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Structural phase transitions in GaAs to 108 GPa

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The III-V compound GaAs was studied using energy-dispersive x-ray diffraction with a synchrotron source up to a pressure of 108 GPa. When the pressure was increased to 16.6 GPa, the GaAs sample transformed from the zinc-blende structure to an orthorhombic structure [GaAs(II)], space group *Pmm*2, consisting of a primitive orthorhombic lattice with a basis of (0,0,0) and $(0,\frac{1}{2},\alpha)$, where $\alpha=0.35$. Upon a further increase of pressure to 24±1 GPa, GaAs(II) transformed to another orthorhombic structure [GaAs(III)], space group *Imm*2, consisting of a body-centered orthorhombic lattice with a basis of (0,0,0) and $(0,\frac{1}{2},\Delta)$, where Δ is 0.425 at 28.1 GPa. With increasing pressure, Δ approached $\frac{1}{2}$ and the GaAs(III) structure gradually assumed the symmetry of the simple hexagonal structure. The transition to the simple hexagonal structure [GaAs(IV)] was completed in the vicinity of 60–80 GPa. The structure remains simple hexagonal up to at least 108 GPa, the highest pressure reached in this study.

Recent high-pressure structural studies of germanium[1–3] and GaSb (Ref. 4) emphasize the need for a thorough understanding of the high-pressure structural transitions in the closely related compound GaAs. From previous high-pressure x-ray experiment,[5,6] GaAs is known to transform from the zinc-blende structure to an unidentified orthorhombic structure at a pressure of approximately 17 GPa. A second transition at a slightly higher pressure was suggested by a high-pressure electrical resistance experiment by Minomura and Drickamer,[7] but the existence of this transition has been disputed by at least one high-pressure x-ray study.[5] Theoretical calculations of the stabilities of various proposed structures at high pressure have not been able to present a much clearer understanding of GaAs. Pseudopotential calculations by Froyen and Cohen[8] show that the β-Sn, NiAs, and rock-salt structures are all equally stable at high pressures to within the accuracy of the calculations.

In order to answer the great number of questions concerning the high-pressure structural transitions of GaAs, we have undertaken a new high-pressure x-ray diffraction study of this compound using an x-ray apparatus[9] capable of illuminating and collecting data from portions of the sample as small as 5 μm in diameter, thus reducing the effects of pressure gradients and yielding relatively high quality x-ray data. This study extends the x-ray data on GaAs to pressures beyond 1 Mbar.

All of the x-ray data were collected at the Cornell High Energy Synchrotron Source (CHESS) using energy-dispersive x-ray diffraction (EDXD). Details about the experimental apparatus and technique can be found in Refs. 9 and 10. An extremely pure sample (> 99.9999%) of finely ground GaAs powder was used in all three high-pressure x-ray experiments of our study. For two of the experiments we used a cell with diamond anvils 640 μm in diameter and a 150 μm diameter sample region to a maximum pressure of 43.8 GPa. The third experiment, which attained 108 GPa before being down-loaded, employed a cell with 300-μm-diam diamond culets, 100-μm-diam central flats, 5° bevels, and a 50-μm-diam sample region. In all three experiments the sample pressure was determined by ruby fluorescence.[11] The precision and accuracy of the present technique is discussed in the Appendix.

In agreement with previous high-pressure x-ray studies,[5,6] we found a transition from a zinc-blende structure to an orthorhombic structure at about 17 GPa. The volume collapses by 17.2% at this transition. The diffraction peaks from the new phase, GaAs(II), were first evident at 16.6 GPa, and the transition to GaAs(II) was complete by 22.9 GPa. Figure 1 shows the EDXD spectrum obtained at this pressure. We found that the InSb(IV) structure of Yu, Spain, and Skelton[12] is in good agreement with the observed diffraction line positions and intensities. This structure has an orthorhombic unit cell with space group *Pmm*2 and atoms at (0,0,0) and $(0,\frac{1}{2},\alpha)$, where $\alpha=0.35$ results in diffraction lines in good agreement with those observed at 22.9 GPa. We were unable to determine the ordering of the gallium and arsenic atoms among the various atomic sites because the atomic scattering factors of gallium and arsenic are so similar (atomic numbers 31 and 33, respectively). Table I lists the observed and calculated diffraction-line positions and intensities based on this structure.

