

Compression of lithium fluoride to 92 GPa

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The equation of state and compression behavior of lithium fluoride, LiF, have been determined to 92 GPa by X-ray diffraction in a diamond anvil cell. A neon pressure-transmitting medium was used to minimize the effect of differential stress on the sample. Consistent results using multiple pressure standards were obtained. By fitting the pressure–volume data to a Birch–Murnaghan equation of state, the isothermal bulk modulus and its corresponding pressure derivative at zero pressure were determined to be $K_0 = 70.1 \pm 0.7$ GPa, and $K'_0 = 4.3 \pm 0.1$. If the bulk modulus is fixed at 66.2 GPa, a value well constrained by independent elasticity measurements, we obtain $K'_0 = 4.6 \pm 0.1$. The bulk modulus and its pressure derivative obtained from this work resolve the large discrepancy in previously reported values of K_0 and K'_0 for this material. Consequently, the equation of state of LiF is now sufficiently well constrained to allow its use as a pressure calibrant in high pressure experiments.

Keywords: lithium fluoride; equation of state; X-ray diffraction

Introduction

Alkali halides are prototypical ionic solids and are model systems for studies of many high pressure properties including compressibility,[1,2] equations of state,[3,4] phase transformations,[5] melting,[6] vibrational spectra,[7] and pressure calibration.[8] They have also played a role in the validation of new phenomenological and first-principles theoretical methods over the last 35 years.[9–13] Recent studies have re-examined the properties of LiF at high pressures using first-principles calculations.[14,15] Alkali halides, especially NaCl, are widely used as pressure calibrants, pressure-transmitting media, and insulating materials in high pressure and temperature experiments.

Lithium fluoride, LiF (Griceite), is an alkali halide with the NaCl rocksalt structure. LiF has many attributes that make it a potential alternative to sodium chloride for use as an insulator, pressure-transmitting medium, and pressure standard in diamond anvil cell experiments.[3] As an insulator or pressure transmitter, LiF is considerably less hygroscopic [16] than NaCl and thus is less likely to form reaction products with the sample upon laser heating. Unlike NaCl, LiF undergoes no phase transformations at room temperature up to pressures well above 1 Mbar.[6,15] Historically, the weaker X-ray scattering power of LiF has limited its use as a pressure standard compared with NaCl. However, with the third-generation synchrotron sources, this is less of a

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problem, and the weaker diffraction pattern (and wide phase stability) may be beneficial in cases where interference of diffraction lines among sample materials is to be avoided.

There has also been wide interest in the behavior of LiF under dynamic compression.[17,18] Due to its optical transparency to high compression, LiF is the most versatile window material through which to carry out wave profile measurements for shock compression experiments.[19] It has also recently been characterized for use in ramp compression experiments which dynamically explore lower temperatures compared with shock compression.[20,21]

Despite its importance, the high-pressure static compression behavior of LiF has been determined only up to 9 GPa under hydrostatic conditions [2] and ~ 30 GPa under non-hydrostatic compression.[22] A large number of measurements have been reported of the zero-pressure bulk modulus, K_0 , of LiF using ultrasonic methods and Brillouin spectroscopy.[23,24] Neglecting older measurements (pre-1959) and a few outliers, an average of 10 independent elasticity measurements yields $K_0 = 66.2 \pm 1.5$ GPa. Note that the measured adiabatic bulk moduli have been corrected here to give an average isothermal value. However, the reported value of the pressure derivative of the bulk modulus from previous ultrasonic, differential length change, shock compression, X-ray diffraction, and theoretical studies spans a wide range of values from 3.6 to 5.4.[2,14,15,22,25–30]

In this work, we provide new experimental constraints on the equation of state of LiF. We use X-ray diffraction in diamond anvil cells to investigate the static compression behavior of LiF at pressures up to 92 GPa. We examine LiF in a quasi-hydrostatic (differential stress $<1\%$ of the pressure) pressure medium and evaluate the stress state in the sample.

