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Physica B 304 (2001) 221–227

PHYSICA B

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Pressure-induced phase transitions in bulk $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$

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Received 16 November 2000; received in revised form 22 February 2001; accepted 15 March 2001

Abstract

Energy-dispersive X-ray-diffraction (EDXD) is used to study the pressure-induced transitions of $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ bulk samples, $x = 0.016, 0.026, 0.053, 0.07,$ and 0.24 , below 30 GPa. The EDXD results show that possible structure transitions from the zinc blende (B3) to the sodium chloride phase (B1) for $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$, $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$, $\text{Zn}_{0.947}\text{Mn}_{0.053}\text{Se}$, $\text{Zn}_{0.93}\text{Mn}_{0.07}\text{Se}$, and $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$ occur at 13.1, 12.4, 12.0, 11.8, and 9.6 GPa, respectively. The unloading run (the measurement with decreasing pressure) reveals that a reversible phase transition exists in the bulk $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$. In this work, our EDXD data show that the larger the increase of the fractional volume change at the phase transition from the B3 to the B1 region, the larger is the decrease of the reduction of the semiconductor-metal phase transition pressure. © 2001 Elsevier Science B.V. All rights reserved.

PACS: 62.50.+p; 64.60. - i; 78.30.Fs

Keywords: Pressure-induced phase transitions; $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$; EDXD

ZnSe-based ternary compound semiconductors have attracted much attention due to their potential applications in optical devices, such as blue semiconductor injection lasers, blue-light emitting diodes, and flat-panel displays, and so on. The last two devices are fabricated by some layered ZnSe to form a number of multiple quantum wells and superlattice structures. The semimagnetic semiconductor $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ is a direct band-gap semiconductor with a band gap of 2.67 eV at room temperature. It has the ability to be one of the candidates for optoelectronic devices in the blue-light region of visible spectrum. The presence of the Mn ions has been reported to have many

interesting physical properties, such as tunable band gap [1,2] and can provide a highly useful feature to the superlattice and multiple quantum well [1]. Recently, the crystal structures, electronic and phonon properties of wide band gap II–VI compound dilute magnetic semiconductors (DMS) under high pressure have been extensively investigated by using the powerful photoluminescence tools [3], absorption spectra [4], dielectric permittivity and conductivity [5], micro-raman [6], and energy dispersive X-ray diffraction (EDXD) [7,8]. By measuring the transmission spectroscopy, Ves et al. [9] reported that the reduction of the phase transition pressure of a $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ bulk crystal occurs as the manganese concentration x is increased. It was found that the optical absorption edges of $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ manifest blue shift of the

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direct interband gap when the pressure is applied. In the present work, we show that by using energy dispersive X-ray diffraction (EDXD), the same phenomenon exists in $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ bulk crystals, with manganese concentrations $x = 0.016, 0.026, 0.053, 0.07,$ and 0.24 under high pressure.

Our $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ bulk crystals were grown by modified Bridgman method. Due to the high melting point ($\sim 1500^\circ\text{C}$) of $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ crystals, high-purity argon gas at about 100 atm was used as the pressurizing gas during the crystal growth to prevent the quartz container from becoming too soft to be useful. The EDXD measurement was used to characterize the structure of $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ crystals. Experimental details of EDXD measurements and the details in the data fitting of the equation of state (EOS) were described in earlier work [6–8,10–12]. EDXD was performed by using the superconductor wiggler synchrotron beam line X17C of the National Synchrotron Light Source of Brookhaven National Laboratory. The diamond cell was mounted on a sample stage which has $x, y, z, \chi, \omega,$ and θ movements. The stage was controlled by a micro-VAX computer. Alignment of the incident beam, sample, and detector can be obtained with a positional accuracy of $1\ \mu\text{m}$ and an angular accuracy of 0.001° . The energy-dispersive Germanium detector was set in the position where the diffracted angle (θ) was changed to 6° . So, the relation of the energy of reflection, E , versus d-spacings, d , was $Ed = 59.317\ \text{KeV}\ \text{\AA}$.

