ESTIMATION OF THE UNIAXIAL STRESS COMPONENT
IN A DIAMOND-ANVIL CAMERA

The specimen is pressurized in an opposed-anvil set-up by the application of uniaxial load. In the absence of a gasket and a fluid pressure transmitting medium, the pressure is not truly hydrostatic. As discussed in Chapter 3, the stress distribution on the specimen can be expressed as the superposition of a uniaxial stress component (USC) and a hydrostatic stress component. The method of determining the USC in a tungsten carbide opposed-anvil camera was discussed in Chapter 3. A method of measuring the USC in a diamond-anvil set-up has been reported by Weaver et al. (47) and Kinsland and Bassett (50, 51). This requires a specially designed diamond-anvil camera wherein the incident x-ray beam passes perpendicular to the loading direction. The x-ray diffraction geometry is different from that in a conventional diamond-anvil camera used for high pressure work. The complete diffraction ring is recorded on a photographic film. In the presence of USC, the diffraction ring becomes elliptical. From the ellipticity of the ring the USC is computed. The disadvantage in this method is
that the x-ray beam passes through the entire region of the specimen and therefore passes through regions of large pressure gradients. This type of data is of limited value as it cannot be used to obtain any other information such as equation of state.

In the present study, a method has been suggested for estimating the USC by analysing the diffraction data collected with a conventional diamond-anvil camera. The results on the measurements of USC in sodium chloride up to 1 GPa are reported.

A.2.1 Experiments

A diamond-anvil camera of the type designed by Bassett et al. (17) was used for the present experiments. Sodium chloride in the form of a fine powder was pressurized between the two diamonds. The x-ray diffraction patterns at several pressures were recorded, using MoKα, photographically. To measure the lattice parameters accurately, a two-film cassette (122) was used. Exposures of the order of 40 hours were given to record the diffraction pattern as intense lines. The lattice parameter was determined from each (hkl) reflection; the standard deviation in 'a' is of the order of 0.005 Å. The linear strain on each (hkl) plane was calculated from the values of 'a'. The pressure on the sample was estimated using
the theoretical equation of state of sodium chloride (52).

A.2.2 Analysis and Discussion

The linear strain (123) in the case of a sample belonging to the cubic system, pressurized in the diamond-anvil camera, is given by

$$\epsilon(hkl) = \epsilon_p + s_{11} t \sin^2 \Theta + s_{12} t \cos^2 \Theta + \frac{s_{13} t}{2} \cos \Theta + \frac{1}{2} s_{44} t \cos \Theta$$

Expressing $\sin^2 \Theta$ in terms of $\cos^2 \Theta$, this can be written as

$$\epsilon(hkl) = (\epsilon_p + s_{11} t) + (s_{12} - s_{11}) t \cos^2 \Theta + \frac{s_{13} t}{2} \cos \Theta + \frac{1}{2} s_{44} t \cos \Theta$$

... ... ... (1)

where

$$\epsilon(hkl) = \frac{a(hkl)}{a_0} - 1$$

$\epsilon_p$ = the strain arising from the hydrostatic component

$S = s_{11} - s_{12} - \frac{1}{2} s_{44}$

$S_{ij}$ = the elastic compliances

$t$ = the uniaxial stress component

$$\gamma(hkl) = \frac{h^2 k^2 + k^2 l^2 + l^2 h^2}{(h^2 + k^2 + l^2)^2}$$

$\Theta$ = the Bragg angle corresponding to the $(hkl)$ reflection.

$a(hkl)$ = the lattice parameter determined from the $(hkl)$ reflection under pressure and

$a_0$ = the lattice parameter at atmospheric pressure.
It is to be noted that Eq. (1) holds good only for substances crystallizing in the cubic system. The quantities \( \xi(hkl) \) and \( \Theta \) have been experimentally measured. An analysis of the linear strains has been made by fitting them to Eq. (1).

The first six reflections - namely (200), (220), (222), (400), (420) and (422) - from sodium chloride were generally observed on each pattern. Thus, at each pressure, six equations were obtained. These were treated as a system of simultaneous equations with the three unknown parameters \( (\xi_p + s_{11}t) \), \( (s_{12} - s_{11})t \), and \( St \). The values of these parameters were determined by the method of least squares, using the programme listed in Section A.2.2.1.

The strain component \( \xi_p \) can be regarded as the average of strain on the (hkl) reflections observed. The value of \( s_{11}t \) can then be estimated. Thus we have the three quantities - \( St \), \( (s_{12} - s_{11})t \) and \( s_{11}t \). These are presented as a function of pressure in Fig. 32. The scatter in the data is typical of such plots. The large scatter arises because the accuracy in the lattice strain measurement in high pressure experiments is not very high.

