

High-pressure phase transitions in $\text{Zn}_{1-x}\text{M}_x\text{Se}$ ($M=\text{Cd}, \text{Fe}, \text{and Mn}$)

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Energy-dispersive x-ray diffraction was employed to study pressure-induced phase transitions in $\text{Zn}_{0.84}\text{Fe}_{0.16}\text{Se}$, $\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$, and $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$ crystals up to 21.0, 23.3, and 24.3 GPa, respectively. Our result shows that the $B3$ to $B1$ structure transitions for these crystals occurred at 11.4 ± 0.5 , 9.5 ± 0.3 , and 9.6 ± 0.5 GPa, respectively. Compared to the phase-transition pressure (P_t) of ZnSe (14.4 GPa), a reduction of about 3–5 GPa is exhibited in these ternary compounds. This reduction in phase-transition pressure ΔP_t in the ternary compounds suggests that the fractional volume change, $(\Delta V/V_0)$, of the $B3$ - $B1$ phase admixture might be the source of this reduction. Our results indicate that P_t (with respect to the phase transition pressure 14.4 GPa of ZnSe) is related to the fractional volume change $(\Delta V/V_0)$ by the expression $P_t = [14.4003 + 0.1568(\Delta V/V_0) - 0.0281(\Delta V/V_0)^2]$ GPa. [S0163-1829(98)05426-5]

ZnSe-based ternary compound semiconductors, which are random mixtures of ZnSe and magnetic or nonmagnetic ions, have attracted much attention due to the study of basic physical properties such as the variation of long wavelength optical phonon vibration modes versus the mole fraction of nonmagnetic ions¹ and its ability to tune both the band gap and the lattice constant for application in optoelectronic devices. The diluted magnetic semiconductor² (DMS) $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ is one of the ZnSe ternary compound semiconductors, which have been reported to have many interesting physical properties, such as intermediate-mode behavior^{3,4} and an anomalous dependence of the band gap on the magnetic ion composition.⁵ Some important results of the pressure-induced phase transition of ZnSe ternary compound semiconductors containing magnetic ions have been obtained; e.g., Qadri *et al.*⁶ used the energy-dispersive x-ray diffraction (EDXD) method to investigate the pressure effects of the $\text{Zn}_{0.83}\text{Fe}_{0.17}\text{Se}$ crystal, and Arora and co-workers and others applied a Raman scattering experiment to study the phase transitions of $\text{Zn}_{1-x}\text{Fe}_x\text{Se}$,⁴ $\text{Zn}_{1-x}\text{Co}_x\text{Se}$,⁷ and $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$ (Refs. 8 and 9) under high pressure. It was found that the existence of magnetic ions in the ZnSe crystal resulted in a reduction of the transition pressure. Such a reduction was believed due to the hybridization of $3d$ orbitals into the tetrahedral bonds,^{6,10} while Qadri *et al.*¹¹ reported that the phase transition pressure decreased when the lattice parameter increased. Usually, the semiconductor-metal transition pressure (P_t) identified by the EDXD result corresponds to a change of the crystal structure which accompa-

nies the disappearance of the local magnetic ions and the longitudinal optical (LO) phonon modes according to Raman scattering results.⁴ In this work, we study the phase transition of $\text{Zn}_{1-x}\text{M}_x\text{Se}$, $M=\text{Cd}, \text{Fe}, \text{and Mn}$, crystals by the EDXD method under high pressure.

