Effect of sample size on simulations and measurements of the phase transition in $(GaAs)_{1-x}Ge_{2x}$ and related alloys

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Monte Carlo simulations have been made of the growth of $(GaAs)_{1-x}Ge_{2x}$ in a $\langle 100\rangle$ direction by using the Davis-Holloway model with various sample sizes. Small samples give overestimates of the zinc blende order in the vicinity of the zinc blende to diamond phase transition. This is due to inadequate sampling over the mixture of phase and antiphase domains of GaAs that exists in this region. The effect is relevant to experimental determination of the critical composition at which the phase transition occurs because x-ray measurements on thin films are subject to significant limitations on sample size. Consequently experimental measurements will give overestimates of the Ge concentration at which the phase transition occurs because the zinc blende order will appear to exist beyond this point. These results also apply to other alloys between group III-V and group IV semiconductors and similar effects are to be expected with other growth directions that give a phase transition.

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I. INTRODUCTION

Metastable alloys between group III-V and group IV semiconductors have been a subject of interest, and some controversy, for more than two decades. Despite small equilibrium miscibilities,[1] macroscopically homogeneous alloys with apparently unlimited shelf life have been made by epitaxial growth of thin films using a variety of methods, i.e., pyrolytic decomposition,[2] sputtering,[3,4] and molecular-beam epitaxy[5,6] (MBE). Most of the work has been with $(GaAs)_{1-x}Ge_{2x}$,[2,5,7] and $(GaSb)_{1-x}Ge_{2x}$,[4,8] but other alloys that have been studied include $(GaAs)_{1-x}Si_{2x}$,[3,9] $(GaSb)_{1-x}Sn_{2x}$,[10] and $(GaAs)_{1-x}(Ge_{1-y}Sn_{y})_{2x}$.[11] There has been much debate[12–15] about the appropriate method for calculating the electronic band structure and the energy gap, which is tangential to the present work, and about a transition between the zinc blende (ZB) and diamond (D) structures, which is reexamined here.

GaAs and GaSb have the ZB structure that is shown in Fig. 1. The Ga and As (or Sb) atoms occupy a pair of equivalent fcc sublattices. This separation into Ga and As (or Sb) sites will be referred to as long-range order (LRO). Locally each Ga atom has four As (or Sb) nearest neighbors (NN's) and vice-versa. This local environment will be termed short-range order (SRO). The group IV semiconductors Si and Ge, and the gray allotrope of Sn have the D structure, which is like ZB except that all sites are occupied by the same kind of atom. For alloys between group III-V and and group IV semiconductors the LRO of ZB may or may not be maintained.

For simplicity, the analysis that follows will be given in terms of $(GaAs)_{1-x}Ge_{2x}$ with the proviso that it applies equally to other alloys of this kind. As a measure of the LRO we use the standard definition of an antisite fraction

$$f = \mathrm{Ga}_{\mathrm{As}}/(\mathrm{Ga}_{\mathrm{Ga}} + \mathrm{Ga}_{\mathrm{As}}), \tag{1}$$

where $\mathrm{Ga}_{\mathrm{Ga}}$ and $\mathrm{Ga}_{\mathrm{As}}$ are the numbers of Ga atoms on Ga and As sites, respectively. (An identical value arises from a definition in terms of As atoms.) The order parameter is then defined as

$$M = (1-x)(1-2f), \tag{2}$$

where the two factors arise from dilution of GaAs by Ge and the mixing of the two sublattices, respectively. (In the literature some use is made of an alternative order parameter $S = 1\text{-}2f$. Where appropriate this will be converted to M.)

X-ray measurements of $(GaSb)_{1-x}Ge_{2x}$,[16] and later of $(GaAs)_{1-x}Ge_{2x}$,[17] showed that the LRO decreased sharply to zero for $x \gtrsim 0.3$. These alloys were grown on {100}-oriented substrates, a fact whose crucial significance was overlooked in much of the early work. Newman and Dow[18] proposed an explanation in terms of a thermodynamically driven phase transition. The thermodynamic calculations were criticized,[1,19] but such points are made moot by recent work (described below) that shows thermodynamic explanations to be untenable.