Experiments

The starting material was LiF (99.99% purity) powder obtained from Alfa Aesar. The zero-pressure volume at ambient conditions was measured to be $65.19(1) \text{ \AA}^3$, which is consistent with the literature value of 65.31 \AA^3 . [25] Three individual experiments were conducted on LiF using high-pressure synchrotron X-ray diffraction in diamond anvil cells. Anvils were supported by tungsten carbide seats with 50° conical openings. We used symmetric cells with 200 or 300 μm diamond culets. LiF samples were loaded into 100- or 160- μm -diameter holes in rhenium gaskets which were pre-indented to $\sim 30\text{-}\mu\text{m}$ thickness before sample loading. In each run, a different pressure calibrant was used. For the first sample, we used a piece of $\sim 7 \mu\text{m}$ -thick Mo foil and a ruby sphere for pressure measurements. For the second run, LiF powder was mixed with 10–15 wt% Au (99.999% purity). For the last experiment, LiF was mixed with 50 wt% MgO. Neon was loaded as a pressure-transmitting medium using the COMPRES/GSECARS gas loading system.[31]

Angle-dispersive X-ray diffraction experiments were performed at beamlines 13-ID-D at GSECARS and 16-ID-B at HPCAT of the Advanced Photon Source, Argonne National Laboratory. A monochromatic beam was focused to $\sim 5 \times 6 \mu\text{m}^2$ (GSECARS) and $\sim 5 \times 10 \mu\text{m}^2$ (HPCAT) using Kirkpatrick–Baez mirrors. Diffraction patterns were collected using a MarCCD detector. The detector position and orientation was calibrated with a CeO_2 powder standard using the program FIT2D.[32] Pressures were determined based on the ruby fluorescence scale [33] and equations of state of Au [33] and MgO.[34]

Results and discussion

LiF samples were compressed in $\sim 2\text{--}5$ GPa intervals to maximum pressures of 56, 92, and 38 GPa in the three runs. Figure 1 shows representative X-ray diffraction patterns from the three

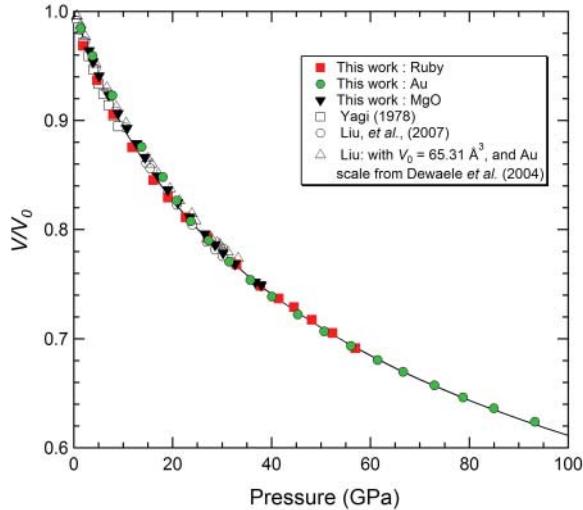


Figure 2. Pressure–volume relations for LiF from this work and previous measurements. This work: solid symbols. The solid line is a third-order Birch–Murnaghan fit to the volume data from the three runs. Open symbols are from previous X-ray diffraction measurements.[2,22] Open triangles show data from Ref. [22] corrected to the literature V_0 and pressure scale used in this work.