$\text{Zn}_{1-x}\text{M}_x\text{Se}$, $M=\text{Cd}, \text{Fe}, \text{and Mn}$, crystals grown by the modified Bridgman method were grounded to $1 \mu\text{m}$ size for use in the EDXD measurement. A Mao-Bell-type diamond anvil cell (DAC) with T304 stainless steel gaskets which were preindented to 15.0 GPa was used; the sample hole was $200 \mu\text{m}$ in diameter. The anvil parameters were $1/3$ carats with a $600 \mu\text{m}$ culet. Experimental details were described earlier.¹² For the DAC EDXD experiment, the superconductor wiggler synchrotron beam line X17C of the National Synchrotron Light Source (NSLS) of Brookhaven National Laboratory was used. The beam size was $50 \times 50 \mu\text{m}^2$, a germanium energy dispersive detector was set in the position where the diffracted angle (θ) was 5° . So the relation of the energy of reflection, E , versus d spacings, d , is $Ed = 71.137 \text{ keV \AA}$. Methanol-ethanol 4:1 fluid was used as a hydrostatic pressure medium and the internal gold standard¹³ was employed in the pressure determination in the EDXD measurement, respectively. The peak positions were read out by a peak search program provided by a VAX computer in the beam line X17C; the equation of state (EOS) data were fitted to the Murnaghan equation by use of a fitting program reported earlier.¹⁴

For the case of nonmagnetic impurities, a series of spectra of the $\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$ loading run is shown in Fig. 1, which

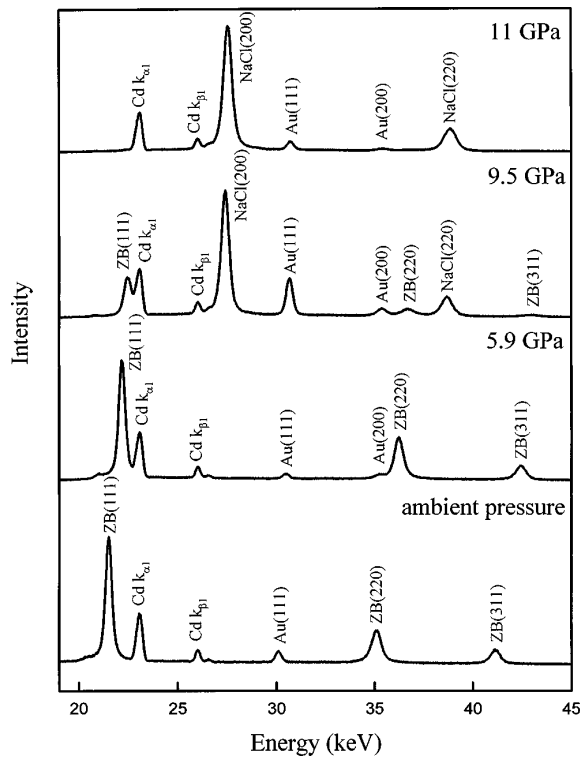


FIG. 1. A series spectra of $\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$ at various pressure recorded in a loading run which contains the x-ray-emission lines of Cd $K\alpha_1$ and Cd $K\beta_1$ and the standard identified pressure lines of Au(111).

contains the x-ray emission lines of Cd and the standard identified pressure lines of internal gold. $\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$ has a lattice parameter of 5.649 ± 0.001 Å from the EDXD measurement at ambient pressure. Figure 1 shows that there are only three reflections (111), (220), and (311) of the B3 [zincblende (ZB)] phase, by the relation of $Ed = 71.137$ keV Å; those which appear are with d spacings 3.272, 2.002, and 1.714 Å, respectively, at ambient pressure. When the pressure was increased to 9.5 GPa, the reflections (200) and (220) of the B1 [rocksalt (RS)] phase appeared at the high-energy side of the reflections (111) and (220) of the B3 phase, respectively. The d spacings at 9.5 GPa are 3.158, 1.939, and 1.646 Å for the B3 phase and 2.59 and 1.835 Å for the B1 phase, while the lattice parameters are 5.472 ± 0.001 Å and 5.185 ± 0.001 Å for B3 and B1 phases, respectively. The ambiguity pressure range of the B3 and B1 phases is from 9.5 to 11.0 GPa; in this pressure region a mixing of the B1 and B3 phases coexisted. The onset pressure of the transition from the B3 to B1 phase is 9.5 GPa; the reflections of the B3 phase of $\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$ disappeared completely and only reflections of the B1 phase appeared apparently above 11.0 GPa. Since one can argue on thermodynamic grounds that the transition pressure should be nearer, or equal to, the onset pressure, therefore, the transition pressure of B3 to B1 for $\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$ is taken as 9.5 GPa. The B1 reflections (200) and (220) were found to exist up to 23.3 GPa. For the case of the magnetic impurity ternary compound of ZnSe, a series of $\text{Zn}_{0.84}\text{Fe}_{0.16}\text{Se}$ and $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$ loading run spectra are similar to those of $\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$. $\text{Zn}_{0.84}\text{Fe}_{0.16}\text{Se}$ and $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$ have lattice parameters 5.639 ± 0.001 and 5.708 ± 0.001 Å, respectively,

