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Elastic properties of amorphous selenium at high pressure

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The elastic constants C^l and C^t for amorphous Se have been measured as a function of hydrostatic pressure up to 16 kbar. The isothermal bulk modulus K_T is evaluated and found to exhibit a softening effect with increased pressure. The pressure dependence of K_T is interpreted in terms of an interatomic potential composed of two terms; a Lennard-Jones term, and a phenomenologically introduced attractive term due to intrachain-interchain interaction. It is shown that this new potential predicts the well-known transition at 130 kbar and that K_T derived from it agrees very well with the data. In the limit of zero pressure it appears that the Lennard-Jones term alone is sufficient to describe the elastic properties of a-Se.

I. INTRODUCTION

The most important electronic bands responsible for the structure of selenium are those which correspond to the electronic configuration $4s^2p^4$. However, the detailed nature of the bands and structure of amorphous selenium (a-Se) is not as well understood as is crystalline selenium (c-Se). Recently, some gains into the subject have been advanced by Shevchik, Cardona, and Tejeda,[1] through their ultraviolet and x-ray photoemission measurements and by Kramer, Maschke, and Laube,[2] through their band-structure calculations. The main results show that the electronic band structure of a-Se is very similar to that of c-Se, compelling Kramer, Maschke, and Laube to conclude that a-Se exhibits the same short-range order as does c-Se. They view the disordered nature of a-Se as small statistical variations of bond lengths and angles about their mean values. If these conclusions are correct, the nature of the bond forces in a-Se should be basically the same as in c-Se. In order to test these observations we measured the bulk modulus K (inverse compressibility) of a-Se as a function of hydrostatic pressure. The reason for choosing to measure K is that K is derivable from an interatomic potential, thus making it possible to test, from its pressure dependence, any potential form proposed by theory.

Extensive information about bond forces was obtained from studies of elastic and optical properties of trigonal Se and Te as a function of pressure.[3-8] The main results show the elastic properties $C_{33} \gg C_{11}$ and that C_{11} increases more rapidly with pressure than does C_{33}. This is expected from what is known about the structure and nature of interbond forces in c-Se, as discussed below.

The crystal structure[9] of trigonal Se consists of helical chains which spiral around axes parallel to the c axis. The helices are arranged in a hexagonal array. The fact that $C_{33} \gg C_{11}$ indicates that atoms in the chains are strongly bound to each other, whereas the interaction between chains is much weaker, and is believed to be of Van der Waals character.[10] The application of hydrostatic pressure results in denser packing of the chains and relatively little deformation in the stiff chains. It is well known that with increased pressure Se eventually is transformed to the polonium structure at a pressure of 130 kbar.[11,12] This is in accord with the nature of the bonding of the VI group of the Periodic Table, as described by Martin, Lucovsky, and Helliwell,[4] Von Hippel,[9] Grosse,[13] and Gspan, Drope, and Grosse.[14] They show that progression in that group from sulfur toward polonium as well as the application of hydrostatic pressure promotes transfer of electrons from the intrachain bonding orbitals to bonding states between chains, resulting in eventual equalization of charges between these two types of bonds in the polonium structure. This effect was recently demonstrated theoretically by Joannopoulos, Schlüter, and Cohen,[15] who calculated the band structure of Se and Te and have shown how the interchain distance can affect the relative charge distribution between interchain and intrachain bonds. The effect of pressure to transfer charge from intrachain to interchain orbitals causes the interchain force constant to stiffen rapidly at the expense of the intrachain force constant. As mentioned above, this is manifested by a faster rate of increase with pressure of the elastic constant C_{11} in comparison with C_{33}.

The effect of pressure on the elastic properties of a-Se was measured by several authors,[16-18] but their studies were limited to the 1-atm range. Bridgman[19] and Singh and Kennedy[20] have measured the compression of a-Se up to 50 kbar, but under nonhydrostatic pressure conditions. Also, their resolution was not fine enough to allow a detailed study of the most compressible range between 0 and 10 kbar. Weir's[21] measurements of compression up to 10 kbar were under hydrostatic conditions, but his results appear to be in error. Through the use of a liquid cell and ultrasonic techniques, we were able to study directly and with high resolution the elastic properties of a-Se as a function of hydrostatic pressure up to 16 kbar. The experimental procedures are discussed in Sec. II. Results, discussion, and conclusion follow in Sec. III, IV, and V, respectively.

