\sigma_{Z \text{ ave}}^{(B)}$</u>	<u>Theoretical Pressure, kbars</u>	<u>$\frac{\sigma_{Z \text{ ave}}^{(B)}}{c_T}$</u>
23	0.311	4410	9	10	0.90
	0.490	4360	8	10	0.80
43	0.350	4560	15	18	0.83
	0.490	4460	11	18	0.61
	0.875	4350	7	18	0.39
45	0.490	4490	12	20	0.60
	0.875	4380	8	20	0.40
68	0.490	4510	13	30	0.43
	0.875	4475	11	30	0.37
86	0.350	4870	27	37	0.73
	0.490	4695	20	37	0.54
	0.875	4470	11	37	0.27
93	0.490	4565	15	40	0.37
	0.875	4585	16	40	0.40
129	0.35	5090	35	56	0.62
	0.490	4820	25	56	0.45
163	0.527	4770	23	73	0.31
	0.75	4600	16	73	0.22
173	0.400	5190	39	75	0.52

(a) See Figure 7 for definition.

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FIGURE 13. Experimental Calibration Curve for Diamond High Pressure Cell

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FIGURE 14. Pressure Reduction Factor for Diamond High Pressure Cell

Neg 0671111-2

FIGURE 14. Pressure Reduction Factor for Diamond High Pressure Cell

Neg 0671107-1

FIGURE 15. Effect of Washers on the Pressure Gradients in Samples Between Diamond Anvils as Determined by the Optical Absorption Edge of TlBr. Dark area represents part of sample above stated pressure.

The major result of these tests is the demonstration that the pressure on a powdered sample surrounded by a metal washer, which is compressed between diamond anvils, is considerably lower than that predicted by the theoretical F/A ratio. This is in direct contrast to the situation encountered when surrounding the sample with a soft washer material⁽²²⁾ or with no washer at all^(12,13,17, 20,21) (Figure 15). The factor of 2 or 3 reduction is far in excess of any experimental error, and the fact that the same results were obtained with two completely independent methods of measurement tends to confirm the hypothesis.

Not only is there a reduction in pressure but the amount of reduction depends on the size of the hole in the washer. It was shown, again with both X-ray and optical analysis, that as the hole diameter increases, so does the deviation from the theoretical pressure.

This situation is exactly what is predicted by Equation 10, as is seen in Figure 16. In this figure the ordinate is the experimentally measured average pressure and the abscissa is the pressure $\sigma_{Z \text{ ave}}^{(B)}$ calculated from Equation 10. The values of t , σ_T , are used from Tables I and II, f/h is assumed to be 10, the ratio of the yield strength of molybdenum to NaCl (or TlBr) is taken as 8 and $a = 0.010$ in., $h = 0.005$ in. These latter two values have been experimentally determined. Bridgman⁽²⁸⁾ finds that the shear strength of molybdenum at 50 kbars is ~ 12 kbars and that of NaCl at 20 kbars is 1.5 kbars, so that at high pressures $k = 8$ is a reasonable value. Considering the fixed values of " a " and " h ", the assumption of $f/h = 10$ may be somewhat low because $f \cong 0.05$. However, this f refers to the coefficient of friction between the plastic NaCl or molybdenum and the diamond. This is a factor of two lower than the reported coefficient of friction between a smooth diamond surface and metal at 1 atm.⁽²⁹⁾ Considering the assumptions made in the derivation of Equations 9 and 10, it is thought that the use of this order of magnitude of the friction coefficient is justified.

Neg 0671111-4

FIGURE 16. Comparison of the Pressure on a Washer-Encased Sample as Measured Experimental (ordinate) and calculated from Equation 10 (abscissa)

Figure 16 should be viewed only in a qualitative manner. Because there are too many variables (h, c, f, k) and some simplifying assumptions in the theory of plasticity, it is difficult to obtain more precise information.

For example, it was noticed in some tests that at the higher pressures the c/a ratio increased from its initial value by some 10 to 20%. Certainly the k ratio must be changed, probably to lower values at higher pressures, and the coefficient of friction will probably increase. Time did not permit a detailed study of all of these parameters.

The apparent qualitative agreement between experimental data and theory has great significance even if exact quantitative predictions cannot be made. It not only confirms the pressure loss that most workers in the field have noticed in the past, but it gives a semi-quantitative way of predicting the magnitude of this loss. For sample-washer combinations with values of $k > 8$, i.e., solidified liquids and very weak solids, theory predicts that the pressure loss will be significantly more than with materials such as NaCl and TlBr. On the other hand, if combinations with values of $k < 8$ are used, i.e., metallic powders, the pressure loss should be less than ~50 to 80% found in this experiment. Furthermore, theory and experiment predict that the larger the hole in the washer, the more the actual pressure to the sample will deviate from the theoretical pressure. One can also state that the higher the theoretical pressure, the more the sample pressure will deviate from it.