A series of spectra of $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$ and $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$ bulk crystals for different loading run and the process of decompressing to ambient pressure are shown in Figs. 1 and 2. The standard pressure is identified by the gold chips put in the sample chamber. Lines of internal gold (111), (200), (220), (311), and (222) are also manifested in Figs. 1 and 2, respectively, for standard pressure identification. The peak positions are read out by a peak search program provided by the VAX computer in the beam line X-17C of Brookhaven National Lab [7,8]. For the loading run, the lattice parameters of $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$ and $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$ bulk samples are 5.666 ± 0.001 and $5.668 \pm 0.001\ \text{\AA}$ obtained from EDXD measurements at ambient pressure, respectively, whilst

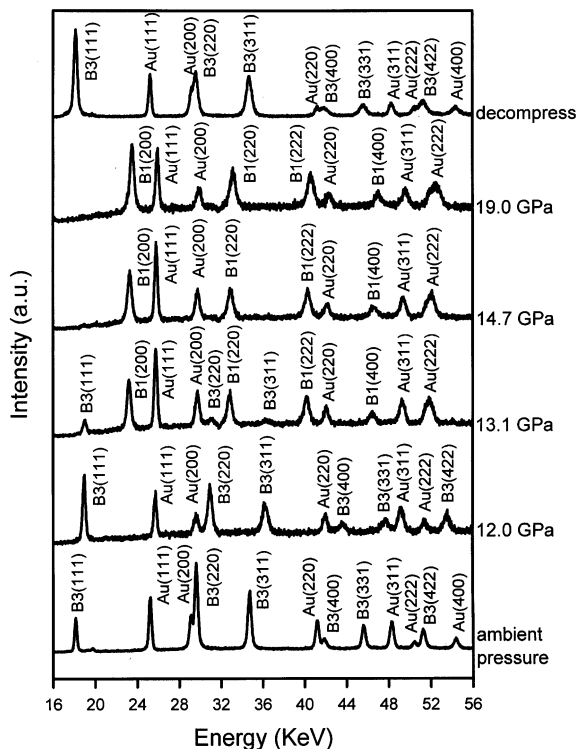


Fig. 1. A series spectra of bulk $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$ at various pressure recorded in a loading run and process of decompression to ambient pressure. The spectra contain the X-ray emission lines of the standard identified pressure lines of internal gold peaks.

Figs. 1 and 2 show that there are six reflections (111), (220), (311), (400), (331), and (422) of B3 (zinc-blende, ZB) phase. By the relation $Ed = 59.317\ \text{KeV}\ \text{\AA}$, the d-spacings of those reflections appearing in Figs. 1 and 2 are 3.271, 2.003, 1.709, 1.416, 1.300 and 1.157 \AA for $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$ and 3.269, 2.018, 1.708, 1.413, 1.300 and 1.156 \AA for $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$, respectively. Because the diffracted energies are very close to each other, the peaks Au(200) and B3(220); Au(220) and B3(400); and Au(222) and B3(422), respectively, are overlapping. At high pressure, all the peaks can be observed clearly. The B3 peaks appearing at the high-energy side of the reflection of gold peaks shows that bulk $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ crystals are more compressible than gold. When the pressure is increased to the onset pressure of 13.1 and 12.4 GPa, in Figs. 1 and 2,

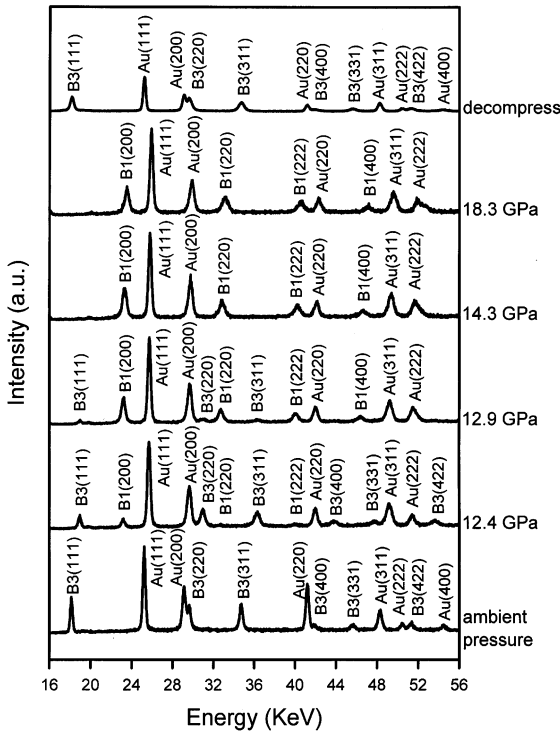


Fig. 2. A series spectra of bulk $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$ at various pressure recorded in a loading run and during the process of decompression to ambient pressure. The spectra contain the X-ray emission lines of the standard identified pressure lines of internal gold peaks.