II. EXPERIMENT

Selenium-glass samples were prepared by melting selenium (99.99% purity from Ventron, Alfa Products) under vacuum in Pyrex tubes. The tube was heated to 400–600 °C for 2–5 h, while continuously rotated to ensure homogeneity. The glass was formed by quenching the tube in air. For the ultrasonic experiments, samples of 6.3 mm diameter and 5.0 mm length were cut and their two basal surfaces were polished and made parallel within 0.0001 in. The density of the samples was determined by the Archimedes method with a microbalance. Toluene was used as a liquid, in order to prevent air bubbles from adhering to the sample. The density was 4.278 g/cm^3.

The sound velocity was measured using the McSkimin pulse superposition technique, recently automated by Litov.[22] All measurements were carried out with 10-MHz X-cut or Y-cut 6.3-mm-diam quartz transducers from Valpey. The transducers were bonded to the samples with a glycerine-phthalicanhydride mixture glue. The glue was prepared by mixing 1M glycerine and 1M phlthalicanhydride which was then heated to 130 °C until it appeared to be completely transparent.

The pressure dependence of the sound velocities was measured with an end-loaded piston cylinder apparatus described by Hargarth, Getting, and Kennedy.[23] The measurements were carried out in a liquid cell configuration using Bridgman unsupported area seals, as shown in Fig. 1. A liquid mixture of 1 pentane–1 isopentane was chosen as a pressure transmitting medium, since it did not dissolve the glycerine-phthalicanhydride glue. The liquid mixture is known to freeze at pressures of 32 kbar, which is above the pressure range used in the present work. The seal for the lead wires was a combination of tooth cement (Kerr "Smile"), epoxy (Epon 815) and an elastometer coating (Scotch-Clad 776). With this technique several reliable runs could be carried out without any leaks.

Pressure changes were measured with a 50-Ω manganin wire gauge wrapped around the sample holder. The gauge resistance was measured to high precision (1:10^4) with a four-wire-leads technique using L&N K-3 potentiometer. The applied pressure was determined from the hydraulic ram pressure and the ratio of areas of the ram and the high-pressure piston. The hydraulic pressure was read on the Heise gauge, which has a resolution of better than 0.1%. This corresponds to a resolution of the sample pressure of 0.037 kbar. The manganin gauge was calibrated against the Heise gauge. However, due to piston friction the manganin values had to be averaged between compression and decompression cycles. The total uncertainty in the sample pressure was estimated to be ±0.1 kbar. The pulse repetition frequency was automatically monitored as the pressure was changed.

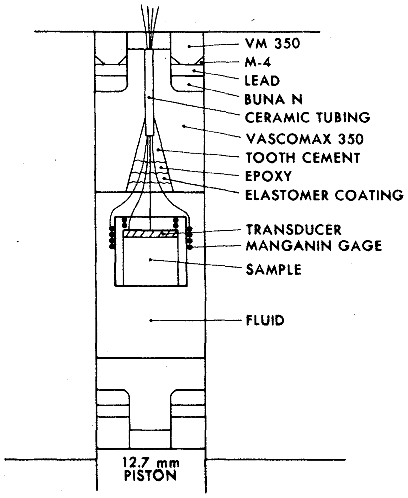

FIG. 1. High-pressure fluid cell.

III. RESULTS

The elastic constants from which the bulk modulus is calculated for a-Se are C^l and C^t, where l is longitudinal waves and t is transverse waves. For isotropic materials the bulk modulus K is

$$K = C^l - \tfrac{4}{3}C^t \ . \tag{1}$$

The pressure dependence of the adiabatic elastic constants C_s is governed by the expression

$$C_s = C_{0s}\frac{\rho v^2}{\rho_0 v_0^2} = C_{0s}\frac{\rho}{\rho_0}\left(\frac{fl}{f_0 l_0}\right)^2 , \tag{2}$$

where ρ is the density, l is the acoustical path length, and f is the pulse repetition rate. The zero subscripts in Eq. (2) refer to 1-bar values of the various quantities; the nonsubscripted quantities represent the values at finite pressure. The variations in ρ/ρ_0 and l/l_0 with pressure were taken into account following Cook's method.[24] Define $s = l_0/l$, then

$$\rho = \rho_0 s^3 , \tag{3}$$

$$v = 2fl = 2fl_0/s , \tag{4}$$

and, from (2),

$$C_s = C_{0s}(f/f_0)^2 s \ . \qquad (5)$$

Substituting Eq. (5) into Eq. (1) gives

$$K_s = \left[C^l_{0s}\left(\frac{f^l}{f^l_0}\right)^2 - \tfrac{4}{3}C^t_{0s}\left(\frac{f^t}{f^t_0}\right)^2\right] s \ . \qquad (6)$$

