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Pressure-Induced Electronic Collapse and Structural Changes in Rare-Earth Monochalcogenides

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The compressibilities of the monotellurides of Pr, Sm, Eu, Tm, and Yb and the monoselenide and sulfide of Sm have been investigated to ~300 kbar using high-pressure x-ray-diffraction techniques. SmTe, SmSe, TmTe, and YbTe show abnormal volume changes in the 20–50-, 15–40-, 15–30-, and 150–200-kbar regions of pressure, respectively. SmS shows an abrupt decrease in volume at 6.5 kbar. Since there is no change in structure, the anomalously large volume changes have been explained on the basis of a pressure-induced $4f$-$5d$ electronic collapse which involves a change in the valence state of the rare-earth ion from 2^+ towards the 3^+ state. The results of high-pressure x-ray studies on Sm chalcogenides are consistent with the conclusions drawn in the earlier work from high-pressure resistivity measurements. PrTe, SmTe, and EuTe exhibit a phase transition from NaCl-type to CsCl-type structure at pressures of about 90 ± 10, 110 ± 10, and 110 ± 10 kbar, respectively. It appears that a pressure-induced NaCl-to-CsCl transition may be commonly encountered in rare-earth monochalcogenides.

INTRODUCTION

Rare-earth monochalcogenides have attracted much attention in recent years because of their interesting magnetic and electrical properties. They crystallize in the NaCl-type structure[1] and are semiconducting if the rare-earth ion is in the divalent state and metallic if trivalent.[2,3] Recent high-pressure resistivity studies[4–6] on Sm chalcogenides and TmTe revealed that these undergo a pressure-induced semiconductor-metal transition; the transition is found to be continuous in the case of SmTe, SmSe, and TmTe, while discontinuous in the case of SmS. This phenomenon was interpreted as due to the promotion of a $4f$ electron of the rare-earth ion into the $5d$ conduction-band states, as the energy separation between the localized $4f$ electronic state and the latter decreased with pressure. Such an electronic transition involves a change of the valence state of the rare-earth ion from divalent to a higher valence state tending towards trivalency. Since the ionic radius of the trivalent ion is substantially smaller than that of the corresponding divalent ion, the occurrence of $4f$-$5d$-electron promotion should be reflected in the pressure-volume behavior, and hence the pressure-volume relationship should provide conclusive evidence for $4f$-$5d$-electron promotion. Therefore, we undertook a high-pressure x-ray study of a number of rare-earth monochalcogenides of interest in this connection. The results will be presented and discussed in this paper.

EXPERIMENTS AND RESULTS

The pressure-volume data up to nearly 300 kbar were obtained from lattice-parameter measurements, using a diamond-anvil high-pressure x-ray camera.[7] In the pressure range 1–50 kbar, measurements were also made using the McWhan-Bond high-pressure camera.[8] Pressure was estimated using NaCl or Ag as an internal standard. At pressures above 50 kbar, the diffraction lines from NaCl became too weak when using the diamon-anvil camera, due to the extrusion of the salt from the center of the anvil. However, silver proved quite satisfactory at higher pressures. The theoretical equation of state[9] for NaCl and the experimental equation of state[10] for silver have been used for converting the volume to pressure. Since the specimen under investigation contains elements of high atomic number (high x-ray-scattering factor), sharp and intense Debye-Scherrer lines could be obtained even at the highest pressure (~ 300 kbar) within reasonable exposure times (~ 30 h), using a fine-focus x-ray generator with Mo target.

The samples used in the present investigation are from the lots used in earlier studies.[4-6,11] The lattice parameters of the compounds used in the present study are given in Table I. These agree well with the published values. The errors in the determination of the volume and pressure are estimated to be 1 and ~ 5%, respectively.

SmTe, SmSe, and SmS

The $(\Delta V/V_0)$-vs-P curves for SmTe,[12] SmSe, and SmS are shown in Figs. 1–3, respectively. In the case of SmTe, the compressibility appears to be normal in the 1–20-kbar range but becomes anomalously large in the 20–60-kbar range (usually the compressibility decreases with pressure). No structure change is associated with this anomalously large volume compression; the NaCl-type structure is retained. The compressibility markedly decreases beyond 60 kbar and exhibits normal behavior. At about 110 kbar a discontinuity occurs due to a phase transformation from the NaCl- to CsCl-type structure (this will be discussed later). The anomalously large volume compression without change of structure reveals that an electronic collapse is occurring in the 30–60-kbar region. This is precisely the behavior that is to be expected if a continuous 4f-5d–electron promotion took place in this pressure region.