respectively, the reflections (2 0 0), (2 2 0), (2 2 2), and (4 0 0) of the B1 (rock salt, RS) phase appear at high-energy side of the reflections (1 1 1), (2 2 0), (3 1 1), and (4 0 0) of the B3 phase, respectively, while the (4 0 0) reflection of the B3 (zinc-blende, ZB) phase disappears in Fig. 1. The d-spacings at 13.1 GPa in Fig. 1 are 3.124, 1.904, and 1.634 Å for (1 1 1), (2 2 0), and (3 1 1) of the B3 phase and the lattice parameter is 5.405 ± 0.001 Å. The d-spacings at 13.1 GPa in Fig. 1 are 2.561, 1.814, 1.482 and 1.276 Å for (2 0 0), (2 2 0), (2 2 2), and (4 0 0) of the B1 (rock salt, RS) phase and the lattice parameter is 5.129 ± 0.001 Å. Similarly, the d-spacings at 12.4 GPa in Fig. 2 are 3.130, 1.916, 1.634, 1.353, 1.243, and 1.106 Å for (1 1 1), (2 2 0), (3 1 1), (4 0 0), (3 3 1), and (4 2 2) of B3 phase and the lattice parameter is 5.418 ± 0.001 Å. The d-spacings at 12.4 GPa in Fig. 2 are 2.560, 1.814,

and 1.484 Å for (2 0 0), (2 2 0), and (2 2 2) of the B1 (rock salt, RS) phase and the lattice parameter is 5.130 ± 0.001 Å. The reflections of the B3 phase of $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$ and $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$ bulk crystals disappear completely and only reflections of the B1 phase appear apparently above 14.7 and 12.9 GPa, respectively. The transition pressure, P_t , of B3 to B1 for $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$ and $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$ bulk crystals are assigned as the onset pressure of 13.1 and 12.4 GPa, respectively. The ambiguous region in which the B3 and the B1 phases mixed together, exists from 13.1 to 14.7 GPa and from 12.1 to 12.9 GPa for $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$ and $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$, respectively. The B1 reflections, (2 0 0), (2 2 0), (2 2 2), and (4 0 0) are found to exist up to 19.0 and 18.3 GPa for $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$ and $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$ bulk samples, respectively. The unloading run reveals that bulk $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ samples have reversible pressure property.

The variations of the interplanar distances d_{hkl} (Å) for loading run of bulk $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ crystals, $x = 0.016, 0.026, 0.053, 0.07,$ and 0.24 , are shown in Figs. 3–7. The lattice parameters for $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$, $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$, $\text{Zn}_{0.947}\text{Mn}_{0.053}\text{Se}$, $\text{Zn}_{0.93}\text{Mn}_{0.07}\text{Se}$, and $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$ are 5.666 ± 0.001 , 5.668 ± 0.001 , 5.672 ± 0.001 , 5.677 ± 0.001 and 5.708 ± 0.001 Å at ambient pressure, respectively. This is consistent with the results obtained previously from measuring the lattice parameters of $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ in which the lattice parameters are found to increase with the increasing of Mn concentration [13–16]. All the interplanar distances decrease as the pressure is increased for both the B3 and the B1 phases. The results indicate that the decrease of the interplanar distances with pressure is due to a decrease of the lattice parameter.

Fig. 8 shows the equation of state relations as a function of pressure for bulk $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ crystals. V_0 is the volume at ambient pressure. The data for the B3 and the B1 phases are fitted to the Murnaghan equation by a fitting process as reported previously [7,8,12]. The values of K_0 , the isothermal bulk modulus at ambient pressure, and K'_0 , the pressure derivative of the isothermal bulk modulus evaluated at ambient pressure, of the Murnaghan equation for $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ bulk