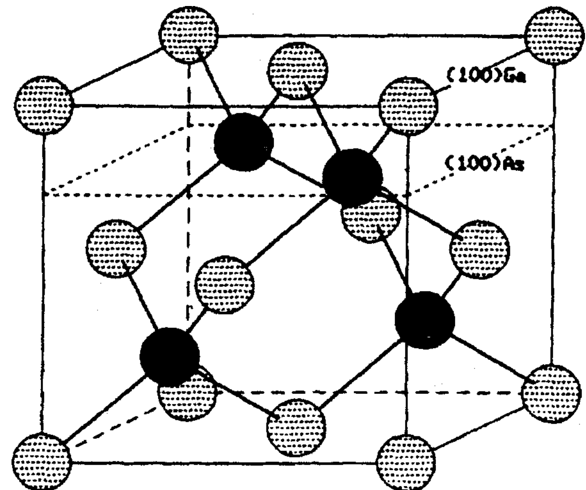

FIG. 1. The zinc-blende structure of GaAs.

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Alternative explanations for the phase transition have been based on structural models. The argument that segregation of Ga and As onto separate sublattices requires some continuity of the GaAs leads naturally to a consideration of percolation on the D lattice.[20] Percolation models[12,21] can give a phase transition, but have failed to account for the value of the critical Ge concentration, $x_c \approx 0.3$, at which the transition occurs.

A more fruitful approach is to consider stochastic growth models of which the first was proposed by Kim and Stern[22] (KS). The KS model used a computer simulation of plane-by-plane growth in the $\langle 100\rangle$ direction. Lattice sites were filled by random selection from Ge, Ga, and As atoms. Equal probabilities were assigned to the Ga and As atoms to ensure stoichiometry of the GaAs component. The KS growth rules prohibited the incorporation of Ga or As atoms at sites that would lead to Ga-Ga or As-As NN pairs. These rules are consistent with (i) the octet rule,[1] which would make Ga-Ga and As-As bonds energetically disfavored; (ii) calculations showing that substantial numbers of As-As NN's would close the energy gap in contradiction to experimental data;[12] and (iii) extended x-ray-absorption fine-structure (EXAFS) measurements[23] that demonstrate maintenance of ZB-like SRO in $(\mathrm{GaSb})_{1-x}\mathrm{Ge}_{2x}$, even at compositions where the LRO has disappeared (although the EXAFS results do not rule out the existence of some wrong NN pairs). The KS model gives a good account of $\langle 100\rangle$ growth with a phase transition at x_c in the range 0.24–0.29. Moreover, KS recognized that x_c would depend on the growth direction, with $\langle 111\rangle$ growth differing from $\langle 100\rangle$ growth. However, application of the KS growth rules to the $\langle 111\rangle$ direction gave a physically implausible segregation of the Ge atoms onto alternate $\{111\}$ planes and the model failed to yield a composition dependence of the order parameter for this growth direction. An extension of the KS model[24,25] allowed for sticking coefficients that depend on the added atom and the lattice site, although symmetry between Ga and As must be maintained to preserve GaAs stoichiometry.

An alternative growth model was proposed by Davis and Holloway[26] (DH). Again growth was plane by plane, but the choice of arriving atoms was between only Ga and Ge. Any Ga atom that was incorporated acquired an NN As atom in the following plane. This was intended to emulate MBE growth of GaAs, which is typically done with a relatively large flux of As_2 of which the excess reevaporates. As with the KS model, Ga-Ga and As-As NN pairs were forbidden. The major structural difference from the KS model is the absence of isolated Ga and As atoms in the Ge matrix. Monte Carlo (MC) simulations of $\langle 100\rangle$ growth gave $x_c \approx 0.34$ and an analytic approximation that neglected second and higher neighbor correlations and treated only Ga-Ga NN interactions gave the exact result $x_c = \frac{1}{3}$. It should be emphasized that, unlike the thermodynamic model, neither growth model involves adjustable parameters.

Application of the preceding model by Holloway and Davis[27] (HD) to growth in a $\langle 111\rangle$ direction gave the surprising prediction that in this case the phase transition would disappear. Instead the order parameter would steadily decrease with increasing Ge content to reach zero only at pure Ge. This clear-cut result was obtained both with MC simulations and with an analytic approximation. HD pointed out that, if confirmed, this dependence of the phase transition on growth direction would eliminate any possibility of an explanation in terms of bulk thermodynamic properties.