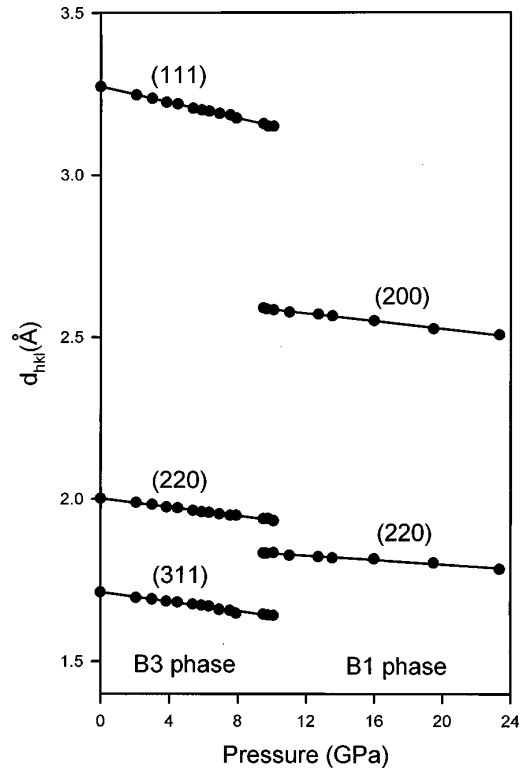


FIG. 2. The variation of d_{hkl} (Å) of $\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$ with pressure (GPa) for the B3 and B1 phases.

as obtained at ambient pressure from EDXD measurements. For the case of $\text{Zn}_{0.84}\text{Fe}_{0.16}\text{Se}$, only (111), (220), and (311) of the B3 phase peaks appeared below 11.4 GPa. The d spacings at 11.4 GPa are 3.112, 2.556, and 1.802 Å for the B3 phase and are 1.861 and 1.634 Å for the B1 phase, respectively. The lattice parameters of the B3 and B1 phases were 5.357 ± 0.001 and 5.104 ± 0.001 Å at 11.4 GPa, respectively. Above 11.4 GPa, peaks of the B3 phase disappeared and alternatively the peak of the B1 phase occurred apparently. The B1 peaks (200) and (220) were found to exist up to 21.0 GPa. In the case of $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$, only reflections (111), (220), and (311) of the B3 phase with d spacings 3.302, 2.030, and 1.725 Å, respectively, appeared at ambient pressure. At 9.6 GPa, the reflections (200) and (220) of the B1 phase appeared at the high-energy side of the reflections (111) and (220) of the B3 phase, respectively. The d spacings at 9.6 GPa are 3.189, 1.946, and 1.668 Å for the B3 phase and 2.593 and 1.844 Å for the B1 phase. At 9.6 GPa the lattice parameter of the B3 phase is 5.519 Å and is 5.202 Å for the B1 phase. Above 9.6 GPa, the reflections of the B3 phase of $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$ disappeared and the reflections of the B1 phase occurred apparently. The B1 reflections (200) and (220) were found to exist up to 24.3 GPa. Importantly, no two-phase coexistence ambiguity range occurred in the case of magnetic impurity ternary compound semiconductors of ZnSe. The structure phase-transition pressures were 11.4, 9.6, and 9.5 GPa for $\text{Zn}_{0.84}\text{Fe}_{0.16}\text{Se}$, $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$, and $\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$, respectively. By the way, the width of the transition zone of $\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$ is larger than in the other two samples.