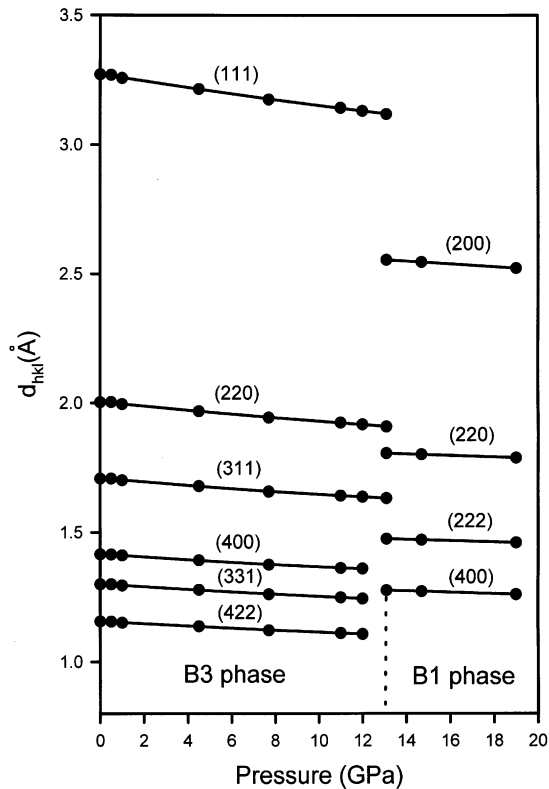


Fig. 3. The variation of d_{hkl} (Å) of bulk $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$ with pressure (GPa) for the B3 and the B1 phases.

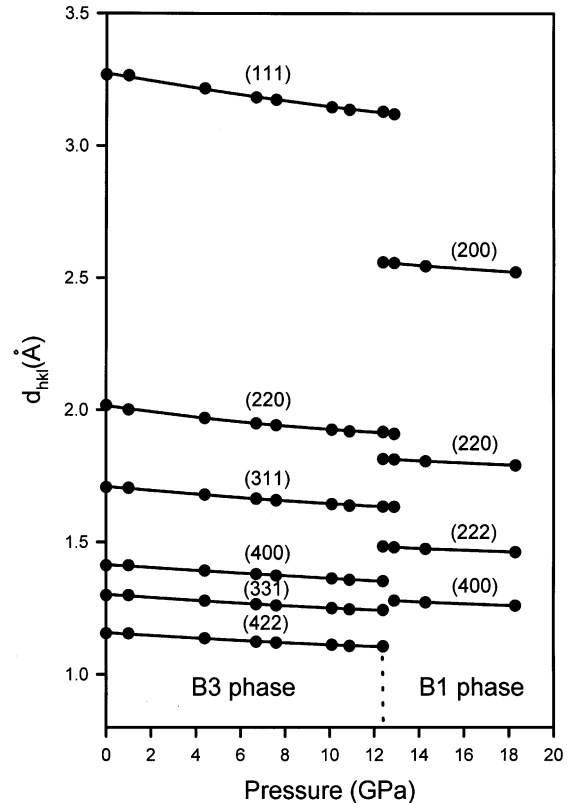


Fig. 4. The variation of d_{hkl} (Å) of bulk $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$ with pressure (GPa) for the B3 and the B1 phases.

crystals in the B3 and the B1 phase transitions obtained from the fitting process are listed in Table 1. The values of K'_0 are consistent with the slopes of the d-spacings below and above the phase transitions (the B3 and the B1 phases) in the loading run spectra. In general, the pressure derivative of the B3 phase is larger than that of the B1 phase in both bulk and thin film [7,8]. It also shows that $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ bulk crystals in the pressure region (B1) above the phase transition are less compressible than that in the pressure region (B3) below the phase transition. The isothermal bulk moduli evaluated at low- and high-pressure regions at ambient pressure slightly decrease with increasing the Mn concentration which indicates that the softening of the lattice is due to the substitution of Zn by Mn. The V/V_0 versus pressures of $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$, $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$, $\text{Zn}_{0.947}\text{Mn}_{0.053}\text{Se}$, $\text{Zn}_{0.93}\text{Mn}_{0.07}\text{Se}$, and $\text{Zn}_{0.76}$

$\text{Mn}_{0.24}\text{Se}$ crystals are shown by using hollow circles, hollow squares, hollow triangles up, hollow triangles down, and hollow diamonds, respectively. The EDXD results show that possible zinc blende(B3) to sodium chloride phase (B1) structure transition for $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$, $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$, $\text{Zn}_{0.947}\text{Mn}_{0.053}\text{Se}$, $\text{Zn}_{0.93}\text{Mn}_{0.07}\text{Se}$, and $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$ occur at 13.1, 12.4, 12.0, 11.8, 9.6 GPa, respectively. Similar to previous work [7], we can explain the reduction of the phase transition pressure of the Mn impurity mixing ZnSe semiconductor by considering the volume change of the unit cell for phase transition from the B3 to the B1 phase. One can note from Table 2 that the increasing of the percentage of the reduction of phase transition pressures with respect to 14.4 GPa of ZnSe relates prominently with the increasing percentage of the reduction of the volume changes for our $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ bulk