The quantities \( s_{11}t \), \( (s_{12} - s_{11})t \) and \( St \) have been used to estimate the average values of the ratios \( s_{11}/S \) and \( (s_{12} - s_{11})/S \). The results obtained are - 2.50 for
Fig. 32. Plot of (a) $St$, (b) $(s_{12} - s_{11})t$ as a function of pressure.
Fig. 32(c). Plot of $s_{11}t$ as a function of pressure.
$s_{11}/S$ and 2.31 for $(s_{12} - s_{11})/S$. The single crystal elastic constants of NaCl (59) give these values respectively as -1.924 and 2.317 at one atmosphere. Considering the large scatter involved, the values of the ratios obtained by the present analysis agree well with the single crystal measurements. This agreement supports the usage of Eq. (1) for estimating the USC.

The values of $s_{ij}$ can be regarded as equal to the values at atmospheric pressure, since the range of pressure in the present experiments is small. Using the $s_{ij}$ values from single crystal measurements (59), the values of $t$ have been determined. The mean values of $t$ determined from $St$, $(s_{12} - s_{11})t$ and $s_{11}t$ are -0.27 GPa, -0.11 GPa and -0.15 GPa. The average value of $t$ thus turns out as -0.18 GPa.

The uniaxial stress component in sodium chloride was measured by Kinsland and Bassett (50,51). The diamond-anvil cell was modified to allow the incident x-ray beam in a direction normal to the loading direction. The x-ray diffraction pattern was recorded on a flat film. The diffraction ring, in the presence of USC, appeared in the form of an ellipse. The USC in sodium chloride was estimated by measuring the ellipticity of the (200) and (220) diffraction rings.
The results obtained by Kinsland and Bassett have been reproduced in Fig. 33. The crosses denote data from the experiments of Bridgman (124) on the shearing strength of sodium chloride. The value of $t$ increases to $-0.4$ GPa. The mean value of $t$ up to a pressure of 1 GPa is nearly $-0.1$ GPa.

In the case of a tungsten carbide anvil camera, the USC is estimated (49) from a plot of $\varepsilon(hkl)$ versus $\Gamma(hkl)$. The function $\Gamma(hkl)$ depends only on the indices $h$, $k$ and $l$ and does not depend on the diffraction angle. Thus the variation of $\varepsilon(hkl)$ with $\Gamma(hkl)$ is not affected by the systematic errors. The value of USC (57) in sodium chloride, as measured with a tungsten carbide anvil camera, is $-0.24$ GPa.

It is clear from Eq. (1) that $\varepsilon(hkl)$ in the case of a diamond-anvil camera, depends on $\theta$, the angle of diffraction. The value of $\theta$ is generally less than $20^\circ$ since MoKα radiation is used for recording the diffraction pattern. In the low angle region, the systematic errors, arising from factors like the absorption of radiation in the sample, uncertainty in the sample-to-film distance, are significant. The systematic errors are dependent on $\theta$. Since $\varepsilon(hkl)$ depends on $\theta$, these errors are involved in the estimation of $t$, the USC. To eliminate the systematic errors,
FIG 33. DATA OF KINSLAND AND BASSETT (51): STRENGTH VERSUS PRESSURE FOR NaCl AS DETERMINED FROM THE DIFFRACTION FROM (a) THE (2 2 0) AND (b) THE (2 0 0) LATTICE PLANES.
a two-flim cassette (123) was used for the studies reported here so that a meaningful estimate of t could be made.

The present results compare well with the value of USC as measured by Kinsland and Bassett (51) and also with the value derived from measurements (57) with a tungsten carbide anvil camera.

The present method has an advantage over the method suggested by Weaver et al. (47) and Kinsland and Bassett (50, 51) in that the high pressure diffraction data obtained from a diamond-anvil camera can be straightaway used. No separate data with a special camera need be collected to estimate the USC.

It is customary now to use a gasket and a liquid pressure transmitting medium to eliminate the USC and provide truly hydrostatic pressure. Even in such cases, there always exists the uncertainty about the containment of the liquid pressure transmitter in the gasket. The present method can be used easily to detect the presence of USC in the routinely collected high pressure x-ray diffraction data.
A 221.

PROGRAM TO FIND THE COEFFICIENTS F(x)

DIMENSION F(7C0), H(7C0), K(7C0), L(7C0), 7(7C0), 1(7C0), 1(7C0)

DIMENSION X(7C0), SLC(7C0), TH(7C0), 7(7C0)

INTEGER I

1 FORMAT(4*(I3))

2 FORMAT(4*(F5.4))

3 FORMAT(4*(F4.1))

4 FORMAT(1*(I3), 1*(F4.1), 1*(F4.1), 1*(I3))

REAL X, T

REAL 7, 1

REAL F(J(I)), C(I), S(I), A(C(I)), T(I)

REAL 7, 1, L(1), 7(1), T(1)

M=1

L=1

C(I)=0

DO 15 I=1, 7

15 CONTINUE

F(I)=A(I)/A(I)

S(I)=C(I)-A(I)*L(I)*A(I)

S(I)=-S(I)

SLC=SLC+C(I)

MST=1

F(I)=F(I)+MST

1 I=I+1

15 CONTINUE

C(I)=T(I)
\begin{verbatim}
C(12) = \text{STOP(12)}
\end{verbatim}