Upon an increase in pressure to 25.1 GPa, several subtle changes in the diffraction patterns took place, indicating another structural phase transition. This transition is characterized by the merging of the (120) and (111) lines and the disappearance of the (121) line of GaAs(II). Except for these changes, the diffraction spectrum of the new phase, GaAs(III), at 25.1 GPa is very similar to that of GaAs(II) at 22.9 GPa. Because of the subtle nature of these changes and the close proximity of this transition to the first one at 16.6 GPa, this second transition might have been undetected by previous x-ray studies. Note that this II-III transition is consistent with the second drop in resistance observed by Minomura and Drickamer[7] at a pressure slightly above the I-II transition.

The diffraction pattern obtained for GaAs(III) at 28.1 GPa (Fig. 2), just above the II-III transition, appeared very similar to that of a simple hexagonal structure, the rough positions and relative intensities of ten diffraction lines agreeing fairly well with the simple hexagonal structure. A closer examination of the GaAs(III) data, however, revealed that many of the diffraction lines were shifted slightly from their proper positions. Furthermore, there was an additional diffraction peak, marked by an arrow in Fig. 2, that could not be indexed according to the simple hexagonal structure. To ensure that this peak was genuinely from the GaAs sample and did not result from the steel gasket, the ruby chip, or the diamond anvils, the entire 150-μm-diam sample region was scanned with a 20-μm-diam x-ray collimator. The presence of the additional peak throughout the sample and its failure to correspond to any gasket peak convinced us that this additional peak was a true GaAs(III) peak. The existence of this peak was also confirmed by studies with a second high-pressure cell loaded with GaAs.

Given the close resemblance between the GaAs(III) diffraction pattern and that expected from a simple hexagonal structure, we hypothesized that the structure of GaAs(III) resulted from a slight distortion of the simple hexagonal structure, resulting in slight shifts of the diffraction lines and breaking the symmetry of the hexagonal lattice so that the additional peak appears. We found that the orthorhombic structure shown in Fig. 3 describes the observed diffraction pattern quite well, explaining the shifts in the diffraction lines and the presence of the additional diffraction peak. The structure has space group *Imm*2 with atoms at (0,0,0) and $(0,\frac{1}{2},\Delta)$. Again, we were unable to determine the ordering of the gallium and arsenic atoms among these sites. Note that when $\Delta=\frac{1}{2}$ and $b=\sqrt{3}c$, this structure becomes the simple hexagonal structure. Changing Δ from $\frac{1}{2}$ results in (312) and (132) doublet which constitutes the additional peak. At 28.1 GPa, b/c was $1.05\sqrt{3}$ and Δ was approximately 0.425, indicating that the structure was significantly distorted from the simple hexagonal structure at this pressure. Table II lists the observed and calculated diffraction-line positions and intensities based on this structure. To within experimental error, we could detect no discontinuous volume collapse at the II-III transition.

As the pressure was increased above 28.1 GPa, the intensity of the additional peak and the shifts in the diffraction lines gradually decreased, and the spectra increasingly corresponded to the simple hexagonal structure. Finally, at 65.5 GPa, the additional peak had disappeared and the spectrum agreed very well with the simple