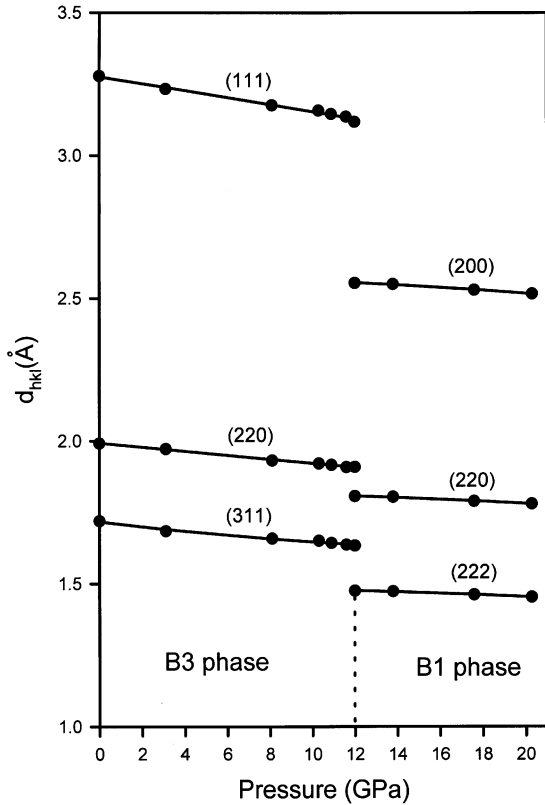


Fig. 5. The variation of d_{hkl} (Å) of bulk $\text{Zn}_{0.947}\text{Mn}_{0.053}\text{Se}$ with pressure (GPa) for the B3 and the B1 phases.

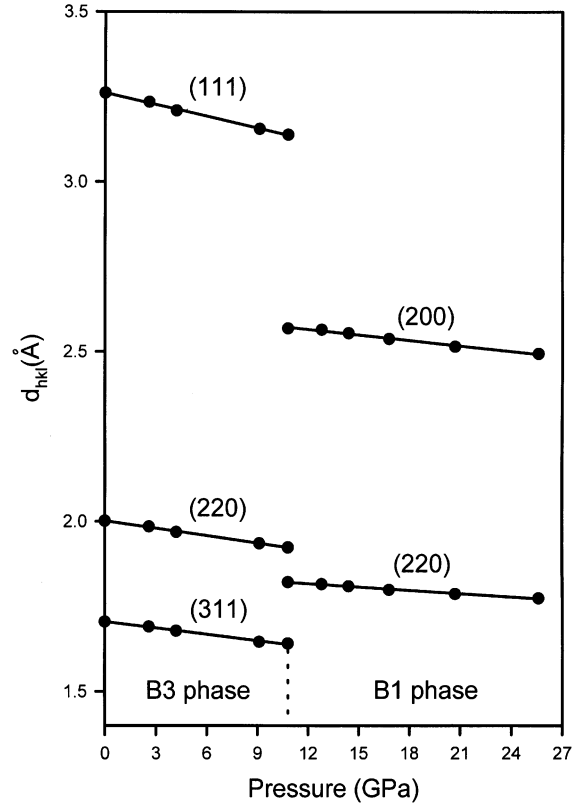


Fig. 6. The variation of d_{hkl} (Å) of bulk $\text{Zn}_{0.93}\text{Mn}_{0.07}\text{Se}$ with pressure (GPa) for the B3 and the B1 phases.

crystals while phase transition from B3 to the B1 occurs. The percentage of the reduction of the volume changes is the ratio of volume changes from B3 to B1 with respect to B3 volume at P_t times 100% at P_t . Above measurements indicate that decreasing in the phase transition pressure P_t for phase transition from B3 to B1 phase can be related to the increasing percentage of the reduction of volume changes, $(\Delta V/V_0)$, by the expression $P_t = [14.4024 + 0.5053(\Delta V/V_0) - 0.0492(\Delta V/V_0)^2]$ in GPa for our cases of the $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ bulk system. Therefore, the larger the decrease of the relative reduction of the volume of B3 to B1 phase transition, the larger is the relative reduction of the phase transition pressure that can be obtained in ZnSe-based ternary compound semiconductors. Hence, the relative change of the volume, $(\Delta V/V_0)$, in the B3 to B1 phase transition may play an important role in the

reduction of the phase transition from B3 to B1 in ZnSe compound semiconductors with Mn impurity ions.