In Eq. (6), f^l, f^t, C^l_{0s}, and C^t_{0s} are measurable quantities. However, s, which is the measure of the sample's compression, cannot be measured directly, but can be evaluated in terms of K_T, where K_T^{-1} is isothermal compressibility. This is done by using the relationship between K_s and K_T,

$$K_s = (1 + \alpha\gamma T)K_T = (1 + \Delta)K_T \ , \qquad (7)$$

where α is the coefficient of expansion, γ is the Grüneisen constant, T is temperature, and $\Delta = \alpha\gamma T$. At room temperature and 1-bar pressure the value of Δ for a-Se is $\Delta = 0.033$. Within our experimental range and uncertainty, Δ was assumed to be independent of pressure. Substituting Eq. (7) into (6) yields the desired relationship

$$K_T = \frac{1}{1+\Delta}\left[C^l_{0s}\left(\frac{f^l}{f^l_0}\right)^2 - \tfrac{4}{3}C^t_{0s}\left(\frac{f^t}{f^t_0}\right)^2\right] s = \kappa s \ , \qquad (8a)$$

where

$$\kappa = \left[C^l_{0s}\left(\frac{f^l}{f^l_0}\right)^2 - \tfrac{4}{3}C^t_{0s}\left(\frac{f^t}{f^t_0}\right)^2\right]\frac{1}{1+\Delta} \ . \qquad (8b)$$

For isotropic solids it is easily shown that

$$K_T = \tfrac{1}{3}s\frac{dp}{ds} \ . \qquad (9)$$

Combining Eqs. (8) and (9) we get, after integration,

$$s = 1 + \frac{1}{3}\int_0^p \frac{dp}{\kappa} \ . \qquad (10)$$

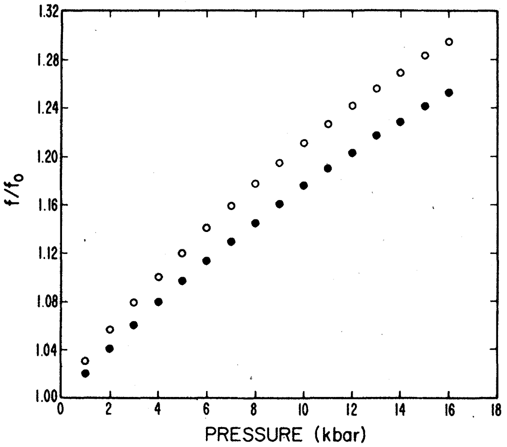

FIG. 2. Reduced-pulse repetition rate f/f_0 vs p for longitudinal waves (open circles) and transverse waves (filled circles).

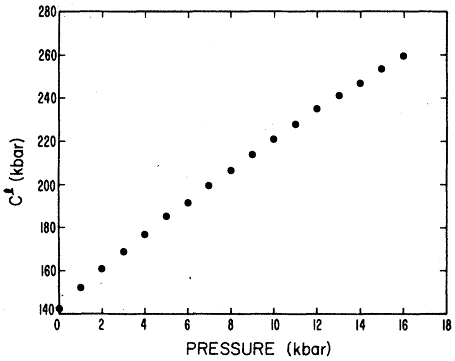

FIG. 3. Elastic constant C^l of a-Se vs p.

Since κ is measureable quantity, s can now be evaluated and substituted in Eq. (8) to give K_T.