experiments. A typical ruby fluorescence spectrum is shown in the inset of Figure 1(a). The R_1 and R_2 peaks of ruby remained well separated to 56 GPa. Because the peaks of Mo were weak, errors on the Mo cell volumes are high, and Mo was not used to determine pressure. Unit cell volumes were determined from the least-squares fit to two or three diffraction peaks from among (111), (200), and (220) of LiF. Pressure was determined from ruby fluorescence in run 1. In runs 2 and 3, pressures were determined based on $a(111)$ of Au and $a(200)$ of MgO, respectively, which are the orientations least sensitive to differential stress.[35] The volumetric strain (V/V_0) of LiF as a function of pressure is shown in Figure 2. The compression data are in good agreement with previous static compression X-ray measurements.[2,22] Although experiments by Liu et al.(2007) were performed without a pressure-transmitting medium, their V/V_0 values coincide with our results (Figure 2). In that study, the adopted value for the zero-pressure volume, $V_0 = 65.61(3) \text{ \AA}^3$ was larger than the commonly used initial volume of 65.31 \AA^3 , [25,36] resulting in lower relative volumes. They also used an older Au scale [37] that underestimates pressures by approximately $\sim 4\%$ compared with the more recent scale by Dewaele et al. used here.[33] The differences in V_0 and pressure scale may explain this study’s apparent agreement with the present results despite the difference in stress conditions.

The pressure–volume data from our three runs were fit together to a third-order Birch–Murnaghan equation of state, yielding an isothermal bulk modulus, K_0 , of 70.1 ± 0.7 GPa and the corresponding pressure derivative $K'_0 = 4.3 \pm 0.1$ ($K'_0 = dK_0/dP$ at $P = 0$ GPa). We fixed the zero-pressure volume at 65.19 \AA^3 in the fitting. If we use the literature value $V_0 = 65.31 \text{ \AA}^3$, similar results are obtained: $K_0 = 69.0 \pm 0.7$ GPa and $K'_0 = 4.3 \pm 0.1$. The fit results are listed in Table 1 and compared with previous studies. If we fit the data from the three runs individually, nearly the same results are obtained with the ruby fluorescence or Au scale: ruby scale: $K_0 = 67.5 \pm 1.4$ GPa, $K'_0 = 4.5 \pm 0.1$; gold scale: $K_0 = 68.8 \pm 1.4$ GPa, $K'_0 = 4.4 \pm 0.1$. However, fitting the data of run 3 using the MgO scale gave a higher value of $K_0 = 76.4 \pm 0.5$ GPa, and lower value of $K'_0 = 3.7 \pm 0.1$. This may be due to the more limited pressure range of this run compared with the other two and the tradeoff between K_0 and K'_0 .

The adiabatic bulk modulus of LiF has been determined repeatedly in experiments using ultrasonic interferometry or Brillouin scattering [23,24] (Table 1). Excepting a few outliers, these

Table 1. Equation of state parameters for LiF from this and previous work.

$V_0(\text{\AA}^3)$	$K_0(\text{GPa})$	K'_0	Method	Pressure scale	Reference
65.19(1) ^a	70.1(7)	4.3(1)	XRD	Ruby ^b , Au ^b , MgO ^c	This work
	66.2 ^a	4.6(1)	XRD	Ruby ^b , Au ^b , MgO ^c	This work
	66.2(9)	3.6(3)	XRD	NaCl ^d	Yagi [2]
65.61(3)	74.4(8)	3.69(7)	XRD	Au ^e	Liu et al.[22]
	65.31	5.26	US		Miller and Smith [25]
	66.6	4.65	US		Drabble and Strathen [26]
	66.45(5)	5.40(18)	DLC		Kim et al.[27]
65.04	65.32(23)	5.10(21)	DLC		Boehler and Kennedy [28]
	67.9(1.0)	4.51(7)	SC		Van Thiel. [29]
	67.1(8)	4.56(4)	SC		Marsh [30]
68.62	70.16	4.02	<i>Ab initio</i>		Sun et al.[14]
65.02	73.8	3.76	<i>Ab initio</i>		Smirnov [15]

Notes: XRD, X-ray diffraction; US, ultrasonic interferometry; DLC, differential length change; SC, shock compression. Adiabatic values from ultrasonic measurements and shock compression have been corrected to the isothermal values.

^aFixed value.

^bDewaele et al.[33].

^cSpeziale et al.[34].

^dDecker [8].

^eAnderson et al.[37].

studies are very consistent with each other, and, when corrected to isothermal conditions, an average bulk modulus of 66.2 ± 1.5 GPa is obtained. If K_0 of LiF was fixed to this value of 66.2 GPa, we obtain an average value of K'_0 of 4.6 ± 0.1 using the data from individual and multiple runs.