In summary, our EDXD data show that the bulk modulus of the $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ sample decreases as the Mn concentration is increased, e.g. for $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$, $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$, $\text{Zn}_{0.947}\text{Mn}_{0.053}\text{Se}$, $\text{Zn}_{0.93}\text{Mn}_{0.07}\text{Se}$, and $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$; K_0 are 62.26 ± 0.31 , 62.17 ± 0.21 , 61.93 ± 0.29 , 61.82 ± 0.81 , and 60.48 ± 0.26 GPa before phase transition and the pressure derivatives are 4.39 ± 0.18 , 4.43 ± 0.19 , 4.29 ± 0.21 , 4.32 ± 0.19 , and 4.37 ± 0.16 , respectively. The relative volume change from B3 to B1 phase is 12.6, 13.3, 13.6, 13.7, and 16.3% for $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$, $\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$, $\text{Zn}_{0.947}\text{Mn}_{0.053}\text{Se}$, $\text{Zn}_{0.93}\text{Mn}_{0.07}\text{Se}$, and $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$, respectively. The B3 to B1 phase transition pressure is 13.1, 12.4, 12.0, 11.8, and 9.6 GPa for $\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$,

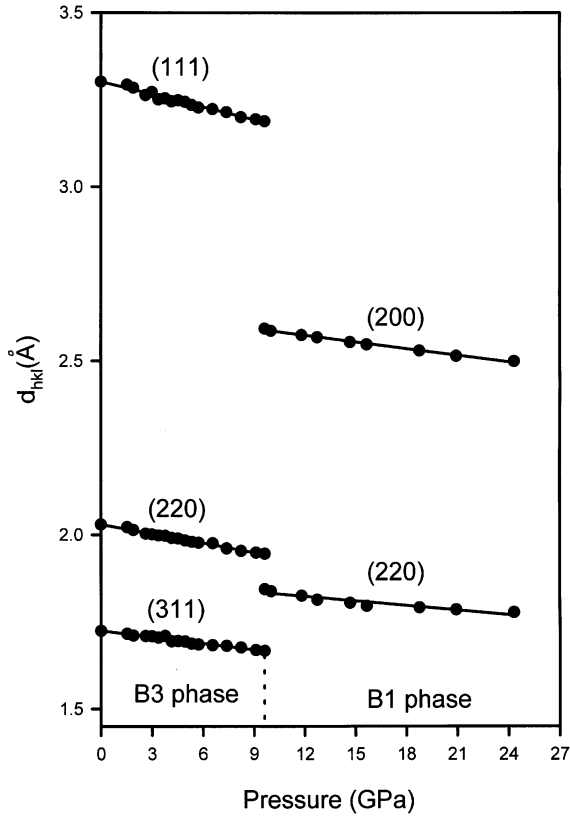


Fig. 7. The variation of d_{hkl} (Å) of bulk $Zn_{0.76}Mn_{0.24}Se$ with pressure (GPa) for the B3 and the B1 phases.

