Uniaxial stress component in diamond anvil high-pressure x-ray cameras

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The effect of the uniaxial stress component on the lattice strains measured with a diamond anvil x-ray apparatus has been discussed. The results are compared with those published earlier for the tungsten carbide anvil apparatus.

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The experimental setups based on the principle of opposed anvils have been extensively used in x-ray diffraction studies of solids at elevated pressures.¹⁻⁵ In these devices, the sample is pressed between the anvils (with or without a gasket) and the sample is pressurized by the application of an uniaxial load. Because of the fact that the sample possesses a finite yield strength which invariably increases with increasing pressure and that the sample is pressurized by an uniaxial load, the pressure on the sample is nonhydrostatic. The stress distribution is cylindrically symmetric about the direction of loading, the axial component of stress being larger than the radial stress component. Such a pressure distribution can be better approximated by the superposition of a hydrostatic component and an uniaxial stress component along the direction of loading. For a tungsten carbide anvil setup it was shown that the uniaxial stress component produces a strain which depends on the indices of the reflection.⁶ Thus, the observed strain, which is the sum of the strains arising from the hydrostatic component and from the uniaxial stress component, also depends on the indices of the reflection. Further, it was shown that the magnitude of the uniaxial stress component could be estimated by analyzing the x-ray diffraction data. The variation with pressure of the uniaxial stress component in sodium chloride? was measured using this technique.

In this paper, the effect of the uniaxial stress component on the lattice strains measured with a diamond anvil camera is presented. These results are compared with those derived earlier for the tungsten carbide anvil apparatus.⁶

The diffraction geometry for a diamond anvil apparatus is shown in Fig. 1. The incident x-ray beam passes parallel to the direction of applied load. For this reason this geometry will be referred to as "parallel geometry" (PL-G) in the subsequent discussion. This diffraction geometry is different from that encountered in a tungsten carbide anvil setup.⁶ In this case the incident x-ray beam passes perpendicular to the direction of applied load. This diffraction geometry will be termed as the "perpendicular geometry" (PD-G).

Let t and p denote, respectively, the uniaxial and the hydrostatic components at an applied load w between the anvils. The lattice strain, for a sample belonging to a cubic system, in a direction (hkl) is given by

$$\begin{aligned}\epsilon_w(hkl) &= [d_w(hkl) - d_0(hkl)]/d_0(hkl) \\ &= [a_w(hkl) - a_0]/a_0 \\ &= \epsilon_w^p + \epsilon_w^t(hkl),\end{aligned}\quad (1)$$

where $d_w(hkl)$ and $d_0(hkl)$ are the interplanar spacings, respectively, at load w and at zero load.

With similar meanings, $a_w(hkl)$ and $a_0(hkl)$ denote the lattice parameters. ϵ_w^p and $\epsilon_w^t(hkl)$ denote, respectively, the strains due to p and t . It can be shown in PL-G by following the arguments similar to those given in Ref. 6 that

$$\begin{aligned}\epsilon_w^t(hkl) &= S_{11}t \sin^2\theta + S_{12}t \cos^2\theta + (S_{11} - S_{12} - \frac{1}{2}S_{44}) \\ &\quad \times \Gamma(hkl)t(1 - 3 \sin^2\theta),\end{aligned}\quad (2)$$

FIG. 1. The diffraction geometry of a diamond anvil high-pressure x-ray camera.

where, S_{ij} are the single crystal elastic compliances, θ is the diffraction angle, and

$$\Gamma(hkl) = (h^2k^2 + k^2l^2 + l^2h^2)/(h^2 + k^2 + l^2)^2.$$

In general, S_{ij} and t are functions of pressures.

In deriving Eq. (2), the condition of stress continuity across the interface of two crystallites has been assumed. If, on the other hand, the condition of strain continuity is assumed, the following equation for $\epsilon_w^t(hkl)$ is obtained:

$$\epsilon_w^t(hkl) = (1/E \sin^2\theta - \nu \cos^2\theta) t,$$

where

$$\nu = (C_{11} + 4C_{12} - 2C_{44})/(2C_{11} + 3C_{12} + C_{44})$$

and

$$E = (C_{11} - C_{12} + 3C_{44})(C_{11} + 2C_{12})/(2C_{11} + 3C_{12} + C_{44}).$$

The conditions of the stress and the strain continuities constitute the two extremes, and the condition prevailing in reality lies between these two extremes. In calculating the elastic constants of a polycrystalline sample in terms of the elastic constants of a single crystal, it is customary to take the average of the values obtained for the two extreme cases. This is often termed as Voigt-Reuss-Hill average.⁸ However, there are indications that the conditions of constant stress exist in the samples at high pressures. For this reason, the state of constant stress only will be used in the subsequent discussions. However, the arguments can easily be extended to any other combination of the two states with the help of Eqs. (2) and (3) as was discussed in an earlier paper.⁶

It is clearly seen from Eq. (2) that $\epsilon_w^t(hkl)$ depends on the diffraction angle θ , in addition to the other parameters such as $\Gamma(hkl)$, S_{ij} , and t . For a given set of values of $\Gamma(hkl)$, S_{ij} , and a compressive t , $\epsilon_w^t(hkl)$ is positive for small values of θ . As θ increases, the magnitude of $\epsilon_w^t(hkl)$ decreases linearly with $\sin^2\theta$, passes through zero, and becomes negative.