The equation of state of LiF has also been studied extensively by shock compression.[29,30] Fitting the Hugoniot data (shock velocity, U_s , particle velocity, u_p) to a shock wave equation of state, $U_s(\text{km/s}) = c_0 + su_p$, enables us to determine the corresponding adiabatic bulk modulus and its pressure derivative following from [38]:

$$c_0 = \left(\frac{K_{0s}}{\rho_0} \right)^{1/2}, \quad (1)$$

$$s = \left(\frac{K'_{0s} + 1}{4} \right). \quad (2)$$

Here K_{0s} and K'_{0s} refer to the adiabatic bulk modulus and its pressure derivative at 0 GPa, respectively. From a fit to the combined shock wave data of [29,30] ($U_s(\text{km/s}) = (5.16 \pm 0.023) + (1.35 \pm 0.01)u_p$) and converting from adiabatic to isothermal conditions, we obtain $K_0 = 67.4 \pm 0.6$ GPa and $K'_0 = 4.6 \pm 0.04$. Shock compression and ultrasonic studies thus consistently yield a bulk modulus of 66–67 GPa. In contrast, a previous static compression study of LiF reported $K_0 = 74.4(8)$ GPa,[22] a value that is $\sim 12\%$ higher than that of the mean of the elasticity studies and $\sim 6\%$ higher than the value from this study. Two recent theoretical calculations [14,15] yield values for K_0 (70.2 and 73.8 GPa) although a portion of the difference with experiment in this case may be attributable to the temperature differences between 0 and 300 K.

It is well known that K'_0 may suffer from tradeoffs with V_0 and K_0 . In order to evaluate this effect, we fixed V_0 at 65.19\AA^3 and varied K_0 from 55 to 80 GPa. The solid line in Figure 3 shows the locus of K'_0 values from fits to the third-order Birch–Murnaghan equation of state. K_0 and K'_0 from other studies are also shown in Figure 3 for comparison. The dash-dot lines show the one-sigma uncertainty about the average values of $K_0 = 66.2 \pm 1.5$ GPa from the previous ultrasonic and Brillouin work. It is notable that when K_0 is fixed to this value, the obtained pressure derivative, K'_0 , is consistent with independent shock wave data. While previous ultrasonic data yield a well-constrained value of K_0 , they exhibit a wide range of reported values of K'_0 values (Figure 3).

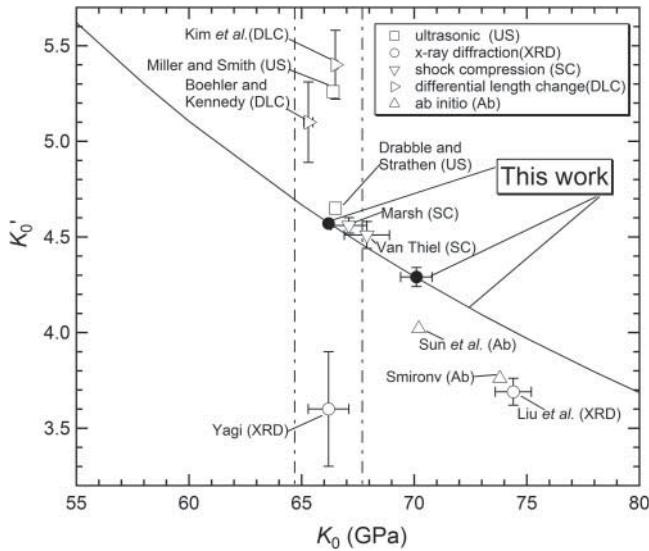


Figure 3. Tradeoff curve showing values of bulk modulus, K_0 , and its pressure derivative, K'_0 , that satisfy the present data with $V_0 = 65.19 \text{ \AA}^3$. Dash-dotted lines show one standard deviation uncertainty on K_0 from the mean of 10 previous ultrasonic and Brillouin measurements.[23,24] Individual results from static compression, ultrasonic interferometry, and differential length change, and theory that report both K_0 and K'_0 are also shown (see Table 1 for references). The values derived from shock wave studies are discussed in the text.