In a high-pressure x-ray-diffraction study[13] of SmTe, Rooymans reported a discontinuous isostructural transition near 40 kbar, with 15% volume decrease, which he attributed to a valence transition of the Sm^{2+} ion to the trivalent state. The results of the present study, however, show that the electronic transition is not discontinuous, and this is consistent with the earlier resistivity measurements under pressure.[4]

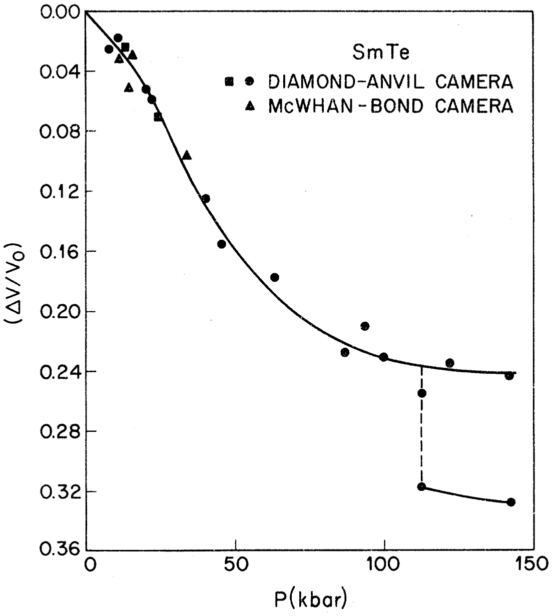

FIG. 1. Pressure-volume data for SmTe.

TABLE I. Lattice parameters of rare-earth chalcogenides studied.

Compounds	Lattice parameter[a] (Å)	Ref. 1
PrTe	6.315 ± 0.001	6.322
SmS	5.974 ± 0.001	5.970
SmSe	6.223 ± 0.001	6.200
SmTe	6.595 ± 0.001	6.594
EuTe	6.591 ± 0.001	6.585
TmTe	6.337 ± 0.001	6.338[b]
YbTe	6.361 ± 0.001	6.361

[a] The diffraction patterns were recorded with a 114-mm camera using Cu$K\alpha$ radiation. The lattice parameters were obtained from Nelson-Riley plots.

[b] A. Iandelli and A. Palenzona, *Propriêtês des dêrivês semimetalliques* (Centre Nationale de la Recherche Scientifique, Paris, 1967), p. 397.

In Fig. 2 the pressure-volume relationship for SmSe is shown. The behavior is similar to that of SmTe except that the anomalous region of compression is somewhat steeper than in SmTe and the transition is essentially complete by ~ 45 kbar pressure. This behavior is again in good agreement with the reported logarithmic decrease in resistivity with pressure up to about 40 kbar followed by saturation at higher pressures.

The pressure-volume data for SmS (Fig. 3) indicate an abrupt volume change near 6.5 kbar without any change in structure. This is in excellent accord with the abrupt drop in resistivity at 6.5 kbar.

EuTe

The pressure-volume relationship for EuTe is shown in Fig. 4. Unlike SmTe the behavior reflects normal compressibility and there is no region of anomalous volume decrease up to about 100 kbar. This is at variance with the results of an earlier high-pressure x-ray study by Rooymans[13] on EuTe. Rooymans reported a discontinuous isostructural transition near 30 kbar and attributed the same to a valence transition of the Eu^{2+} ion to the trivalent state. The results of the present study rule out the

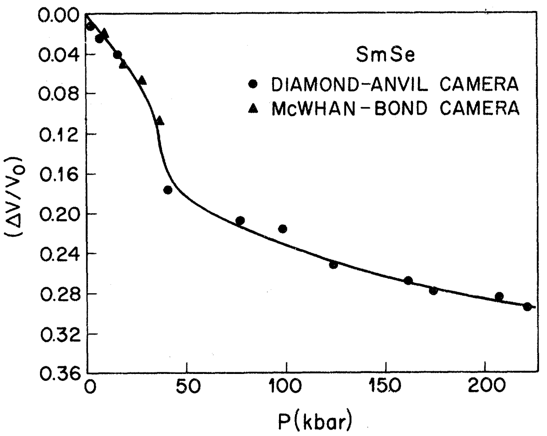

FIG. 2. Pressure-volume data for SmSe.

occurrence of any electronic collapse in the 1–100-kbar region. The discontinuity near 110 kbar is due to a transition from the NaCl- to CsCl-type structure.

