High-pressure behavior of beta-Ga2O3 nanocrystals

Wang, H.; He, Y.; Chen, W.; Zeng, Y.W.; Ståhl, Kenny; Kikegawa, T.; Jiang, J.Z.

Published in:
Journal of Applied Physics

Link to article, DOI:
10.1063/1.3296121

Publication date:
2010

Document Version
Publisher's PDF, also known as Version of record

High-pressure behavior of β-Ga_2O_3 nanocrystals

H. Wang,[1] Y. He,[1] W. Chen,[1] Y. W. Zeng,[1,2] K. Stahl,[3] T. Kikegawa,[4] and J. Z. Jiang[1,a)]
[1]*International Center for New-Structured Materials (ICNSM) and Laboratory of New-Structured Materials, Department of Materials Science and Engineering, Zhejiang University, Hangzhou 310027, People's Republic of China*
[2]*Center of Analysis and Measurement, Zhejiang University, Hangzhou 310027, People's Republic of China*
[3]*Department of Chemistry, Technical University of Denmark, DK-2800 Lyngby, Denmark*
[4]*Photon Factory, Institute of Materials Structure Science, High Energy Accelerator Research Organization, 1-1 Oho, Tsukuba, Ibaraki 305-0801, Japan*

(Received 8 October 2009; accepted 29 December 2009; published online 9 February 2010)

Freestanding nanocrystalline β-Ga_2O_3 particles with an average grain size of 14 nm prepared by chemical method was investigated by angle-dispersive synchrotron x-ray diffraction in diamond-anvil cell up to 64.9 GPa at ambient temperature. The evolution of x-ray diffraction patterns indicated that nanocrystalline monoclinic β-Ga_2O_3 underwent a phase transition to rhombohedral α-Ga_2O_3. It was found that β- to α-Ga_2O_3 transition began at about 13.6–16.4 GPa, and extended up to 39.2 GPa. At the highest pressure used, only α-Ga_2O_3 was present, which remained after pressure release. A Birch–Murnaghan fit to the P-V data yielded a zero-pressure bulk modulus at fixed $B_0'=4$: $B_0=228(9)$ GPa and $B_0=333(19)$ GPa for β-Ga_2O_3 and α-Ga_2O_3 phases, respectively. We compared our results with bulk β-Ga_2O_3, and concluded that the phase-transition pressure and bulk modulus of nanocrystalline β-Ga_2O_3 are higher than those of bulk counterpart.
 [doi:10.1063/1.3296121]

I. INTRODUCTION

Monoclinic gallium oxide (β-Ga_2O_3), a wide-band-gap semiconductor ($E_g=4.9$ eV), exhibits unique conduction and luminescence properties and thus attracts much research interest due to numerous technological application.[1–5] Recently, considerable effort has been devoted to the study of nanostructured β-Ga_2O_3 because of the potential for applications in nanodevices.[6–10] In ambient conditions, monoclinic β-Ga_2O_3 is the thermodynamically stable form and there are other four metastable structures: α-, γ-, δ-, and ε-Ga_2O_3.[11] Different forms have different physical properties. For example, the band gap of the α-Ga_2O_3 polymorph is 2.41 eV, much narrower than that of β-Ga_2O_3.[12] So it is important to clarify the relationship between different structures, which can be accessed by research on high-pressure phase transition. Especially for nanocrystal materials, investigation of the high-pressure behavior reveals the contribution of boundary in phase transition and provides additional insights into size-dependent changes in phase stability and mechanical properties.

The high-pressure behavior of β-Ga_2O_3 had not received much attention until recently.[13–21] It is verified that β-Ga_2O_3 undergoes a sluggish transition to α-Ga_2O_3 under high pressure. However, there is still a controversy on the value of the transition pressure. Machon *et al.*[15] indicated that β- to α-Ga_2O_3 phase transition occurred at 20–22 GPa, while Lipinska-Kalita *et al.*[16] found that transition began at 6.5–7 GPa at ambient temperature. Theoretical calculations based on density-functional theory gave the transition pressure of 2.6 GPa (Ref. 18) when the d electron of Ga was treated as core state, and of 6 GPa (Ref. 18) and 9.5 GPa (Ref. 19) when the d electron of Ga as valence state. Lipinska-Kalita *et al.*[16] also reported that the transition pressure with and without transmitting medium were 6.5–7 and 3 GPa, respectively.