The variations of the interplanar distances d_{hkl} (Å) shown in Fig. 2 for $\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$, $\text{Zn}_{0.84}\text{Fe}_{0.16}\text{Se}$ and $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$

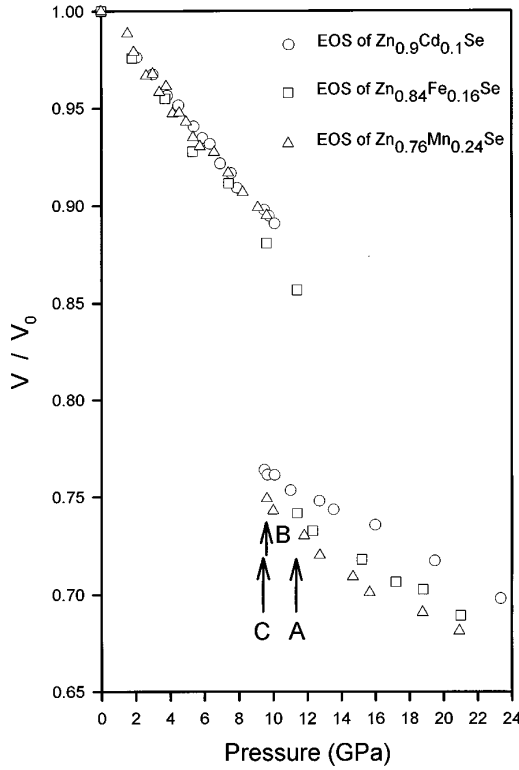


FIG. 3. V/V_0 vs pressure for the $B3$ and $B1$ phases of $Zn_{0.84}Fe_{0.16}Se$, $Zn_{0.76}Mn_{0.24}Se$, and $Zn_{0.9}Cd_{0.1}Se$, respectively. The symbols A, B, and C indicate the transition pressures of the $B3$ to $B1$ phase for $Zn_{0.84}Fe_{0.16}Se$, $Zn_{0.76}Mn_{0.24}Se$, and $Zn_{0.9}Cd_{0.1}Se$, respectively.

have the same relations as that of $Zn_{0.9}Cd_{0.1}Se$. All the interplanar distances decreased as the pressure was increased for both $B3$ and $B1$ phases. Figure 3 shows the EOS relations as a function of pressure for $Zn_{0.84}Fe_{0.16}Se$, $Zn_{0.76}Mn_{0.24}Se$, and $Zn_{0.9}Cd_{0.1}Se$, respectively. V_0 is the volume at ambient pressure. The arrows labeled A, B, and C at 11.4, 9.6, and 9.5 GPa indicate transition pressures of the $B3$ to $B1$ phase for $Zn_{0.84}Fe_{0.16}Se$, $Zn_{0.76}Mn_{0.24}Se$, and $Zn_{0.9}Cd_{0.1}Se$, respectively. The data for both phases were fitted to the Murnaghan equation by a fitting process of Xu *et al.*¹⁴ 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 these three samples in the region below and above the phase transitions ($B3$ and $B1$ phases) obtained from the fitting process are listed in Table I. The values of K'_0 were consistent with the slopes of d spacings for $B3$ and $B1$ in the loading run spectra. It shows that all samples in the pressure region ($B1$) above the phase transition were less compressible than that in the pressure region ($B3$) below the phase transition.