In a recent study Rodriguez *et al.*,[28,29] made x-ray measurements that confirmed the disappearance of the phase transition with $\langle 111\rangle$ growth of $(\mathrm{GaAs})_{1-x}\mathrm{Ge}_{2x}$. Further, in an extension to the previously unexplored growth directions, $\langle 211\rangle$ and $\langle 311\rangle$, which are intermediate between $\langle 111\rangle$ and $\langle 100\rangle$, their x-ray studies gave correspondingly intermediate values for x_c. Strikingly, the composition dependence of the energy gap was identical for specimens grown in four directions ($\langle 100\rangle$, $\langle 311\rangle$, $\langle 211\rangle$, and $\langle 111\rangle$) in confirmation of an earlier prediction[30] that the energy gap would be independent of the LRO. Similar results were obtained[31] for the LRO of $(\mathrm{GaAs})_{1-x}\mathrm{Si}_{2x}$ over the composition range ($x \leqslant 0.43$) where alloys were obtained.

With the DH model now firmly established it is appropriate to examine some implications of its details. One of these is the existence of phase and antiphase GaAs domains. GaAs domains have been observed experimentally in $(\mathrm{GaAs})_{1-x}\mathrm{Ge}_{2x}$,[5,32–34] and they also appear in MC simulations[22,30] using both the KS and the DH models. An analysis of the mean cluster sizes is given in DH and this has been verified by Rodriguez *et al.*[29] A feature of the DH analysis that is particularly relevant to the present paper is a steady decrease in the size of the GaAs domains as the Ge concentration is increased beyond x_c. In the following we shall consider the effect of GaAs domains on determinations of x_c both by MC simulations and by x-ray measurements.

II. MONTE CARLO SIMULATIONS

MC simulations were done using the same procedure that was described by DH. Briefly, the simulated crystal was grown by adding successive $\{100\}$ planes as follows: A randomly selected vacant site in a plane was filled by choice of either a Ge or a Ga atom with probabilities P_{Ge} and $1\text{-}P_{\mathrm{Ge}}$, respectively. If the choice was Ge the atom was added to the site. If the choice was Ga it was added along with an NN As atom in the following plane provided that this could be done without creating either Ga-Ga or As-As NN pairs. If these conditions could not be met the choice reverted to a Ge atom. This process continued until the plane was filled and was then repeated with the following plane. Identical growth rules were used by HD to implement the DH model with $\langle 111\rangle$ growth and more recently by Rodriguez *et al.*[29,31] to implement it with $\langle 211\rangle$ and $\langle 311\rangle$ growth. In all cases there was agreement between the model and x-ray determinations of the order parameter.

Simulations were done with planes of 30×30 to 100×100 atoms that had periodic boundaries along orthogonal $\langle 110\rangle$ directions. Up to 10^5 planes were grown to give sample sizes up to 10^9 atoms. In each simulation the results from the first 1000 planes were discarded to ensure attainment of a steady state. Inspection showed this to be a more than adequate

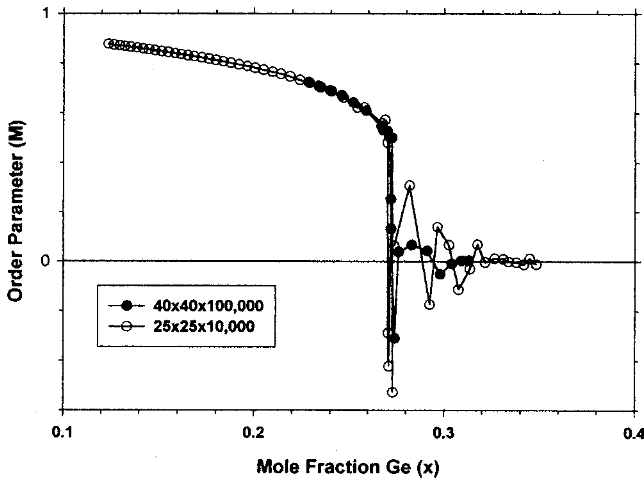

FIG. 2. Composition dependences of the order parameter M of ⟨100⟩-grown $(GaAs)_{1-x}Ge_{2x}$ from Monte Carlo simulations that used two different sample sizes.