Research on high-pressure phase transition of nanocrystalline β-Ga_2O_3 can only be found in the work of Lipinska-Kalita *et al.*[21] They reported the β- to α-Ga_2O_3 transition, beginning at 6 GPa, in nanocrystalline β-Ga_2O_3 embedded in an amorphous silica matrix. However, they also mentioned that the silica glass matrix undergoes structural and density changes within this pressure range,[22,23] so that it is not yet known if the structural changes are intrinsic to nanocrystalline β-Ga_2O_3, or are promoted by anomalous densification among the silica matrix. These results encourage us to re-examine the high-pressure behavior of freestanding β-Ga_2O_3 nanoparticles by angle-dispersive x-ray diffraction (XRD) technique.

II. EXPERIMENTS

Monoclinic β-Ga_2O_3 nanoparticles were synthesized as follows: 0.7 g $GaCl_3$, 0.8 g NaOH and 4 ml PEG (Mw 400) were first dissolved in 10 ml ethanol, stirred with a magnetic stirrer. The precursory solution was stirred at 60 °C for 1 h to yield a transparent solution in an alumina crucible. The solution in the alumina crucible was then heated in a furnace at 900 °C for 3 h. The powder was white after annealing treatment. Finally, the white powder was washed using alcohol and centrifuged for three times, and then dried at 70 °C. The average crystallite size of Ga_2O_3 nanoparticles was determined by XRD with a Cu $K\alpha$ radiation and by transmission electron microscopy (TEM).

In situ angle-dispersive XRD measurements of nanocrystalline β-Ga_2O_3 were performed at beam line BL-13A in High Energy Accelerator Research Organization (KEK).

a)Electronic mail: jiangjz@zju.edu.cn.

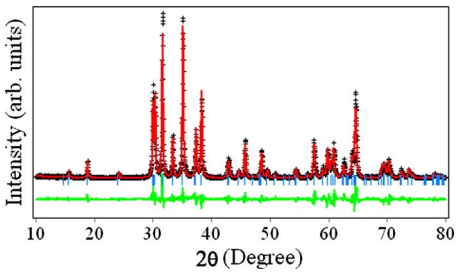

FIG. 1. (Color online) Full profile Rietveld refinement of diffraction pattern for nanocrystalline β-Ga_2O_3. Asterisks, upper, and lower solid lines represent experimental, calculated, and difference pattern, respectively. Bars are marked at the positions of diffraction peaks.

Monochromatic x-ray (λ=0.42713 Å) was collimated to 50×50 μm^2 and irradiated at the center of the sample. Diffraction patterns were collected by image plate detector. The samples were compressed into a 120 μm diameter hole drilled in a T301 stainless steel gasket of a Mao-Bell diamond-anvil cell. A mixture of methanol, ethanol, and water in the volume ratio of 16:3:1 was used as the pressure-transmitting medium. The pressures were determined by the ruby fluorescence method.[24]

III. RESULTS AND DISCUSSION

Figure 1 shows the XRD pattern of as-prepared Ga_2O_3 nanoparticles which was used to identify the structure and to estimate the mean grain size. A Rietveld full profile refinement[25] confirmed that all the peaks in the XRD pattern corresponded to the monoclinic β-Ga_2O_3 (space group $C2/m$). No peaks of any other phases were detected. The refined β-Ga_2O_3 lattice parameters and cell volume were a=12.233(3) Å, b=3.038(5) Å, c=5.806(4) Å, β =103.836(4)°, and V=209.51(4) $Å^3$, in agreement with the values of bulk sample [a=12.233(10) Å, b=3.038(3) Å, c =5.807(5) Å, β=103.821(7)°, and V=209.56(4) $Å^3$].[16] The average grain size of nanocrystalline β-Ga_2O_3 was estimated to be about 14 nm using the Scherrer formula, consistent with the average value of the grain size discerned in the TEM micrograph of Fig. 2(a). Each nanocrystal was roughly spherical and almost composed of a single-crystalline domain, as shown in Fig. 2.