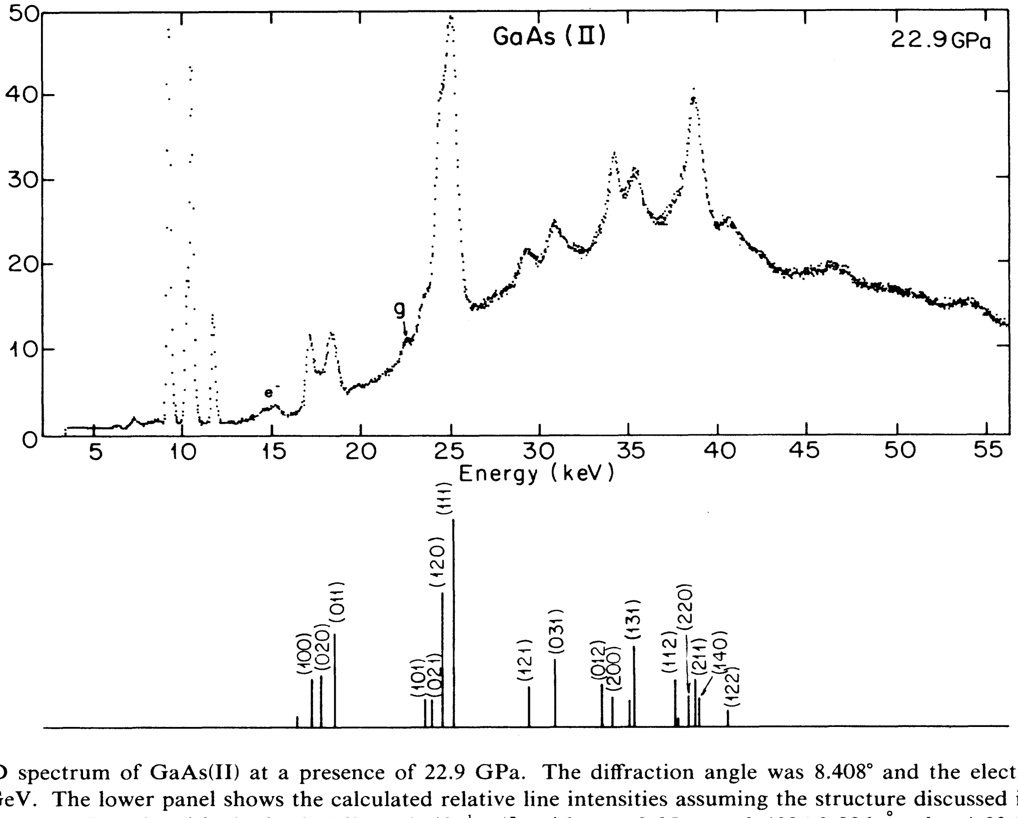

FIG. 1. EDXD spectrum of GaAs(II) at a presence of 22.9 GPa. The diffraction angle was 8.408° and the electron energy at CHESS was 5.3 GeV. The lower panel shows the calculated relative line intensities assuming the structure discussed in the text for GaAs(II) [space group *Pmm*2 with basis (0,0,0) and $(0,\frac{1}{2},\alpha)$] with $\alpha=0.35$, $a=2.482\pm0.006$ Å, $b=4.83\pm0.01$ Å, and $c=2.618\pm0.006$ Å. The lattice parameters have a fractional error of 2.5×10^{-3}; see Appendix. The sample thickness was set to 45 μm. The large, sharp peaks between 8 and 13 keV are gallium and arsenic fluorescence peaks. The peak labeled *g* is a diffraction line from the steel gasket; those labeled e^- are germanium escape peaks.

TABLE I. List of the observed and calculated interplanar spacings and intensities (int.) of GaAs(II) at 22.9 GPa assuming an orthorhombic structure (space group *Pmm*2) with a basis of (0,0,0) and $(0,\frac{1}{2},\alpha)$. The lattice parameters are $a=2.482\pm0.006$ Å, $b=4.83\pm0.01$ Å, and $c=2.618\pm0.006$ Å. The calculation of the diffraction-line intensities is discussed in Ref. 10. A sample thickness of 45 μm was assumed. The parameter α was set to 0.35. The lattice parameters and the observed interplanar spacings have a fractional error of 2.5×10^{-3}; see Appendix.