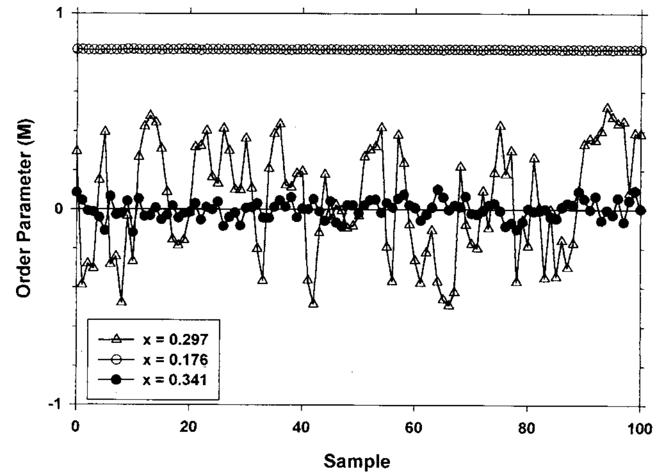

FIG. 3. Order parameter of 100 successive blocks of ⟨100⟩-grown $(GaAs)_{1-x}Ge_{2x}$ each containing 1000 {100} lattice planes. The data are shown for three different values of the Ge mole fraction x.

lead-in. From the analytic results shown in Figs. 8 and 9 of DH one would expect a steady state to be attained after growth of 30–50 planes.

III. INFLUENCE OF DOMAIN STRUCTURE

Figure 2 shows the composition dependences of the order parameter of ⟨100⟩-grown $(GaAs)_{1-x}Ge_{2x}$ that were obtained with simulations that used two different sample sizes. In each case M shows oscillations between positive and negative values when $x \approx x_c$. The oscillations damp out with increasing x and the damping is more rapid with the larger sample size. The negative values of M arise when the samples include a predominance of antiphase over phase GaAs domains and the damping arises from the progressive decrease in domain size that results in inclusion of more domains in the sample. This follows from the analysis of mean cluster size shown in Fig. 2 of DH. Further detail is shown in Fig. 3, which plots the average order for each of 100 successive blocks of 1000 {100} planes in simulations that grew samples with 10^5 planes. With x well below x_c M is essentially constant. With x in the vicinity of x_c there are wild fluctuations and for $x > x_c$ the fluctuations die out as the size of the domains relative to the sample decreases.

A cross section of a simulated alloy that shows phase and antiphase GaAs domains is given in Fig. 4. The Ge concentration here is close to x_c, but we should note that no dramatic change in appearance is observed as the value of x is increased though x_c. Instead there is a progressive decrease in domain size as x is increased beyond x_c. Again this is in accord with the analysis in DH. The lack of LRO for $x > x_c$ is a consequence of equal volumes of phase and antiphase GaAs domains, rather than of a truly random occupancy of the two fcc sublattices.

It is curious that the ZB to D transition in $(GaAs)_{1-x}Ge_{2x}$, which is driven by a stochastic growth process, mimics the fluctuations that occur in phase transitions that are thermodynamic in origin, such as critical opalescence.[35] Figure 1.5 of Ref. 35 shows domain structures that arise from a computer simulation of an Ising model that are remarkably like those in Fig. 4 of the present paper. The resemblance may be a consequence of growth and Ising models being related via formulation as cellular automata.[36]

IV. DETERMINATIONS OF THE CRITICAL COMPOSITION

At this point our study branches into two. First, we try to minimize the effect of inadequate sampling to obtain as good an estimate as possible of x_c from MC simulation. Second, we estimate the effect of the sampling limitations that are