Figure 2 shows the reduced pressure dependence of f^l and f^t uncorrected for variations in path length l and density ρ. Using Eqs. (5) and (10) the longitudinal and shear-wave elastic constants C^l and C^t were calculated, and the results are shown in Figs. 3 and 4. The bulk modulus K_T was calculated using Eqs. (8) and (10), and the results are shown in Fig. 5 and numercal values in Table I. Since the pressure dependence of K_T essentially embodies all the elastic properties, we will concentrate our discussion on it. It is noted in Fig. 5 that the initial slope $K_0 = \partial K \ \partial p|_0 = 8.0$ is relatively high compared to many solids, and decreases rapidly to a value below 4.0 around 10 kbar. The 1-bar value of $K_{T_0} = 91$ is also relatively low, indicating that a-Se is a very compressible substance. Our 1-bar K_T and K_T' values are in excellent agreement with several previous works, as shown in Table II. Since compressional data by other authors is mostly available in term of compression $v = V/V_0$, we can conveniently compare our results by plotting $v = 1/s^3$ vs pressure. The results are shown in Fig. 6, and numerical values in Table I. For comparison with other works, we have super-

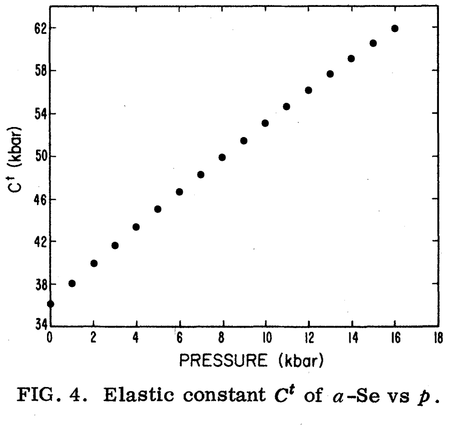

FIG. 4. Elastic constant C^t of a-Se vs p.

TABLE I. Bulk modulus and volume compression of amorphous selenium as a function of pressure; $v(p) = 1/s^3$.

p (kbar)	$K(p)$	$v(p)$
0	91.07	1.0000
1	98.11	0.9895
2	104.02	0.9797
3	109.1	0.9706
4	114.0	0.9619
5	118.7	0.9537
6	123.7	0.9459
7	128.3	0.9384
8	132.9	0.9312
9	137.4	0.9243
10	141.6	0.9177
11	145.9	0.9114
12	149.8	0.9053
13	153.5	0.8993
14	157.1	0.8935
15	160.9	0.8879
16	164.5	0.8825

imposed the results of Bridgman,[19] Singh and Kennedy,[20] and Weir.[21] It is noted that our results basically follow Bridgman and Singh and Kennedy up to 12 kbar but remain slightly lower above that pressure. Weir's V/V_0 values are much smaller than those reported in the present work and those of Bridgman and Singh, and appear to be in error. Regression analyses for both v vs p and K_T vs p were carried out up to the fifth power in the polynomial, and the various coefficients are presented in Table III.

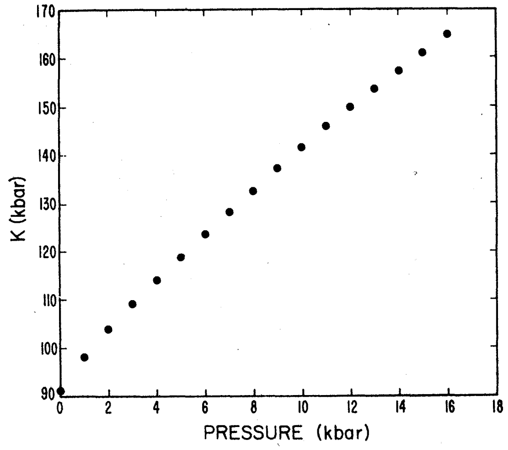

FIG. 5. Bulk modulus K_T of a-Se vs p.

TABLE II. K_{T_0} and K'_{T_0} for amorphous selenium.

K_{T_0} (kbar)	K'_{T_0}	Technique	Ref.
91.1	···	ultrasonic	17
91.4	···	ultrasonic	18
91.6	8.5	ultrasonic	16
92.9	3.2	static	19
67.8	7.4	static	21
87.3	7.07	static	20
91.07	8.01	ultrasonic	This work

IV. DISCUSSION

The analysis of the pressure dependence of the bulk modulus K_T in terms of an interatomic potential will now be discussed. It has long been recognized by Von Hippel[9] and Vedam, Miller, and Roy,[10] that in amorphous as well as trigonal Se, bonding forces between chains and rings are mostly of the Van der Waals type. Their conclusions were based on the fact that interchain distances of 3.5–3.8 Å were close to the Van der Waals diameter for Se, which is 4.00 Å. However, with increased pressure, the promotion of charge from the intrachain orbitals toward interchain orbitals will increase the attractive interchain forces, thus lowering the total energy. This effect appears to be a very important one, particularly at high pressures where, as we shall later show, it is responsible for destabilizing the structure, leading to a phase transformation. Hence it must be included in our analysis for the correct description of K. Since the major effect of pressure is to decrease the interchain distance R,[11] we have assumed in analyzing the data, that the total potential energy ϕ is simply a function of R, excluding any dependence