The measured lattice strain is a sum of ϵ_w^t and $\epsilon_w^t(hkl)$ as indicated by Eq. (1). Since ϵ_w^t is negative, and $\epsilon_w^t(hkl)$ is positive for low θ values and a compressive t , the measured lattice strain is smaller in magnitude than ϵ_w^t . However, since $\epsilon_w^t(hkl)$ can become negative for large θ values, $\epsilon_w(hkl)$ can become larger than ϵ_w^t . In PD-G, $\epsilon_w(hkl)$ is always smaller than ϵ_w^t .

For a comparison of PL-G and PD-G, the numerical values of $\epsilon_w^t(hkl)/t$ have been calculated for both PL-G and PD-G, for sodium chloride and silicon (Table I). It is readily seen that $\epsilon_w^t(PL-G)$ is smaller than the corresponding $\epsilon_w^t(PD-G)$. With MoK α radiation, reflections over an approximate range $5^\circ < \theta < 20^\circ$ are observed. Over this θ range, the average value of $\epsilon_w^t(hkl)$ in PL-G is roughly one-half of the corresponding value in PD-G. However, $\epsilon_w^t(PL-G)$ can become larger than $\epsilon_w^t(PD-G)$ if a sufficiently large θ value is reached.

The behavior of $\epsilon_w^t(hkl)$ in PD-G was discussed in an earlier paper.⁶ In PD-G, $\epsilon_w^t(hkl)$ depends on $\Gamma(hkl)$, t , and S_{ij} ; since it does not depend on θ , the interpretation of the $\epsilon_w(hkl)$ -vs- $\Gamma(hkl)$ plot is simple. The $\epsilon_w(hkl)$ -

TABLE I. A comparison of $\epsilon_w^t(hkl)$ in PD-G and PL-G.

Reflection	$-\epsilon_w^t(PD-G)/t$ (10^{-2} GPa $^{-1}$)	$-\epsilon_w^t(PL-G)/t$ (10^{-2} GPa $^{-1}$)	$\epsilon_w^t(PL-G)/\epsilon_w^t(PD-G)$
Silicon			
111	0.0960	0.0880	0.9168
220	0.1255	0.1010	0.8048
311	0.1584	0.1201	0.7579
400	0.2140	0.1467	0.6853
331	0.1169	0.0607	0.5193
422	0.1255	0.0519	0.4135
511	0.1892	0.0842	0.4450
440	0.1255	0.0213	0.2175
531	0.1392	0.0256	0.1839
Sodium chloride			
200	0.4650	0.4213	0.9060
220	0.7688	0.6523	0.8485
222	0.8699	0.6808	0.7826
400	0.4650	0.2900	0.6237
420	0.6594	0.3880	0.5884
422	0.7688	0.4193	0.5454
442	0.8250	0.2766	0.3353

vs- $\Gamma(hkl)$ plot is a straight line; the slope of the line is given by $(S_{11} - S_{12} - \frac{1}{2}S_{44})t$ and the intercept by $\epsilon_w^t + S_{12}t$. If S_{ij} and its pressure dependence are known, t can be estimated from the measured value of the slope.^{6,7}

The dependence of $\epsilon_w(hkl)$ on θ , in addition to the parameters S_{ij} , t , and $\Gamma(hkl)$ for PL-G, does not permit a simple interpretation of the $\epsilon_w(hkl)$ data in the same way as could be done in PD-G. It might still seem possible to evaluate t from the measured values of $\epsilon_w(hkl)$, by fitting to the $\epsilon_w(hkl)$ values an equation of the form given in Eqs. (1) and (2). One, however, faces serious practical problems in using such a procedure to estimate t . The first problem arises from the smallness of the magnitude of $\epsilon_w^t(hkl)$ in PL-G. As has been discussed earlier in this paper, the magnitude of $\epsilon_w^t(hkl)$ in PL-G is, on the average, nearly one-half the $\epsilon_w^t(hkl)$ value in PD-G. The second problem is connected with the influence of the systematic errors of the measurement on the estimation of t . The systematic errors⁹ arising from the geometrical and the physical factors depend on θ and, since $\epsilon_w(hkl)$ also depends on θ , the systematic errors will interfere with the estimation of t . However, if the systematic errors are minimized, e. g., by the use of a two-film cassette,¹⁰ it might still be possible to obtain a meaningful estimate of t . It may be recalled that in PD-G, a $\epsilon_w(hkl)$ -vs- $\Gamma(hkl)$ plot is used to estimate t .^{6,7} Since there is no correlation between $\Gamma(hkl)$ and θ , the θ -dependent systematic errors appear as random errors, and do not affect the estimation of t .

The discussions presented in this paper indicate that in PL-G the effect of the uniaxial stress component is less pronounced than in the PD-G. For this reason, if the aim of the experiment is to estimate the uniaxial stress component, then the PD-G is preferable to PL-G.¹¹ However, if the aim of the experiment is to measure the equation of state, then the PL-G is preferred to have a slight advantage over the PD-G at least in cases wherein the samples can support large uniaxial stress component. If only a few low-angle reflection are recorded, then the magnitudes of $\epsilon_w^t(hkl)$ are comparable in the two diffraction geometries.

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