(*hkl*)	d_{obs}	d_{calc}	Obs. int. (%)	Calc. int. (%)
(100)	2.476	2.482	11.2	23.9
(020)[a]	2.418	2.415	11.3	25.7
(011)	2.305	2.302	20.8	45.3
(120)	1.736	1.731	43.0	64.7
(111)	1.687	1.688	100.0	100.0
(121)	1.445	1.444	10.2	19.4
(031)	1.374	1.371	15.9	32.4
(012)+(200)	1.239	1.264,1.241	21.9	34.9
(040)+(131)	1.197	1.207,1.200	22.5	51.5
(220+(211)+(140)	1.093	1.104,1.092,1.086	47.3	51.0
(122)	1.044	1.044	4.4	7.2

[a] Observed position and intensity obtained by deconvolution of this peak from the (100) and (011) peaks.

hexagonal structure. By cycling the pressure on the GaAs sample, we found that the additional peak reappeared when the pressure was decreased to 57.5 GPa, and disappeared again when the pressure was increased to 70.6 GPa. This transition from an orthorhombic distortion of the simple hexagonal structure [GaAs(III)] to an undistorted simple hexagonal structure [GaAs(IV)] appeared to be gradual; no sudden change in either the lattice parameters or the Δ parameter [as calculated from the intensity of the (312) and (132) doublet] was ever observed. Figure 4 shows a plot of Δ versus pressure. Although the additional peak associated with the orthorhombic GaAs(III) phase disappears below the statistical noise in our spectrums at 61.5 ± 3.9 GPa, an extrapolation of this Δ-versus-pressure data (Fig. 4) indicates that the transformation to the simple hexagonal phase may not be complete until roughly 80 GPa. The GaAs sample remained in the simple hexagonal structure from the III-IV transition up to 108 GPa, the highest pressure reached in our study. Figure 5 shows the diffraction pattern obtained for GaAs(IV) at 108 GPa with diffraction peaks now indexed according to the simple hexagonal structure. Finally, the pressure-versus-volume equation of state is plotted in Fig. 6, and the parameters from the Birch[13] first-order fits to the *P*-*V* data are listed in Table III.

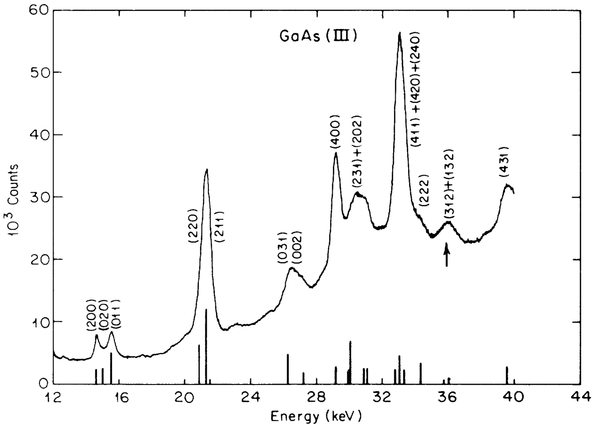

FIG. 2. EDXD spectrum of GaAs(III) at a pressure of 28.1 GPa. The diffraction angle was 9.956° and the electron energy at CHESS was 5.3 GeV. With the exception of the peak marked by an arrow, all the diffraction peaks can be roughly indexed to the simple hexagonal structure. The peaks are shown indexed according to the orthorhombic structure discussed in the text for GaAs(III) [space group *Imm*2 with basis (0,0,0) and $(0,\frac{1}{2},\Delta)$] with $\Delta=0.425$, $a=4.92\pm0.01$ Å, $b=4.79\pm0.01$ Å, and $c=2.635\pm0.006$ Å. Also shown are the calculated relative line intensities based on this structure. The sample thickness was set to 35 μm.