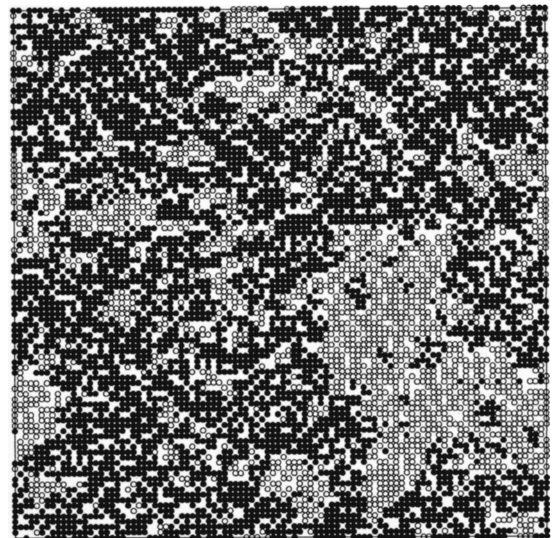

FIG. 4. A {100} plane with 100×100 atoms from a simulated $(GaAs)_{1-x}Ge_{2x}$ crystal with a Ge mole fraction $x = 0.284$. The edges are along orthogonal ⟨110⟩ directions. The filled and open circles represent atoms in phase and antiphase domains of GaAs, respectively. Ge atoms are not shown.

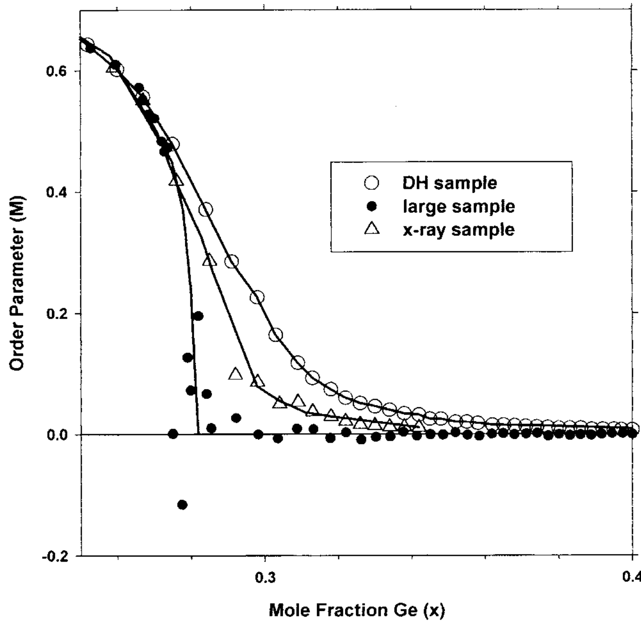

FIG. 5. Composition dependences of the order parameter M of ⟨100⟩-grown $(GaAs)_{1-x}Ge_{2x}$ in the region of the phase transition from three simulations with different sample sizes. The lines are guides for the eye. The sample sizes for the simulations are (i) large sample: 10^5 planes of up to 100×100 atoms, (ii) DH sample: rms average of 50 samples with 2000 planes of 30×30 atoms, and (iii) x-ray sample: rms average of 50 samples with 2000 planes of 80×80 atoms.

inherent in x-ray measurements of the order parameter in real, as distinct from simulated, alloys.

Improved data for M in the vicinity of x_c ($0.27\leqslant x\leqslant0.31$) were obtained using samples with 10^9 atoms (10^5 planes of 100×100 atoms). Away from the critical region sample sizes were reduced to 1.6×10^8 or 9×10^7 atoms. The results for the vicinity of x_c are given in Fig. 5, which shows the points for individual simulations in order to make evident the fluctuations that still remain in this region, even with such large sample sizes. The line through the points is a best estimate of the behavior of M without the effect of the fluctuations. This gives $x_c=0.28\pm0.01$, which is significantly smaller than the value, $x_c=0.34$, obtained earlier by DH from simulations that used much smaller samples, most of which had 2000 planes of 20×20 or 50×50 atoms. (The difference in sample sizes in the two studies reflects the increase in the speed of small computers over the past decade or so.)

To confirm that the difference between the new and previous estimates of x_c is a consequence of different sample sizes further simulations were done with samples that were typical of DH (2000 planes of 30×30 atoms). To reduce the large scatter that arises with such small samples the root-mean-square (rms) averages were taken from 50 such samples at each value of x. (In practice, the samples were taken from 50 successive 2000-plane blocks of the same simulation. This is equivalent to 50 separate simulations with different seeds for the random number generator.) These results are also shown in Fig. 5. The stripping of signs due to rms averaging has converted the damped oscillations of Fig. 2 into an exponential-like tail. The value of x_c is poorly defined because of the tail, but the results are consistent with the DH value $x_c=0.34$.