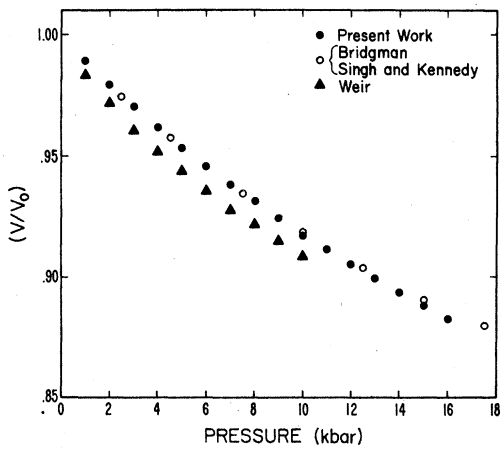

FIG. 6. Compression of a-Se vs p.

TABLE III. Coefficients of fifth-degree polynomials for v and K as a function of pressure p. Data used from Table I.

Coefficient	v	K
a_0	0.999 97	91.298
a_1	−0.010 83	7.392 2
a_2	$0.402\,91 \times 10^{-3}$	−0.718 2
a_3	$-0.212\,01 \times 10^{-4}$	$0.951\,8 \times 10^{-1}$
a_4	$0.874\,9 \times 10^{-6}$	$-0.611\,77 \times 10^{-2}$
a_5	-0.169×10^{-7}	$0.143\,10 \times 10^{-3}$

on the intrachain distance r. However, since we are dealing here with an amorphous structure, in which there is a statistical fluctuation of R around $\bar{R}$ and since hydrostatic pressure basically senses the average distance $\bar{R}$, we will further assume that ϕ depends only on $\bar{R}$.

We first demonstrate that the Van der Waals potential

$$\phi(\bar{R}) = A/\bar{R}^m - B/\bar{R}^n , \tag{11}$$

where A and B are constants, $m=12$ and $n=6$, is applicable only at zero pressure range. It is easily shown that the bulk modulus K and its first *pressure* derivative K' as derived from Eq. (11), are

$$K = \frac{mA}{3V_0\bar{R}_0^m}\left[\frac{m+3}{3}\left(\frac{\rho}{\rho_0}\right)^{(m+3)/3} - \frac{n+3}{3}\left(\frac{\rho}{\rho_0}\right)^{(n+3)/3}\right], \tag{12}$$

$$K' = \frac{1}{K}\left\{\frac{mA}{3V_0\bar{R}_0^m}\left[\left(\frac{m+3}{3}\right)^2\left(\frac{\rho}{\rho_0}\right)^{(m+3)/3} - \left(\frac{n+3}{3}\right)^2\left(\frac{\rho}{\rho_0}\right)^{(n+3)/3}\right]\right\}, \tag{13}$$

where ρ is the density. A plot of Eq. (12) is shown in Fig. 7, where it is seen to deviate considerably from the data above zero-pressure range. Nonetheless, at zero-pressure range the Van der Waals potential describes a-Se exactly, as can be seen from the following analysis of Eqs. (12) and (13). In the limit of $\rho \to \rho_0$, we get

$$K_0 = ma(m-n) , \tag{14}$$

$$K_0K_0' = \tfrac{1}{3}ma[(m+3)^2 - (n+3)^2] , \tag{15}$$

where

$$a = \frac{1}{9V_0}\frac{A}{\bar{R}_0^m} = \frac{1}{9V_0}\frac{\phi(\bar{R}_0)}{1-(m/n)\bar{R}_0^n} . \tag{16}$$

Using Eqs. (14) and (15) to solve for K_0' we get

$$K_0' = [(m+3)^2 - (n+3)^2]/3(m-n) .$$

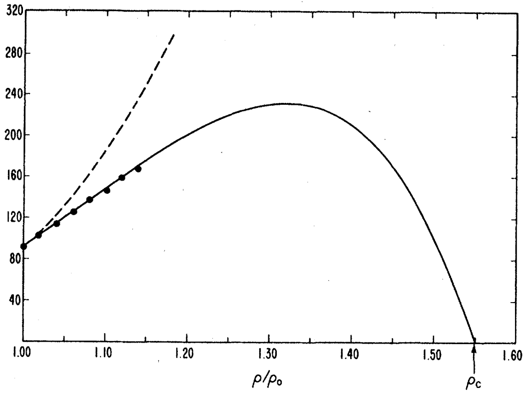

FIG. 7. Bulk modulus K_T of a-Se vs ρ/ρ_0. Broken curve is due to Van der Waals potential only, Eq. (13). Solid curve is due to total potential, Eq. (19). Arrow indicates the density at which the phase transformation occurs.