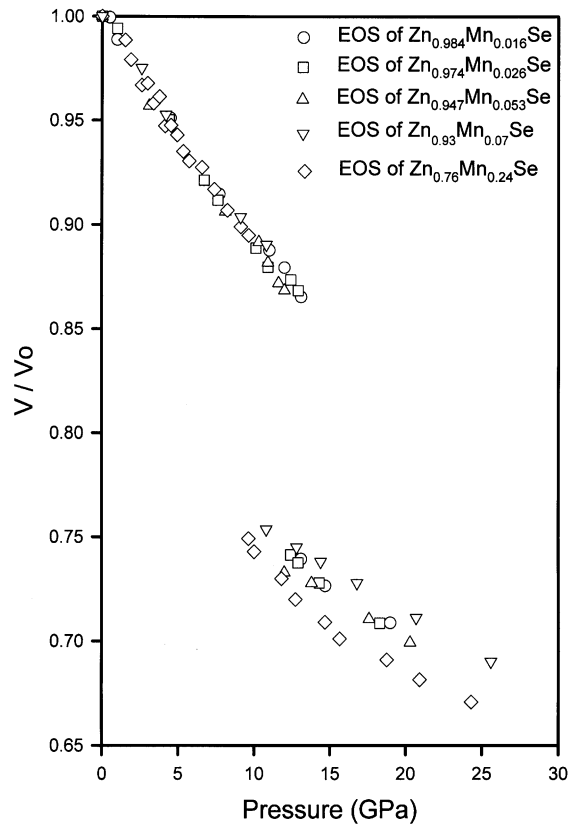


Fig. 8. V/V_0 versus pressure for the B3 and the B1 phases of bulk $Zn_{1-x}Mn_xSe$.

Table 1

The values of K_0 and K'_0 for bulk $Zn_{1-x}Mn_xSe$ under and above the phase transition (B3 and the B1 phase) obtained from the fitting process by Xu et al.^a [12]

Sample	Phase	K_0 (GPa)	K'_0
$Zn_{0.984}Mn_{0.016}Se$	B3	62.26 ± 0.31	4.39 ± 0.18
	B1	80.86 ± 1.73	3.79 ± 0.83
$Zn_{0.974}Mn_{0.026}Se$	B3	62.17 ± 0.21	4.43 ± 0.19
	B1	79.67 ± 1.53	3.82 ± 0.79
$Zn_{0.947}Mn_{0.053}Se$	B3	61.93 ± 0.29	4.29 ± 0.21
	B1	76.58 ± 1.81	3.94 ± 0.93
$Zn_{0.93}Mn_{0.07}Se$	B3	61.82 ± 0.81	4.32 ± 0.19
	B1	73.67 ± 1.79	3.87 ± 0.91
$Zn_{0.76}Mn_{0.24}Se$	B3	60.48 ± 0.26	4.37 ± 0.16
	B1	70.86 ± 1.61	3.64 ± 0.87

^a K_0 is the isothermal bulk modulus at zero pressure, and K'_0 is the pressure derivative of the isothermal bulk modulus evaluated at zero pressure.

Table 2

The phase transition pressure decreased with increasing the volume changed of $\text{Zn}_{0.984}\text{Fe}_{0.016}\text{Se}$, $\text{Zn}_{0.974}\text{Fe}_{0.026}\text{Se}$, $\text{Zn}_{0.947}\text{Fe}_{0.053}\text{Se}$, $\text{Zn}_{0.93}\text{Fe}_{0.07}\text{Se}$, and $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$, respectively

Sample	$(\Delta V/V_0)$: the relative volume change from the B3 to the B1 phase (with respect to B3 volume while at P_t) (%)	$(\Delta P_t/14.4)$: reduction of the transition pressure (GPa) from the B3 to the B1 phase (with respect to 14.4 GPa of ZnSe) (%)	P_t : B3–B1 phase transition pressure (GPa)
$\text{Zn}_{0.984}\text{Mn}_{0.016}\text{Se}$	12.6	9.0	13.1 ± 0.5
$\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$	13.3	13.9	12.4 ± 0.5
$\text{Zn}_{0.947}\text{Mn}_{0.053}\text{Se}$	13.6	16.7	12.0 ± 0.5
$\text{Zn}_{0.93}\text{Mn}_{0.07}\text{Se}$	13.7	18.1	11.8 ± 1.5
$\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$	16.3	33.3	9.6 ± 0.5

$\text{Zn}_{0.974}\text{Mn}_{0.026}\text{Se}$, $\text{Zn}_{0.947}\text{Mn}_{0.053}\text{Se}$, $\text{Zn}_{0.93}\text{Mn}_{0.07}\text{Se}$, and $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$ bulk crystals, respectively. So, the larger the increase of the relative volume change at the phase transition from B3 to B1, the larger is the decrease of the reduction in the semiconductor-metal phase transition pressure. We conclude that the effect of increasing the relative volume change of ZnSe-based ternary semiconductors, as conjectured in our previous paper [7], may be the main reason for the reduction of the stability of the B3 phase under the application of pressure.

Acknowledgements

This work was supported by the National Science Council, Taiwan by the grant number NSC 88-2112-M-134-001 at NHCTC, NSC 89-2112-M-009-038 and NSC90-2112-M-009-018 at NCTU.

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