We now turn to the limitations that arise in experimental studies of the phase transition. Since alloys like $(GaAs)_{1-x}Ge_{2x}$ exist only as thin films it is evident that x-ray measurements will sample thickness of only a few thousand lattice planes. Also, while the x-ray beam will usually cover a relatively large area, the coherently illuminated regions will be much smaller. X-ray measurements of the order parameter depend on measurements of the intensities of diamond-forbidden reflections [such as (200)] for which the intensities are

$$I\propto|f_A-f_B|^2, \tag{3}$$

where f_A and f_B are the average structure factors of the atoms on the two fcc sublattices of ZB. It is easy to show that

$$M\propto f_A-f_B, \tag{4}$$

where the choice of which sublattice to call A and which to call B depends on our definition of phase and antiphase. Thus, conventional x-ray techniques[37] can determine the magnitude, but not the sign, of M. Also, since in most cases the x-ray measurement will sum the intensities from a relatively large area, the resulting value of M will be the rms average of M from many smaller coherently illuminated regions. Simulation of such an x-ray measurement may then be made using the same rms average over small samples that was described above in connection with reexamining the DH result. It remains for us to estimate the size of these small samples.

A recent study[28] of $(GaAs)_{1-x}Ge_{2x}$ used films with thicknesses of 2100–2700 Å in the vicinity of x_c, corresponding to 1500–1900 {100} lattice planes. In our simulation we shall approximate this with blocks of 2000 planes. Somewhat less well defined is the size of the coherently illuminated area, which defines the other dimensions of our sample, because this depends on details of the diffraction experiment. In the following we assume a measurement that has moderate resolution, with the proviso that different geometries will lead to somewhat different sample areas. We assume illumination by a fine-focus line source (foreshortened focus 0.04 mm $\times$8 mm) of Cu $K\alpha_1$ radiation ($\lambda=1.54$ Å and relative linewidth $\Delta\lambda/\lambda=4\times10^{-4}$) at 25 cm from the sample. The angle subtended in the line direction is assumed to be restricted to $\pm0.5°$ by a Soller slit. The measurement is assumed to be of a {200} reflection with $a_0\approx5.65$ Å.

The limitations imposed by deviation from a truly monochromatic point source reduce the coherently illuminated area to less than that of the first Fresnel zone. Along the beam direction the limitation is by the x-ray coherence length[38]

$$\ell = \lambda^2/\Delta\lambda \tag{5}$$

projected onto the specimen surface which gives $\ell \cos\theta_{\text{Bragg}} \approx 3700$ Å. In the direction perpendicular to this, coherence is limited by the angular half width of the source (a), for which we can apply Hopkins' formula[38]

$$w = 0.16\lambda/a, \tag{6}$$

which gives a coherent width of only 28 Å. For the MC simulation of the x-ray results the narrow strip that is coherently illuminated by the x-ray beam was replaced by a square region with the same area. This was done to minimize artifacts that might arise from closely spaced periodic boundaries. The coherently illuminated strip is equivalent to a 320 Å×320 Å square or 80×80 atoms with the 4.0 Å atom spacing along ⟨110⟩ directions of $(\text{GaAs})_{1-x}\text{Ge}_{2x}$.

From the preceding estimates the results of a typical x-ray study were simulated by taking the rms average of M from 50 samples of 2000 planes with 80×80 atoms at each value of x. The results of this simulation are compared with those of the previous two simulations in Fig. 5. It is evident that the spurious tail to the order parameter persists at this sample size, although it is somewhat reduced from the DH simulation by the sevenfold increase in sample area. Since simulations and x-ray measurements are both subject to sampling problems that lead to overestimates of x_c, agreement between their results can give a misleading impression of their accuracy.