TmTe

Among the Tm monochalcogenides, TmTe has an anomalously large lattice constant and is found to be semiconducting. The magnetic-susceptibility measurements, the lattice parameter, and the conductivity data confirm that the Tm ion is in the divalent state.[14] Earlier high-pressure resistivity and preliminary pressure-volume measurements[6] suggested the occurrence of 4*f*-5*d* electronic collapse in TmTe. In the present study the pressure-volume relationship was determined to about 125-kbar pressure and the data are shown in Fig. 5. The compression curve is similar to that of SmSe and provides strong evidence for the occurrence of a continuous 4*f*-5*d* electronic collapse in which the Tm^{2+} ion changes to a higher valence state. Near about 150 kbar, TmTe seems to undergo a structure change and the high-pressure phase appears to have a distorted NaCl-type structure.

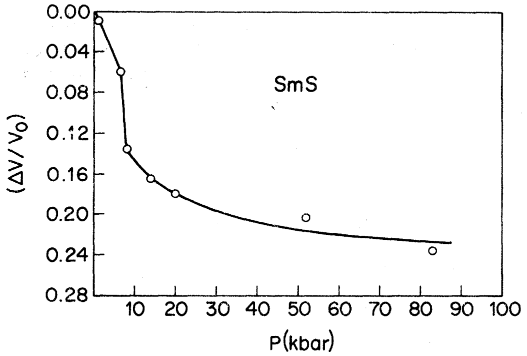

FIG. 3. Pressure-volume data for SmS. Below 20 kbar the compression curve was determined directly in a piston cylinder device. The data points above 20 kbar are from diamond-anvil camera.

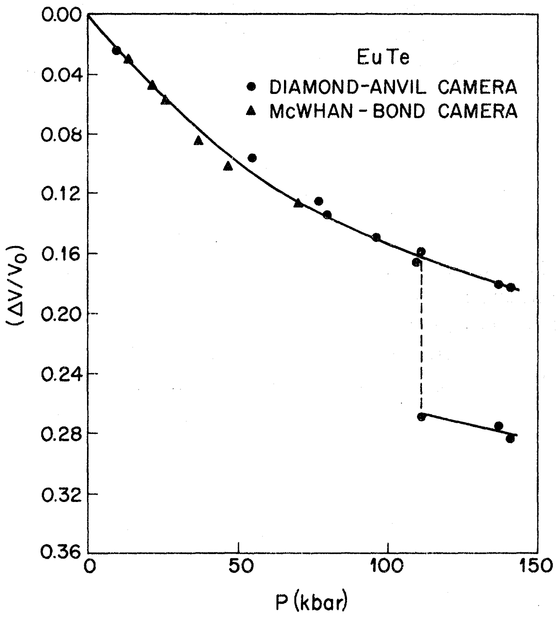

FIG. 4. Pressure-volume data for EuTe.

YbTe

The pressure-volume relationship for YbTe (Fig. 6) was reported[15] by us recently. The initial compressibility of YbTe is about the same as that of EuTe and SmTe. The compression curve is anomalous in the 150–200-kbar region. This is attributed to 4*f*-5*d* electronic collapse. Ytterbium telluride does not show any other phase change up to 300 kbar, the limit of the present study.

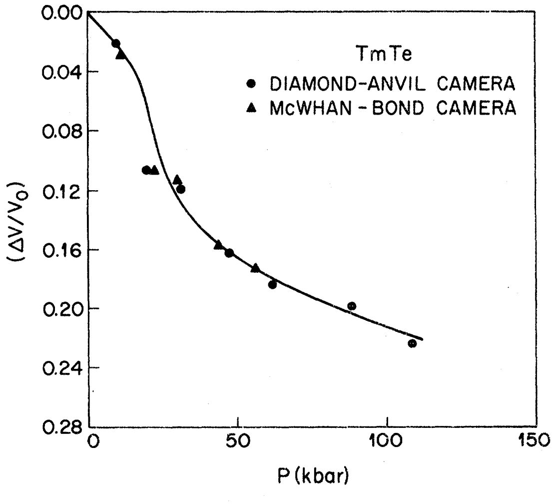

FIG. 5. Pressure-volume data for TmTe.