With these general guidelines and given c/a , f/h , and σ_T it is thought that one could predict the sample pressure to within 20% by using Equation 10.

While this does not preclude the necessity for reporting theoretical pressures, it can cut the uncertainty by a factor of 10 or more at extremely high pressures (>50 kbars).

The reduction of pressure gradients within the sample when it is surrounded by a washer was evident in both X-ray and optical calibration measurements. In the X-ray analysis the reduced gradients resulted in sharper Debye-Scherrer lines. Considering the case of TlBr, the maximum pressure gradients were 10 to 15% (Figure 15). This reduction should make quoted pressure values much more meaningful than those associated with large pressure gradients. The homogeneity of the pressure

will also tend to reduce the shearing effects on phase transitions, as experienced in a previous study with diamond cells.⁽³⁰⁾ Since our diameter-to-height ratios are ~ 20 , the results of this experiment agree with those of Myers et al.,⁽¹⁵⁾ who found a 5% gradient for d/h ratios of 15 or more with metallic anvils.

It should be mentioned at this point that the change in the NaCl lattice parameter was entirely continuous up to 44 kbars at 22 °C with no evidence of any phase transitions. Pistorius,⁽³¹⁾ Lawson,⁽³²⁾ and Vereschagin⁽³³⁾ claim to have observed a phase transition at 17 kbars, the latter group by high pressure X-ray analysis. However, Jameson,⁽³⁴⁾ Corll,⁽³⁵⁾ Johnson,⁽³⁶⁾ and Jeffery et al.⁽³⁷⁾ have tried unsuccessfully to duplicate their results. Our data supports this latter group of investigators.

CONCLUSIONS

A high pressure cell capable of generating 50 kbars or more has been designed to yield X-ray and optical information about samples under extremely high pressures. It has been adapted to function in the environment of a nuclear reactor. Calibration of the cell by X-ray analysis of NaCl and by using the pressure-sensitive optical absorption edge of TlBr has demonstrated that the pressure deviates by a factor of 2 or more from those predicted by a simple F/A ratio. The proposed fcc $\rightarrow$ bcc phase transition of NaCl at 17 kbars was not observed, which is in accordance with the more recent findings of high pressure scientists.

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APPENDIX A

USE OF THE OCTAHEDRAL STRESS THEORY TO SOLVE FOR $\sigma_z(B)$
IN TERMS OF $\sigma_o(A)$ and $\sigma_z(A)$

Starting with Equation 3 in the text and making the following substitutions in Equations 6 - 8,

$$X = \frac{2f}{3h} \quad (A-1)$$

$$\sigma_z(A) = \sigma_o(A)$$

$$\sigma_o(A) = k\sigma_o(B) = \sigma_o \quad (A-2)$$

$$\gamma = \left[a + \frac{c^3 - a^3}{c^2 - a^2} - \frac{c^2(a - c)}{c^2 - a^2} \right] \quad (A-3)$$

$$\lambda = [\sigma_{z(r=c)}(B) - \sigma_o(B)] \quad (A-4)$$

$$A = \frac{2c^2}{c^2 - a^2} \quad (A-5)$$

one finds that

$$\begin{aligned} \sigma_o^2 &= \sigma_2^2 + \sigma_3^2 - \sigma_2\sigma_3 \\ &= (X\sigma_o\gamma + A\lambda)^2 + \sigma_o^2 - X\sigma_o^2\gamma - \sigma_o A\lambda, \end{aligned} \quad (A-6)$$

expanding

$$\begin{aligned} \sigma_o^2 &= X^2\sigma_o^2\gamma^2 + 2XA\sigma_o\lambda + A^2\lambda^2 + \sigma_o^2 \\ &\quad - X\sigma_o^2\gamma - \sigma_o A\lambda, \end{aligned} \quad (A-7)$$

rearranging

$$A^2\lambda^2 + \lambda(2XA\sigma_o\gamma - \sigma_o A) + X^2\sigma_o^2\gamma^2 - X\sigma_o^2\gamma = 0 \quad , \quad (A-8)$$

solving for λ

$$\lambda = \frac{+A\sigma_o(1 - 2X\gamma) \pm \sqrt{A^2\sigma_o^2(1 - 2X\gamma)^2 + 4A^2X\sigma_o^2\gamma(1 - X\gamma)}}{2A^2} \quad , \quad (A-9)$$

substituting Equation A-4 into A-9 and dividing Equation A-9 by σ_o ,

$$\frac{\sigma_{z(r=c)}^{(B)}}{\sigma_o} = \frac{1}{k} + \frac{(1 - 2X\gamma) \pm \sqrt{(1 - 2X\gamma)^2 + 4X\gamma(1 - X\gamma)}}{2A} \quad . \quad (A-10)$$