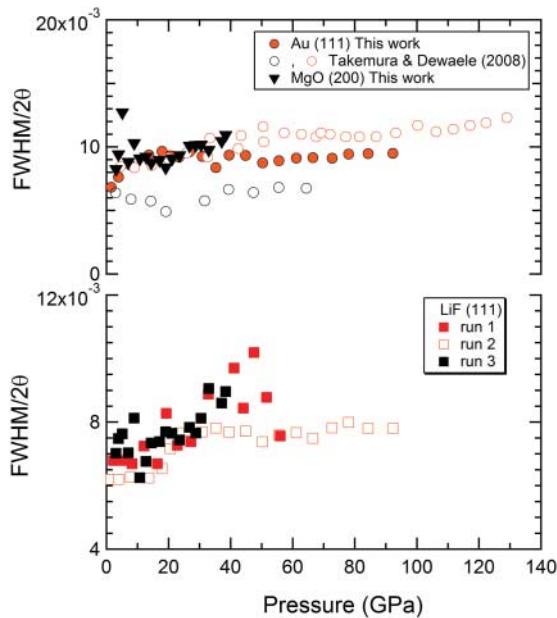


Figure 4. Pressure dependence of the peak width (FWHM) (normalized to diffraction angle 2θ) for LiF (111) (squares), Au (111) (solid and open circles), and MgO (200) (triangles). Orange solid circles are the results for Au (111) from this work; open circles (orange: foil; black: powder) are the data of Au from Takemura and Dewaele in He medium.[35]

As has been observed in other materials, the weaker constraints on pressure derivatives reflects the greater difficulty in determining elastic properties at elevated pressures compared to ambient pressures and the limited pressure range of such measurements. In comparison with the earlier studies, our K'_0 value is in best agreement with the ultrasonic value of Ref. [26].

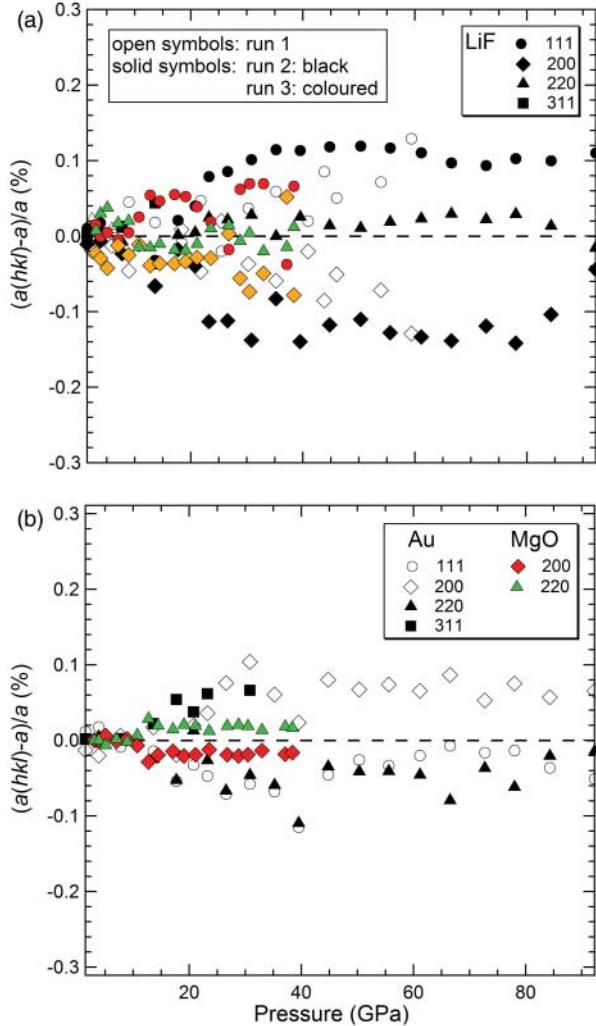


Figure 5. Percent difference between the lattice parameter given by each diffraction peak and the average value at each pressure step for (a) LiF and (b) Au and MgO.