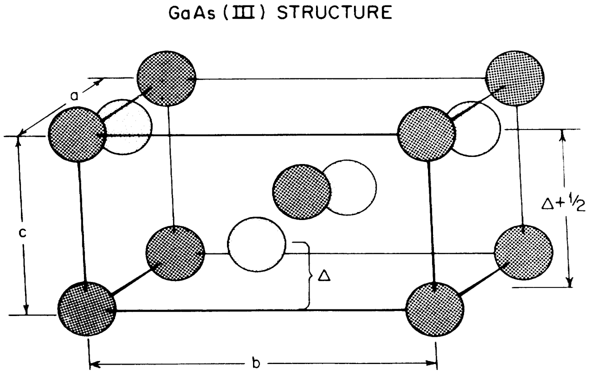

FIG. 3. The proposed structure of GaAs(III). The unit cell is orthorhombic with space group *Imm*2 and a basis of (0,0,0) and $(0,\frac{1}{2},\Delta)$. When $a=b$, $c\cong a/2$, and $\Delta=\frac{1}{4}$, this structure becomes the β-Sn structure. When $b/c=\sqrt{3}$ and $\Delta=\frac{1}{2}$, it becomes the simple hexagonal structure.

TABLE II. List of the observed and calculated interplanar spacings and intensities (int.) of GaAs(III) at 28.1 GPa assuming an orthorhombic structure (space group $Imm2$) with a basis of (0,0,0) and $(0,\frac{1}{2},\Delta)$. The lattice parameters are $a=4.92\pm0.01$ Å, $b=4.79\pm0.01$ Å, and $c=2.635\pm0.006$ Å. The calculation of the diffraction-line intensities is discussed in Ref. 10. A sample thickness of 45 μm was assumed. The parameter Δ was set to 0.425. The lattice parameters and the observed interplanar spacings have a fractional error of 2.5×10^{-3}; see Appendix.

(hkl)	d_{obs}	d_{calc}	Obs. int. (%)	Calc. int. (%)
(200)	2.452	2.457	5.4	7.2
(020)[a]	2.399	2.395	3.7	8.0
(011)	2.308	2.309	9.1	17.3
(220)+(211)	1.684	1.715,1.683	81.9	99.8
(031)+(002)	1.355	1.365,1.317	24.9	44.5
(112)+(400)	1.229	1.230,1.229	32.1	26.3
(040)+(231)+(202)+(022)	1.169	1.197,1.194,1.161,1.154	46.4	100.0
(420)+(411)+(240)	1.085	1.085,1.093,1.076	100.0	73.0
(312)+(132)	0.997	1.004,0.995	11.0	11.1
(431)+(051)+(402)	0.907	0.913,0.900,0.899	20.2	30.6

[a] Observed position and intensity obtained by deconvolution of this peak from the (200) and (011) peaks.

The sequence of high-pressure phase transitions for GaAs [zinc-blende—GaAs(II)—GaAs(III)–simple hexagonal] differs from those found for GaSb and Ge. If we make no distinction between the gallium atoms and antimony atoms in the GaSb structures, GaSb (Ref. 4) and Ge (Refs. 1–3) share the same sequence of structural transitions through their first two transitions: diamond–β-Sn–primitive hexagonal. In contrast, GaAs transforms from zinc-blende to a sixfold-coordinated orthorhombic structure [GaAs(II)] instead of to the β-Sn structure. Furthermore, instead of transforming to the simple hexagonal structure at the second transition, GaAs assumes another orthorhombic structure [GaAs(III)] that gradually transforms to the simple hexagonal structure.