Defining

$$\frac{\sigma_{z(r=c)}^{(B)}}{\sigma_o} = U \quad , \quad (A-11)$$

one can calculate the average pressure in region B by

$$\sigma_z^{(B)}_{ave} = \frac{\int_0^C \sigma_z^{(B)} dr}{\int_0^C dr} = \frac{\sigma_o U \int_0^C \exp \left[2f \frac{(c-r)}{h} \right] dr}{\int_0^C dr} \quad . \quad (A-12)$$

If $D = \frac{2fc}{h}$,

then

$$\frac{\sigma_z(B)_{ave}}{\sigma_o} = \frac{-Ue^D}{D} [e^{-D} - 1] \quad , \quad (A-13)$$

or by letting

$$V = \frac{e^D - 1}{D}$$

one obtains the relation

$$\frac{\sigma_z(B)_{ave}}{\sigma_o} = UV \quad . \quad (A-14)$$

Equation A-14 can be expressed in terms of experimental quantities by the expression:

$$\sigma_{(theoretical)} = \sigma_{(washer)}(1 - t^2) + \sigma_{(sample)}t^2$$

where

$$\sigma_{(theoretical)} = \frac{\text{applied load}}{\text{area of diamond anvil}} = \frac{\text{Load}}{\pi a^2} \quad (A-15)$$

$$\sigma_{(washer)} = \sigma_z(A) = \sigma_o$$

$$\sigma_{(sample)} = \sigma_z(B)_{ave} = \sigma_o UV$$

and

$$t = c/a.$$

Dividing Equation A-15 by $\sigma_{(\text{sample})}$ one finds

$$\frac{\sigma_t}{\sigma_z(B)_{\text{ave}}} = \frac{(1 - t^2)}{UV} + t^2 \quad (\text{A-16})$$

or by inversion

$$\frac{\sigma_z(B)_{\text{ave}}}{\sigma_t} = \frac{1}{\left[\frac{1}{UV} (1 - t^2) + t^2 \right]} \quad (\text{A-17})$$

Equation A-17 allows one to calculate the average pressure on a sample (B) in the plastic state contained in an elastic washer material (A), by choosing geometrical values of a, c, h, and knowing the coefficient of friction between the sample and the anvil along with the ratio of yield stresses under pressure.

APPENDIX B

One can extend the results from Appendix A to the case where the support ring is in the plastic region by assuming that $\sigma_z(A)$ takes the following form:

$$\begin{aligned}\sigma_z(A) &= \sigma_{z(r=a)}(A) \exp \frac{2f}{h} (a - r) \\ &= \sigma_o(A) \exp \frac{2f}{h} (a - r) \quad .\end{aligned}\tag{B-1}$$

The average axial stress in that region is just

$$\begin{aligned}\sigma_z(A)_{ave} &= \sigma_o \exp \frac{2fa}{h} \frac{\int_c^a \exp - (\frac{2fr}{h}) dr}{\int_c^a dr} \\ &= \frac{\sigma_o}{a - c} \left(- \frac{h}{2f} \right) \exp \left(\frac{2fa}{h} \right) \left[\exp - \left(\frac{2fr}{h} \right) \right]_c^a \\ &= \frac{h\sigma_o}{2f} \frac{1}{a - c} \left[\exp \frac{2f}{h} (a - c) - 1 \right] \quad ,\end{aligned}\tag{B-2}$$

or

$$\frac{\sigma_z(A)_{ave}}{\sigma_o} = Z \tag{B-3}$$

where

$$Z = \frac{h}{2f} \frac{1}{a - c} \left[\exp \frac{2f}{h} (a - c) - 1 \right] \quad . \tag{B-4}$$

Substituting into Equation A-14, one finds

$$\frac{\sigma_z(B)_{ave}}{\sigma_T} = \left[\frac{Z}{UV} (1 - t^2) + t^2 \right]^{-1} \quad . \tag{B-5}$$

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