We see that K_0' depends only on the powers m and n and therefore can be used to test the validity of the Van der Waals potential at zero pressure. Letting $m=12$, $n=6$, we get $K_0'=8.0$, which is precisely what was experimentally found. When the value $K_0 = 91.0$ is substituted into Eq. (14), we find $a = 1.264$. It is seen from Eq. (16) that a is related to the depth of the potential ϕ at the equilibrium separation $\bar{R}_0$.

At higher pressures however, the Van der Waals potential, as expected, leads to a stiffer structure than is actually observed (see Fig. 7). Therefore, an attractive potential term must be included to take care of the charge redistribution effect from the intrachain to the interchain orbitals. The rate by which this process occurs with interchain distances is an extremely complicated quantum mechanical problem, and to our knowledge, has not previously been calculated. Martin, Fjeldy, and Richter[3] have pointed out that the nonbinding orbitals overlapping with antibonding states at different chains are the cause for weakening the intrachain bonds. Hence we have decided to introduce this term phenomenologically as being proportional to $\exp(-\lambda\bar{R})$, under the reasonable assumption that the wave function spatial dependence along the lone pair direction is asymptotically exponential. Adding this term to Eq. (11), the total potential takes the form

$$\phi(\bar{R}) = A/\bar{R}^m - B/\bar{R}^n - Ce^{-\lambda\bar{R}} , \tag{17}$$

where C is a constant. A very important feature of the potential $\phi(\bar{R})$ described by Eq. (17) is that it leads to a phase transformation with decreasing $\bar{R}$ (high pressures), see Fig. 8.

Assuming that Eq. (17) characterizes the major interatomic forces, we show below that the phase

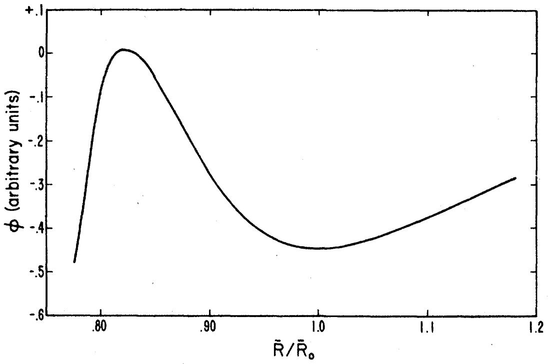

FIG. 8. Interatomic potential $\phi(\bar{R})$, Eq. (18), vs reduced interchain distance $\bar{R}/\bar{R}_0$; values of $\lambda = 21.9$ and $B = 0.6211$ were used, for details see text.

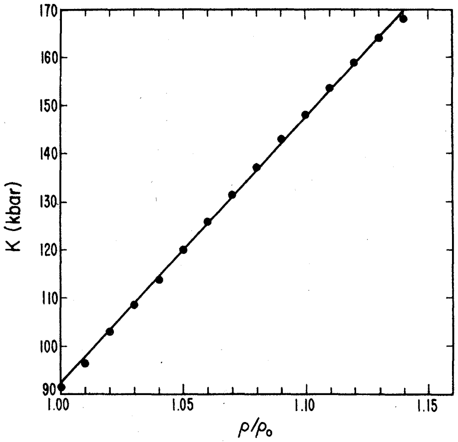

FIG. 9. Bulk modulus K_T of a-Se vs ρ/ρ_0. Curve represents Eq. (19).