The two structures which we have introduced for GaAs(II) and GaAs(III) warrant further discussion. The GaAs(II) structure, which has atoms at (0,0,0) and $(0,\frac{1}{2},0.35)$, is sixfold coordinated, each atom forming a slightly distorted octahedron with its nearest neighbors. An interesting feature of this structure is that it is very easily distorted into the simple hexagonal structure by making small adjustments in the lattice parameters, and slightly displacing the atom at $(0,\frac{1}{2},\alpha)$ from $\alpha=0.35$ to $\alpha=\frac{1}{2}$. The Ewald energy does not stabilize the GaAs(II) structure against such a distortion. In fact, the net force on the $(0,\frac{1}{2},0.35)$ atom due to the Ewald interaction with its nearest neighbors should be strongly forcing this atom to the much more symmetric $(0,\frac{1}{2},\frac{1}{2})$ position. The fact that the GaAs(II) structure is stable with respect to such distortions suggests the existence of covalent bonds restraining the movement of the atom at $(0,\frac{1}{2},0.35)$ to $(0,\frac{1}{2},\frac{1}{2})$. This view is supported by evidence that GaAs(II) is a semimetal or a semiconductor rather than a metal. The resistance of GaAs drops by a factor of 10–20 at the II-III transition (Ref. 7); such a large drop is more characteristic of a semimetal-metal or semiconductor-metal transition than a metal-metal one. Covalent bonds could remain quite strong in the GaAs(II) phase and may be acting to stabilize the GaAs(II) structure. Finally, we note that the GaAs(II) structure can be easily obtained from the rock-salt structure by a slight adjustment of the lattice parameters accompanied by shearing alternate (100) planes in the [001] direction. This shearing corresponds to a soft TA(X)-phonon mode in rock-salt. Since the rock-salt structure had, by a slight margin, the lowest energy of the structures (β-Sn, NiAs, CsCl, rock-salt) which Froyen and Cohen[8] investigated as possibilities for GaAs(II), and since they noted that the TA(X)-phonon mode had a very low frequency, it is plausible that the structure which we propose for GaAs(II) has a lower energy than any of these structures. A pseudopotential calculation of the energy of this structure should still be performed, however.

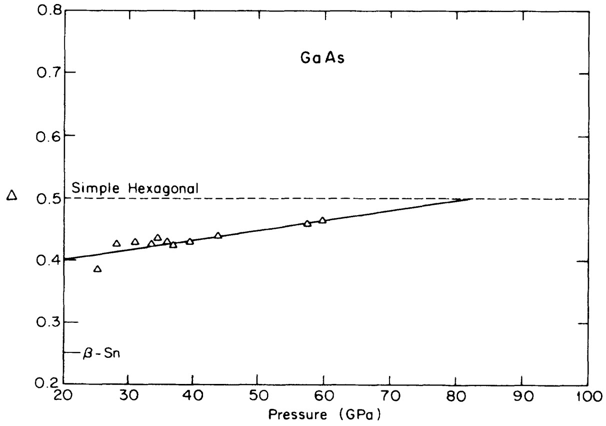

FIG. 4. Plot of Δ vs pressure. A linear extrapolation of the data points indicates that Δ may not assume the value of 0.5, which corresponds to the simple hexagonal structure, until roughly 80 GPa.

The GaAs(III) structure is also an interesting one. This structure can be obtained from the GaAs(II) structure by shearing alternate (100) planes by $(b/2+c/2)$,

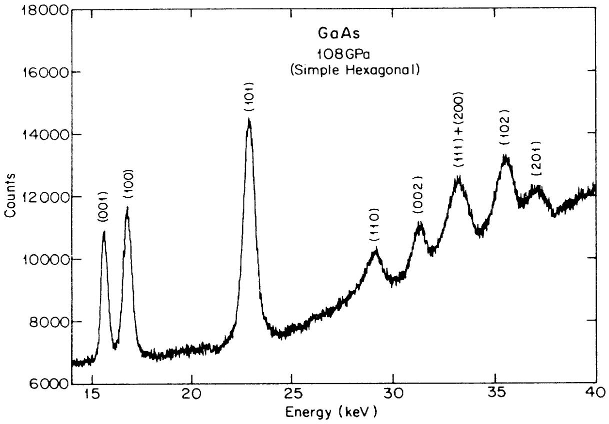

FIG. 5. EDXD spectrum of GaAs in the simple hexagonal structure [GaAs(IV)] at a pressure of 108 GPa. The diffraction angle was 10.049° and the electron energy was 5.3 GeV.