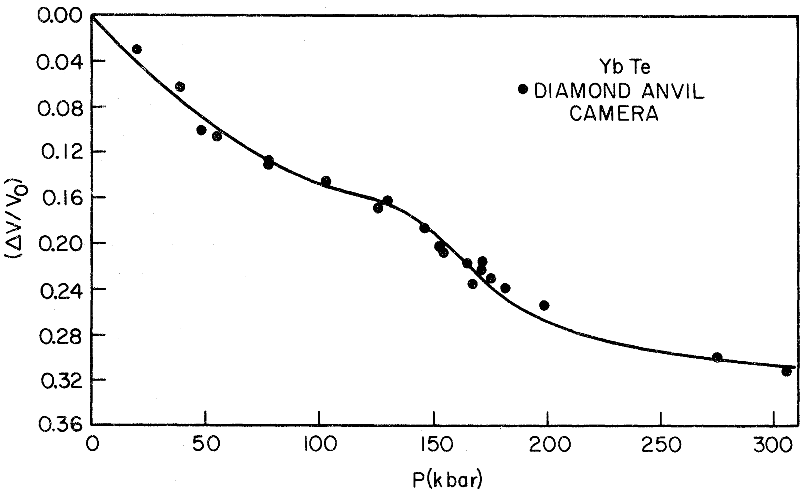

FIG. 6. Pressure-volume data for YbTe.

NaCl-CsCl Transition

Out of the seven substances investigated, SmTe, EuTe, and PrTe exhibit a crystallographic transformation from the NaCl- to CsCl-type structure. The volume discontinuity at 110 ± 10 kbar for SmTe and EuTe and at about 90 ± 10 kbar for PrTe is due to the above transition. In all the three substances, diffraction lines from both phases coexisted over a pressure range. Weak lines from CsCl phase first appear at the pressures quoted above and these grow strong as the pressure is increased. On further increase of the pressure, the lines from the NaCl phase completely vanish, leaving only the lines from the CsCl phase. In the compression curves of SmTe and EuTe the data of both phases are shown for the region of pressure where they coexist. Pertinent data on the NaCl-CsCl transition are collected in Table II. The volume changes associated with these transitions are −11.5, −9.1, and −13.2% for PrTe, SmTe, and EuTe, respectively. The volume change for EuTe is significantly higher than that for SmTe. This may possibly be due to a fractional change in the valence state of the Eu ion. The crystallographic data for the CsCl phase of SmTe are given in Table III. The integrated intensities of the various reflexions were obtained from the microphotometer records. These were corrected for the absorption in the diamond anvil. The calculated intensities were corrected for the Lorentz-polarization factor. The absorption of radiation in the sample was ignored as the thickness of the sample was less than 0.02 mm. The agreement between the observed and the cal-

TABLE II. Data pertaining to the NaCl-CsCl transformation in PrTe, SmTe, and EuTe.

	PrTe	SmTe	EuTe
Lattice constant of NaCl phase at atmospheric pressure	6.315 ± 0.002 Å	6.595 ± 0.002 Å	6.591 ± 0.002 Å
Lattice constant (a_1) of the NaCl phase at the pressure of NaCl-CsCl transition[a]	6.218 ± 0.02 Å	5.992 ± 0.02 Å	6.222 ± 0.02 Å
Lattice constant (a_2) of CsCl phase at the pressure of NaCl-CsCl transition[a]	3.761 ± 0.01 Å	3.656 ± 0.01 Å	3.739 ± 0.01 Å
$\Delta V/V$ associated with the NaCl-CsCl transition[a] $\Delta V/V = (4a_2^3 - a_1^3)/a_1^3$	−11.5%	−9.1%	−13.2%
The pressure of NaCl-CsCl transition (kbar)	90 ± 10	110 ± 10	110 ± 10

[a] These values were obtained from x-ray photographs which showed diffraction lines from both the NaCl and CsCl phases.

TABLE III. Crystallographic data on CsCl phase of SmTe.

hkl	*d*	I_{obs}	I_{calc}
110	2.564	100.0	100.0
200	1.818	8.7	19.2
211	1.482	31.5	41.0
220	1.284	9.7	12.7
310	1.149	13.4	17.7
222	1.048	7.6	4.4
321	0.971	18.3	18.3

culated intensities appears good. Similar results were obtained for the high-pressure phase of PrTe and EuTe.