The phase-transition pressure P_t of ZnSe was found to be 14.4 GPa.⁴ As we mentioned above, our results show that the P_t is decreased as the impurity ion Fe, Cd, or Mn was mixed to form the ternary compounds ZnFeSe, ZnCdSe, or ZnMnSe, respectively. For example, the P_t 's of $Zn_{0.84}Fe_{0.16}Se$, $Zn_{0.76}Mn_{0.24}Se$, and $Zn_{0.9}Cd_{0.1}Se$ are 11.4, 9.6, and 9.5 GPa, respectively. The reason for the reduction of the phase transition pressure was extensively studied by many authors for a decade.^{6,10,15,16} Qadri *et al.*⁶ and Ves

TABLE I. The values of K_0 and K'_0 for $Zn_{0.84}Fe_{0.16}Se$, $Zn_{0.76}Mn_{0.24}Se$, and $Zn_{0.9}Cd_{0.1}Se$ under and above the phase transition ($B3$ and $B1$ phases) obtained from the fitting process by Xu *et al.* (Ref. 14). 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.

Sample	Phase	K_0 (GPa)	K'_0
$Zn_{0.84}Fe_{0.16}Se$	$B3$	58.85 ± 0.22	4.12 ± 0.19
	$B1$	80.15 ± 1.69	3.67 ± 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
$Zn_{0.9}Cd_{0.1}Se$	$B3$	60.23 ± 0.29	4.32 ± 0.18
	$B1$	97.02 ± 1.74	3.97 ± 0.85

*et al.*¹⁵ investigated the variation of P_t of $Zn_{1-x}Mn_xSe$ and $Zn_{1-x}Fe_xSe$ with the impurity concentration x of Mn and Fe. They concluded that the decreasing of the $B3$ to $B1$ phase transition pressure was strongly dependent on the increasing of the impurity concentration. In a later work of Qadri *et al.*,¹⁶ the pressure effect on the phase transition of $Cd_{1-x}Mn_xTe$ was investigated, and a similar result was obtained. Maheswaranathan *et al.* studied $Cd_{0.52}Zn_{0.48}Te$ and $Cd_{1-x}Mn_xTe$ with $0 \leq x \leq 0.52$ (Ref. 10) using a photoemission method; they indicated that the substitution of Zn by Cd in the zinc-blende lattice made the lattice more stable than the substitution of Zn by Mn. They found that Mn, but not Zn, weakened the zinc-blende crystal structure and made it less stable under pressure and suggested that $3d$ orbitals of Mn ions but not Zn ions hybridize into tetrahedral bonds because the $3d$ electrons are less tightly bound in Mn than in Zn. And they also found that Cd and Zn d levels do not hybridize with sp^3 bonding orbitals. Therefore, they concluded that the cause of the reduction of the phase-transition pressure was attributed mainly to the hybridization of the Mn or Fe $3d$ orbitals into tetrahedral bonds in the Mn or Fe ternary alloys. Furthermore, Qadri *et al.*¹¹ and Maheswaranathan *et al.*¹⁰ indicated that in the $(Zn,M)Se$ ($M=Fe, Mn$) system the lattice parameter increased and the transition pressure decreased as the magnetic ion was mixed into ZnSe. The covalent radius of Zn is 1.25 Å, which is larger than the 1.17 Å of Fe and 1.17 Å of Mn, respectively. Thus, the reduction of the phase-transition pressure P_t was also ascribed to the decrease of the covalent radius of the impurity ion as Fe or Mn substituted the Zn ions.

Arora and Sakuntala,⁸ in the investigation of the relationship of P_t versus x in the ternary system $Zn_{1-x}Mn_xSe$, observed that an apparently different behavior existed in the higher-impurity-concentration ternary in contrast to the result of the lower- x ternary investigated by Qadri *et al.*⁶ and Ves *et al.*¹⁵. Thus, the transition pressure did not manifest a strong dependence on the Mn concentration. Furthermore, our results show that a reduction of transition pressure was also present in the nonmagnetic ternary compound $Zn_{0.9}Cd_{0.1}Se$. This is a manifestation of the fact that the hybridization of the $3d$ orbitals of the magnetic ion Fe or Mn might not be the main reason for the reduction of the transition pressure in the ternary alloys of ZnSe. It is also known the covalent radius of Cd is 1.48 Å which is larger than that (1.25 Å) of Zn. Thus, the reduction of the phase-transition

TABLE II. The phase transition pressure decrease with increasing volume change of $\text{Zn}_{0.84}\text{Fe}_{0.16}\text{Se}$, $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$, and $\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$, respectively.