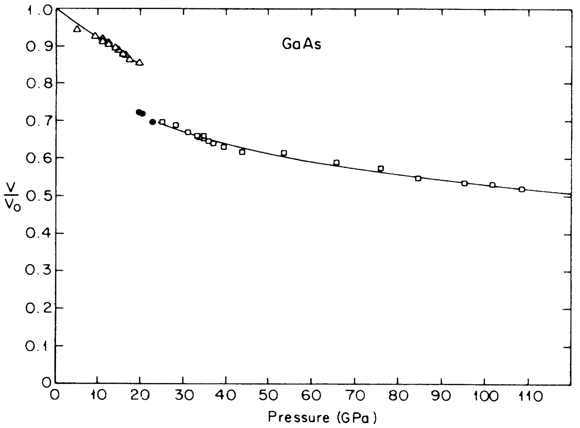

FIG. 6. Plot of the reduced volume V/V_0 of GaAs vs pressure. Fits of the data points to the first-order Birch (Ref. 12) equation are shown by the curves. The fitting parameters are listed in Table III. The data points marked by △ indicate the GaAs(I) (zinc-blende) phase, the points marked by ● the GaAs(II) phase, and the points marked by □ the GaAs(III) and GaAs(IV) (simple hexagonal) phases. We combined the GaAs(III) and GaAs(IV) data points as one set because the III-IV transition was continuous; therefore, there was no clear, sharp III-IV transition pressure.

followed by slight adjustments of the atomic positional parameters. The shearing of alternate (100) layers effectively doubles the a axis of the GaAs(II) unit cell, as can be seen from a comparison of the lattice constant data of GaAs(II) and GaAs(III) in Tables I and II. A notable point about the GaAs(III) structure is its close relation to both the β-Sn and simple hexagonal structures. It has long been known that the β-Sn structure can be continuously transformed to the simple hexagonal structure by displacing one of the body-centered sublattices of β-Sn relative to the other and making slight adjustments in the lattice parameters.[14] This displacement corresponds to the LO(Γ)-phonon mode, and pressure-induced transitions between the β-Sn and simple hexagonal structures have been attributed to the softening of this phonon mode.[3] The soft-mode transition from β-Sn to simple hexagonal can be visualized in terms of the GaAs(III) structure which, with appropriate values for Δ and the lattice parameters, can assume either the β-Sn or simple hexagonal structure. When $\Delta=\frac{1}{4}$ (and $a=b$, $c/a \cong 0.5$), the proposed GaAs(III) structure becomes the β-Sn structure. When $\Delta=\frac{1}{2}$ (and $b=\sqrt{3}c$, $a=2c$), the structure is simple hexagonal. A soft LO(Γ) mode transition from the β-Sn structure to the simple hexagonal structure corresponds to an abrupt change in Δ from $\frac{1}{4}$ to $\frac{1}{2}$, resulting in a sudden shift of one of the body-centered sublattices with respect to the other. Stable or metastable phases forming in a structure with a Δ between $\frac{1}{4}$ and $\frac{1}{2}$, i.e., a structure intermediate between β-Sn and simple hexagonal, are very rare; a metastable alloy of bismuth and indium[15] is one of the few materials known to crystallize in such an intermediate β-Sn–simple hexagonal structure. The discovery of GaAs(III) with a Δ varying from 0.38 to 0.50 marks the first observation of an entire range of intermediate β-Sn–simple hexagonal structures in one compound.

The possible existence of a range of stable intermediate β-Sn–simple hexagonal structures has been previously suggested. In their theoretical examination of the β-Sn to simple hexagonal transition in silicon, Needs and Martin[16] found that at the transition pressure the enthalpy $H=E+PV$ (or, equivalently, the zero-temperature Gibbs free energy) was nearly constant along the transition path from $\Delta=0.25$ (β-Sn) to $\Delta=0.50$ (simple hexagonal). From this fact they concluded that there may be a continuum of stable structures between β-Sn and simple hexagonal near the β-Sn–simple hexagonal transition pressure in silicon.

In this study we identified the orthorhombic structure of GaAs(II), and found and identified two additional phases, GaAs(III) and GaAs(IV), occurring at higher pressures. We can summarize these results as follows.