DISCUSSION

The compression data for SmS, SmSe, SmTe, TmTe, and YbTe exhibit regions of anomalous compression without showing any structure change. This reflects a collapse in the atomic volume, which is usually associated with a change in the electronic structure of the ions involved. In divalent rare-earth monochalcogenides it is the rare-earth ion which undergoes a change in its electronic structure due to $4f$-$5d$ electron promotion, resulting in a higher valence state tending towards trivalency. Since the trivalent radii are strikingly smaller compared to the corresponding divalent radius, the atomic volume decreases and the observed decrease is, in fact, a direct measure of the valence change. However, the volume change associated with the electronic transition would be superimposed on the normal compressibility of the material, but it could be estimated by fitting the high-pressure region of the compression curve to an expression of the form

$$(\Delta V/V_0) = m_0 + m_1 P + m_2 P^2 . \qquad (1)$$

In the above expression, m_0 represents that part of $(\Delta V/V_0)$ associated with the electronic transition and its value is obtained by extrapolating the compression curve of the electronically collapsed phase described by the above quadratic expression to atmospheric pressure. For SmSe, SmTe, TmTe, and YbTe the high-pressure regions thus fitted were 80–120, 100–140, 60–100, and 220–280 kbar, respectively. The $(\Delta V/V_0)_{\text{elec}}$ values obtained by the above-mentioned procedure are given in Table IV and these are compared with the calculated values from the ionic radii[1] of the respective rare-earth ions in their trivalent state. The calculated values, although are near to the observed, appear to be consistently somewhat larger. This suggests that the rare-earth ions in the collapsed state have not become fully trivalent. This, in fact, supports the suggestions of Maple and Wohlleben[18] that in the high-pressure phase of SmS the Sm ion has an intermediate valence. The latter authors measured the magnetic susceptibility of SmS under pressure down to liquid-He temperatures and found no evidence for magnetic ordering nor any large temperature effect on the susceptibility. From these results, they suggested that in the collapsed phase of SmS the $4f$ shell is fractionally occupied and that the conduction band has 0.7 electrons per molecule of SmS. It is argued that this fractional occupation of the $4f$ shell would result in a fluctuating magnetic moment which in the limit $T = 0$ would be equivalent to a nonmagnetic state in the Friedel-Anderson model. Also in TmTe, magnetic-susceptibility measurements under pressure[19] appear to suggest an intermediate valence state for the Tm ion. The pressure-volume data obtained in the present study appear to suggest intermediate valence for the rare-earth ion in all the compounds studied. In Table IV, in the last column, the intermediate valences computed from x-ray data are shown. In Table V the compressibilities are listed.

In the examples where the transition is continuous with pressure, the region of anomalous volume decrease due to electronic collapse can be evaluated using the simple model proposed in Ref. 4 and compared with the observed pressure-volume data. According to this model, the $4f$ electrons are promoted into the $5d$ conduction band, as the energy separation between the two decreases with pressure. When a $4f$ electron is promoted, an Sm ion is converted into the 3^+ state and hence the number of trivalent ions would be given by

TABLE IV. Parameters associated with electronic transitions.

Compounds	Energy gap (eV)	α (meV/kbar)	Mode of transition	Pressure range (kbar)	$(\Delta V/V_0)_{\text{elec}}$ (obs.)	$(\Delta V/V_0)_{\text{elec}}$ (calc.)	Intermediate valence
SmS	~0.40	···	Discontinuous	at 6.5	13 (± 1.0)	15.9	2.8
SmSe	0.46[a]	12	Continuous	1–50	13 (± 1.0)	17.0	2.76
SmTe	0.63[a]	11.9[a]	Continuous	1–60	11 (± 1.0)	16.6	2.66
TmTe		10.0[b]	Continuous	1–40	10 (± 1.0)	14.3	2.70
YbTe	2.0[c]	11.0[d]	Continuous	1–190	13 (± 1.0)	16.2	2.8

[a] Reference 4. [b] Reference 11. [c] Reference 16. [d] Reference 17.

TABLE V. Initial compressibilities (insulator phases) from data taken in the 1–10-kbar region except for SmS which is from data in the 1–5-kbar region.