Sample	$(\Delta V/V_0)$: the fractional volume changes from the $B3$ to $B1$ phase (with respect to the $B3$ volume while at P_t)	$(\Delta P_t/14.4)$: percentage of the reduction of the $B3$ to $B1$ phase-transition pressure (GPa) (with respect to 14.4 GPa of ZnSe)	P_t : $B3$ to $B1$ phase-transition pressure (GPa)
$\text{Zn}_{0.84}\text{Fe}_{0.16}\text{Se}$	13.5%	20.8%	11.4 ± 0.5
$\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$	16.3%	33.3%	9.6 ± 0.5
$\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$	16.1%	34.0%	9.5 ± 0.3

pressure caused by the decrease of the covalent radius of the impurity ion in the ZnMSe system proposed by Qadri *et al.* could not be correct.

To explain the reduction of the phase-transition pressure of the impurity mixing semiconductor, let us consider the volume change of the unit cell for phase transition from the $B3$ to $B1$ phase in Table II. One can note that the increase of the percentage of the reduction of phase-transition pressures with respect to 14.4 GPa of ZnSe relates prominently to the increasing percentage of the reduction of the volume changes for our three samples while phase transition from $B3$ to $B1$ occurred. The percentage of the reduction of the volume changes is the ratio of the $B1$ volume to the $B3$ volume times 100% at P_t . The above measurements indicate that decreasing in the phase-transition pressure P_t for a phase transition from the $B3$ to $B1$ phase can be related to the increase of the percentage of the reduction of the volume changes $(\Delta V/V_0)$, by the expression $P_t = [14.4003 + 0.1568(\Delta V/V_0) - 0.0281(\Delta V/V_0)^2]$ in GPa for our cases of the $\text{Zn}_{1-x}\text{Mn}_x\text{Se}$, $M = \text{Cd, Fe, and Mn}$ system. Therefore, the greater the percentage of the reduction of the volume decrease of the $B3$ to $B1$ phase transition is, the greater the percentage of the reduction of the phase-transition pressure can be obtained in ZnSe-based ternary compound semicon-

ductors. Hence, the fractional volume changes $(\Delta V/V_0)$, while at the $B3$ to $B1$ phase transition might be the source of the reduction of the phase transition pressure from $B3$ to $B1$ for ZnSe compound semiconductors of random kinds of impurity ions.

In summary, our EDXD data showed that the bulk modulus for $\text{Zn}_{0.84}\text{Fe}_{0.16}\text{Se}$, $\text{Zn}_{0.76}\text{Mn}_{0.24}\text{Se}$, and $\text{Zn}_{0.9}\text{Cd}_{0.1}\text{Se}$ is 58.85 ± 0.22 , 60.48 ± 0.32 , and 60.23 ± 0.29 GPa before phase transition and the pressure derivative is 4.12 ± 0.16 , 4.37 ± 0.21 , and 4.32 ± 0.18 , respectively. The greater the increase of the fractional volume changes while at the phase-transition ($B3$ to $B1$) region, the greater the decrease of the reduction in the semiconductor-metal phase-transition pressure can be obtained. No apparent effect of $3d$ electronic hybridization can be observed in our works. We suggest that the effect of increasing the fractional volume change of ZnSe-based ternary semiconductors with any kind of impurity ions may be the main reason to reduce the stability of the $B3$ phase under the application of pressure.

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