Finally, Fig. 6 compares the results of the simulations (now simplified to lines) with x-ray data for $(\text{GaAs})_{1-x}\text{Ge}_{2x}$, $(\text{GaSb})_{1-x}\text{Ge}_{2x}$, and $(\text{GaAs})_{1-x}\text{Si}_{2x}$. The experimental data have much scatter, but within this limitation the three alloys appear to behave identically. (This observation alone would suffice to cast doubt on a thermodynamic origin for the phase transition.) To the extent that the scatter allows a comparison, the data appear to favor the simulations with limited sample sizes and give a rather better fit to the 30×30 atom area of the DH calculation. This suggests that the coherently illuminated area has been somewhat overestimated. (The data for $(\text{GaSb})_{1-x}\text{Ge}_{2x}$ from Ref. 16 were obtained with Mo$k\alpha_1$ radiation for which $\lambda = 0.71$ Å. This would reduce the coherently illuminated area by about a factor of 4 and move the simulated x-ray results closer to the DH simulation.)

The experimental limitations on x-ray sample size might be somewhat eased by using thicker films. However, the 10^5 planes that were used for the large-sample simulations here correspond to a $(\text{GaAs})_{1-x}\text{Ge}_{2x}$ thickness of 14 μm, which seems to be impracticably large, quite apart from the additional limitations that are imposed by primary extinction and the x-ray coherence length. From this we conclude that x-ray measurements of x_c will inevitably give an overestimate because of inadequate sampling over phase and antiphase domains. The persistence of GaAs domains at $x > x_c$ might also account for the observation of residual ZB order in this region from measurements of He^+ channeling.[39]

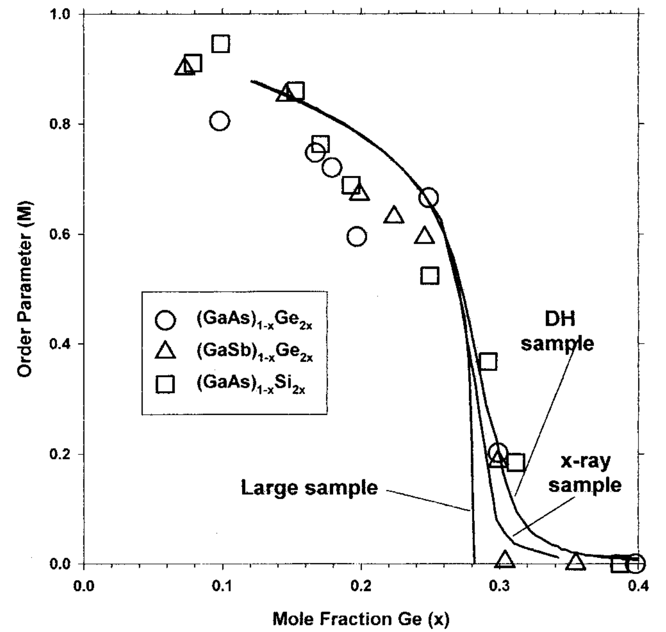

FIG. 6. Experimental values of the order parameter M of $(\text{GaAs})_{1-x}\text{Ge}_{2x}$ (Ref. 30), $(\text{GaSb})_{1-x}\text{Ge}_{2x}$ (Ref. 16), and $(\text{GaAs})_{1-x}\text{Si}_{2x}$ (Ref. 31) compared with the results of simulations. The sample sizes for the simulations are (i) large sample: 10^5 planes of up to 100×100 atoms, (ii) DH sample: rms average of 50 samples with 2000 planes of 30×30 atoms, and (iii) x-ray sample: rms average of 50 samples with 2000 planes of 80×80 atoms.

V. CONCLUSIONS

The phase transition in ⟨100⟩-grown $(\text{GaAs})_{1-x}\text{Ge}_{2x}$ and related alloys, although driven by a stochastic growth process, still exhibits critical fluctuations akin to those associated with phase transitions that are thermodynamic in origin. Large fluctuations near the phase transition lead to a sampling problem that limits the precision with which the critical composition for the phase transition may be determined. X-ray measurements of the order parameter will give overestimates of the Ge concentration at which the phase transition occurs. The results are also applicable to other alloys between group III-V and group IV semiconductors and a similar effect is to be expected for these alloys with other growth directions that exhibit a phase transition.

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