	$K(10^{-4}/\mathrm{kbar})$
SmS	66
SmSe	25
SmTe	25
TmTe	22
YbTe	22

$$N^{3+} = N_0\, e^{-E_g(P)/kT} , \qquad (2)$$

where N_0 is the total number of rare-earth ions, and $E_g(P)$ is the pressure-dependent energy gap between the 4f level and the 5d band. Shifts in the optical-absorption edge with pressure have shown that in Eu and Sm chalcogenides,[20,21] the energy gap decreases with pressure. The energy gap of YbTe also decreases with pressure[17] with a similar pressure coefficient. Using this data and the energy separation between the 4f and 5d states (see Table IV), the change in volume associated with the divalent to trivalent state can be estimated using the equation

$$(\Delta V/V_0)_{\mathrm{elec}} = [1 + (\Delta a/a_0)]^3 - 1 ,$$

where

$$\Delta a = 2(r^{+++} - r^{++})\, e^{-(E_0 - \alpha P)/kT} ; \qquad (3)$$

r^{++} and r^{+++} are the ionic radii of the rare-earth ions, E_0 and a_0 are the lattice parameter and the energy gap at atmospheric pressure, and α is the rate at which E_0 closes with pressure. The region of electronic collapse predicted by the above equation is in reasonable agreement with what is experimentally observed. The true compression curve can be obtained by adding to $(\Delta V/V_0)_{\mathrm{elec}}$ the normal volume compression. The model, however, predicts a sharper drop in the $(\Delta V/V_0)$-vs-P curve than observed in many cases. We have assumed a linear decrease of the energy gap with pressure. This may, however, not be strictly correct. It may be remarked that Eq. (2) is no longer valid after the energy gap between the 4f state and the conduction-band edge becomes zero.

METAL-SEMICONDUCTOR TRANSITION

The electronic phase transitions reported in this paper involve large changes in the electrical conductivity (the resistivity of YbTe has not, however, been studied to very high pressures) and the Sm chalcogenides in their high-pressure phases exhibit metallic conductivity. It has been shown recently that the high-pressure phases of SmS, SmSe, and SmTe show metallic luster[21]; SmS exhibits a golden yellow metallic reflectivity, SmSe a copperlike metallic luster, and SmTe a deep purple specular reflection. The high-pressure phases of the Sm monochalcogenides exhibit electrical conductivities of $10^{-4}\ \Omega$ cm. In the electronic transition a change from semiconducting to metallic behavior is to be expected since approximately one electron per molecule is released when the divalent rare-earth ion becomes trivalent. What is most surprising and interesting is the difference in the mode of occurrence of this transition. In SmS the semiconductor-to-metal transition is discontinuous, while in SmTe, SmSe, and TmTe it appears to take place continuously.

Bucher and Maines[22] have recently studied the system $\mathrm{SmS}_{1-x}\mathrm{Se}_x$ by high-pressure resistivity measurements and have found that the electronic transition goes from discontinuous to continuous smoothly with composition. The transition is discontinuous in alloys with $x < 0.8$ and continuous for $x > 0.8$. Further, the resistance drop associated with the transition appears to diminish in magnitude progressively, until the critical concentration of Se is reached.

While the model discussed in Ref. 4 is applicable in the case of continuous electronic collapse with pressure, the cooperative nature of the transition in SmS and $\mathrm{SmS}_{1-x}\mathrm{Se}_x$ alloys is difficult to understand on this simple model. Falicov and Kimball[23] and Ramirez *et al.*[24] have proposed a model for metal-semiconductor transitions involving a localized state and a conduction band separated by an energy gap. According to this model, metal-semiconductor transitions can be continuous or discontinuous depending on the strength of the electron-hole interaction energy in relation to the gap energy. However, in this model temperature is taken to be the free variable. Bucher and Maines have modified this model for volume rather than the temperature as the free variable and have demonstrated its applicability to the case of $\mathrm{SmS}_{1-x}\mathrm{Se}_x$ alloys. The work of Bucher and Maines suggests critical behavior of the transition with composition.

For SmSe and SmTe, Davis[25] has recently suggested that the semiconductor-metal transition is due to a continuous shrinking of the gap between the valence band and the conduction band and eventual overlap at high pressure. However, the pressure-volume data presented in this paper conclusively show that the valence transformation takes place in the region where the semiconductor-to-metal transition is seen and a simple continuous band overlap is not expected to give an anomalous compression curve. Further, optical-absorption features for SmSe and SmTe[26] are positively identified with transitions from the 4f state to the 5d states in the conduction band and therefore the valence band lies definitely below the localized 4*f* state.

In summary, the pressure-volume studies have conclusively proved the occurrence of 4*f*-5*d* electron delocalization in these rare-earth monochalcogenides and have also shown that the NaCl-CsCl transitions may be of common occurrence in these systems.

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