To establish the equation of state, the sample pressure and volume should be determined with high accuracy. However, a completely hydrostatic environment cannot be sustained above 12 GPa due to the solidification of all known pressure media at room temperature.[39] Since non-hydrostaticity will cause systematic error in the volume measurements, it is important to evaluate the stress states in the sample. The sample in the diamond anvil cell is subjected to differential stress due to the uniaxial stress field, which leads to relative diffraction peak shifts.[40] Additionally, deviatoric stress is produced by heterogeneous strain at grain boundaries of the polycrystalline sample, which broadens diffraction peaks under pressure.[41,42]

The stress conditions in the sample can be evaluated from the relative shifts of diffraction lines [40] and the full width at half maximum (FWHM) of diffraction peaks.[42] Figure 4 shows the FWHM (111) for LiF and Au and FWHM (200) for MgO normalized to the diffraction angle, 2θ , versus pressure. The normalized FWHMs of the LiF and Au peaks steadily increase with pressure before ~ 18 GPa, then flatten at high pressure. Similarly, the normalized FWHM of Au started to saturate at ~ 25 GPa in a helium pressure medium in experiments by Takemura and Dewaele [35]

(Figure 4). These observations agree well with the measurements of the hydrostatic limits of Ne and He pressure media.[43]

Differential stress also results in the deviation of lattice parameters measured for individual diffraction lines with increasing pressure as a function of lattice plane, (hkl) . [40] Figure 5 shows the difference of lattice parameters calculated from each diffraction peak and the average of all the calculated parameters plotted against pressure of LiF (Figure 5(a)) and Au and MgO (Figure 5(b)). Variation of calculated lattice parameters from the mean value is slightly greater for LiF ($\pm 0.14\%$) than for Au ($\pm 0.1\%$) and MgO ($\pm 0.02\%$). The variation of volume of LiF calculated from the measured lattice parameter, $a(hkl)$, is within $\sim 0.3\%$. At the highest pressures examined, the variation of pressures calculated from $a(hkl)$ of Au and MgO are less than 1.3% and 0.8%, respectively. However, nearly the same equations of state parameters are recovered from our data set regardless of whether the (111), (200), or an average of three lines of LiF are used in the volume calculation for the compression curves. These indicate that the differential stresses are sufficiently minimal in these experiments.

Conclusions

We performed X-ray diffraction experiments on LiF at high pressure with a Ne pressure-transmitting media. The equation of state parameters of LiF are well constrained to 92 GPa. The fit to the P - V/V_0 data give a bulk modulus of $K_0 = 70.1 \pm 0.7$ GPa, and pressure derivative of bulk modulus $K'_0 = 4.3 \pm 0.1$. If the bulk modulus is fixed at an ultrasonic value, 66.2 GPa, the fit yields $K'_0 = 4.6 \pm 0.1$. Based on diffraction peak shifts analyzed against the Au pressure calibrant, the Ne-pressure medium maintained quasi-hydrostatic conditions with differential stress $< 1\%$ of the pressure. Analysis of peak widths and individual diffraction peak variation indicates that differential stresses are minimal in these experiments. LiF has many of the attributes of a good pressure calibrant, pressure-transmitting medium, and insulating medium in high pressure and temperature experiments in the diamond anvil cell. Compared with NaCl, LiF has the advantages that it is less hygroscopic and undergoes no phase transformation to above 1 Mbar. However, discrepancies in previous equation of state measurements, especially the wide range of reported K'_0 values precluded reliable use of LiF as a calibrant. Our results enable us to reconcile previous shock, elasticity, and static measurements of equation of state parameters for this material. With a well-constrained value of K'_0 from this study, LiF is now a good candidate as a pressure standard for high pressure experiments.

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