transformation which it predicts corresponds to the well known transformation in a-Se at 130 kbar. This is done by analyzing the bulk modulus K_T derived from Eq. (17). Again, it is easy to show that K_T is given by

$$K_T = a[180x^5 - 54x^3 + (6 - 12B)x^{1/3} \times (2x^{1/3} + x)e^{\lambda(1-1/x^{1/3})}] \tag{18}$$

where $a = A/9V_0$, x is the reduced density, and C was eliminated by the equilibrium conditions. A phase-transformation boundary condition is imposed on Eq. (18) by requiring that the compressibility $\beta = K_T^{-1}$ is infinite at the transition density x_c. Using the McCann and Cartz[11] x-ray data, we find $x_c = 1.55$. These additional constraints on Eq. (18) leave it with only a normalizing constant a and the fitting parameter λ. Constant B is now related to x_c and λ through

$$B = [9x_c^3 - F(x_c;\lambda)]/2[15x_c^5 - F(x_c;\lambda)] \ , \tag{19a}$$

where

$$F(x_c;\lambda) = x_c^{1/3}(2x_c^{1/3} + \lambda)\exp[\lambda(1 - 1/x_c^{1/3})] \ . \tag{19b}$$

Equation (18) for K was fitted to our data and the results are shown in Figs. 7 and 9. The parameters which gave the best fit are $a = 4.019$ and $\lambda = 21.90$; B derived from Eqs. (19) is 0.6211. It is seen that the fit is excellent throughout the pressure range. Furthermore, it is noted that K characterizes the major elastic properties of a-Se at low as well as at high-pressure ranges. At the low pressure range of 1 kbar and up, K starts to deviate from a purely Lennard-Jones K dependence toward a softer structure, and shows a strong inclination for a phase transformation at about 60 kbar. Wendel, Martin, and Chadi[25] have also recently interpreted large pressure effects in the reflecting spectrum in Se by Kastner and Farberg[26] in the low-pressure range as being precursors of the transition of Se to a metallic nonmolecular structure. The McCann and Cartz[11] x-ray observations of the appearance of the first diffraction line in a-Se at 60 kbar is also an indication of an early sign of the transition at low pressures. Current electrical conductivity (σ) measurements by Gupta and Ruoff[27] also show that the transition begins at a pressure as low as 80 kbar, as indicated by the sudden drop of σ at that pressure. Further evidence for the anomalous pressure dependence of K comes from Bridgman's[19] compression data,

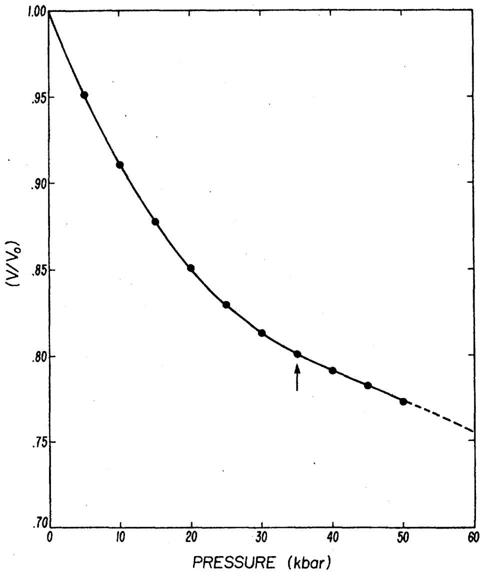

FIG. 10. Bridgman's plot of v vs p for a-Se. Arrow indicates inflection point.

see Fig. 10. He reports that around 34 kbar a very distinct inflection point in V/V_0 versus pressure is observed. Since K is inversely proportional to $\partial v/\partial p$, an inflection point $\partial^2 v/\partial^2 p = 0$ is translated in the K-versus-pressure plot as being the maximum in K. Equation (18) predicts such a maximum but at a much higher pressure, estimated to be around 70–80 kbar. Singh and Kennedy[20] did not find this point below 50 kbar. We believe, however, that with today's advanced high-pressure techniques, this maximum could be firmly established. It should be emphasized, in conclusion, that the potential $\phi(R)$, as described by Eq. (17), is only a reasonable approximation of the interacting forces, and in view of the available data seems to work well in the low-ressure range. A more exact spatial dependence in the high-pressure range will have to be worked out through quantum mechanics and verified when additional data above 50 kbar are available.

V. CONCLUSIONS

We have measured the bulk modulus K_T of amorphous selenium as a function of hydrostatic pressure, up to 16 kbar. We have shown that the data can be interpreted in terms of an interatomic potential, which consists of a Lennard-Jones term and an attractive term due to intrachain-interchain interaction. The new potential was found to characterize all the essential isothermal elastic properties of a-Se at low- as well as high-pressure ranges, and predicts the phase transformation at 130 kbar.

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