TABLE III. Parameters obtained by a fit of the first-order Birch equation (Ref. 13) to the reduced volume-vs-pressure data of GaAs. The GaAs(II) data are limited to a narrow pressure range and the fit parameters are not included in the table. Because the III-IV transition is continuous, the GaAs(III) and GaAs(IV) P-V data points were fitted together as one set.

Phase	V_N/V_0	B_0 (GPa)	B_0'
GaAs(I)	1.000	75.4[a]	4.49[a]
GaAs(III), GaAs(IV)	0.881±0.004	57.0±0.6	4.80±0.05

[a] Reference 17.

(1) GaAs(II) is an orthorhombic sixfold-coordinated structure with space group $Pmm2$ and atoms at (0,0,0) and $(0,\frac{1}{2},\alpha)$, where $\alpha=0.35$. We believe that this phase is a semimetal or semiconductor and that the structure is stabilized by covalent bonds.

(2) A new phase, GaAs(III), occurs at 20.5 ± 1.1 GPa. Its structure is also orthorhombic (space group $Imm2$) with atoms situated at (0,0,0) and $(0,\frac{1}{2},\Delta)$, where $\Delta=0.425$. This structure can be visualized as an orthorhombic distortion of the simple hexagonal structure with the distortion decreasing as the pressure increases.

(3) At pressures above 61.5 ± 3.9 GPa, it appears that the distortion from the simple hexagonal structure is zero and that GaAs(III) has completed its transition to the simple hexagonal structure of GaAs(IV); however, an extrapolation of the Δ-versus-pressure data indicates that the transition to the simple hexagonal structure may not be complete until about 80 GPa.

(4) GaAs assumes the simple hexagonal structure [GaAs(IV)] from 60 to 80 GPa to at least 108 GPa, the highest pressure reached in this study.

Note added in proof. Recently Zhang and Cohen[18] have done first principles pseudopotential calculations for GaAs(II) structure and indeed found it to be energetically favored over other competing structures.

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APPENDIX: PRECISION AND ACCURACY OF INTERPLANAR SPACING MEASUREMENTS

In addition to the random error, there is the potential absolute error which contributes to the overall accuracy of the interplanar spacings.

When calibrating the product Ed (which is equivalent to finding the angle θ), we use gold with a known lattice parameter of 4.0783 ± 0.0001 Å. We then obtain $Ed=42.40\pm0.02$ keV Å. Thus the fractional random error in Ed (obtained for different diffraction peaks of gold) is 0.5×10^{-4}. It is also necessary to obtain the absolute error in Ed. This occurs if the capillary containing the gold is not located at the exact position as the sample. The magnitude of this was directly measured by varying the position of the calibrant over a 400-μm total range in a direction normal to the beam and measuring the product Ed at each position x. It was found that Ed varied linearly with x, so the change in Ed is directly proportional to x according to

$$\Delta(Ed)\ (\text{keV Å})=3.8\times10^{-4}x\ \mu\text{m}\ .$$

Errors due to sample motion along the beam are much smaller and can be ignored here. In our experiments, the sample is located to within 25 μm of the calibrant center, i.e., there is an error of locating each within 12.5 μm. To be extra conservative we set $x=50\ \mu$m, so $\Delta(Ed)=\pm0.02$. Therefore the absolute error is less than $\Delta(Ed)/(Ed)=5\times10^{-4}$ combined with the random error given above, we have for the total fractional error $\Delta(Ed)/(Ed)=1\times10^{-3}$.

The total accuracy in our measurement of an individual interplanar spacing is

$$\Delta d_m/d_m=\Delta E_m/E_m+\Delta(Ed)/(Ed)\ .$$

The random error in finding a peak position is 20 eV at 20 keV, so the fractional random error $\Delta E_m/E_m$ is 1×10^{-3}.

Furthermore, changing the background parameter (going from linear to quadratic background) changes peak position by less than 10 eV or a possible systematic fractional error of 5×10^{-4}, so the total $\Delta E_m/E_m$ is 1.5×10^{-3} and the total term $\Delta(Ed)/Ed$ is 1×10^{-3}. Hence, $\Delta d_m/d_m=2.5\times10^{-3}$.

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