Synchrotron XRD patterns of 14 nm β-Ga_2O_3 nanocrystal in the compression up to 64.9 GPa and the following decompression down to ambient pressure at room temperature were collected to analyze the pressure-induced phase transitions and to retrieve the isothermal bulk modulus. Some selected XRD patterns, which are characteristic of the whole evolution, are shown in Fig. 3. Diffraction line broadening at high pressure might be originated from nonhydrostaticity caused by solidification of pressure-transimitting medium at pressures above about 16 GPa. The diffraction patterns collected at low pressures correspond well to the monoclinic β-Ga_2O_3 phase. With increasing pressure, all diffraction peaks systematically shifted toward higher 2θ angles, and some peaks disappeared while new lines emerged. At 16.4 GPa, a new peak [arrowed in Fig. 3(a)] appeared, indicating occurrence of a phase transition. The phase transition proceeded slowly, and above 39.2 GPa, only peaks from the high-pressure phase remained to the highest pressure used in this study. The high-pressure diffraction patterns could be indexed within the $R\bar{3}c$ space group expected for the rhombohedral α-Ga_2O_3, in accordance with former results.[15,16,21]

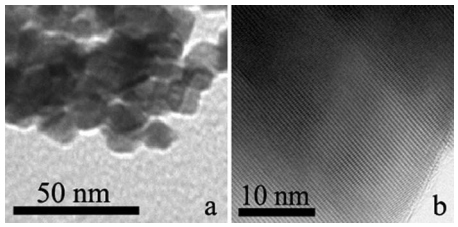

FIG. 2. Transmission electron microscopic patterns of nanocrystalline β-Ga_2O_3: (a) low magnification image and (b) HRTEM image.

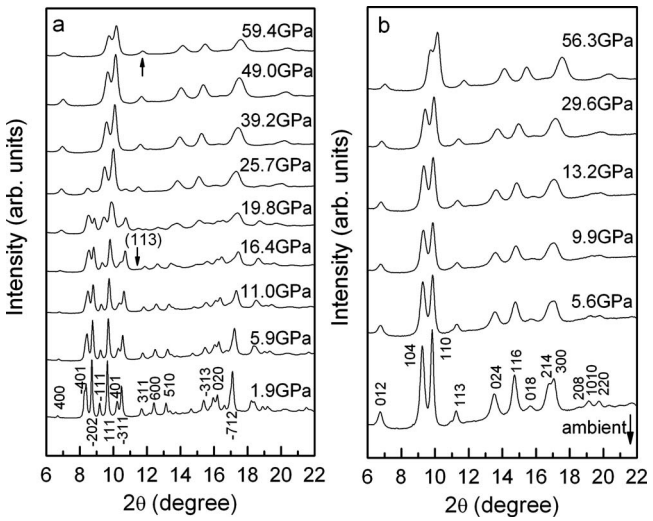

FIG. 3. Selected angle-dispersive synchrotron XRD patterns: (a) compression up to 64.9 GPa and (b) subsequent decompression. The appearance of the (113) peak for α-Ga_2O_3 is arrowed.

At the beginning of phase transition, the (104) and (110) peaks, two strong reflections of α-Ga_2O_3, were overlapped with the ($\bar{1}11$) and (111) peaks of the still dominating β-Ga_2O_3, so the onset pressure of phase transition is imprecisely estimated only according to the appearance of the (113) peak. In order to confirm the pressure range of the β- to α-Ga_2O_3 phase transition and to determine the lattice parameters, Rietveld refinements were performed on all diffraction patterns recorded during compression and subsequent decompression. The evolution of weight percent for β-Ga_2O_3 and α-Ga_2O_3 with pressure is presented in Fig. 4. It is clearly shown that the weight percent of β-Ga_2O_3 keeps close to 100% below 13.6 GPa. For the pattern at 16.4 GPa, the Rietveld refinement confirmed that β- and α-Ga_2O_3 phases coexisted with a ratio of 89.7:10.3 in wt %. It is obvious that the phase transition begins in the range of 13.6–16.4 GPa. The β-Ga_2O_3 was the dominating phase up to 19.8 GPa, and tiny β-Ga_2O_3 could still be detected at 36.8 GPa, indicating a sluggish transition in a wide pressure range of 13.6–39.2 GPa similar to the range of 6.5–40 GPa for bulk Ga_2O_3.[16]

All patterns collected from the decompression sequence only attribute to α-Ga_2O_3, and the gradual shift of diffraction

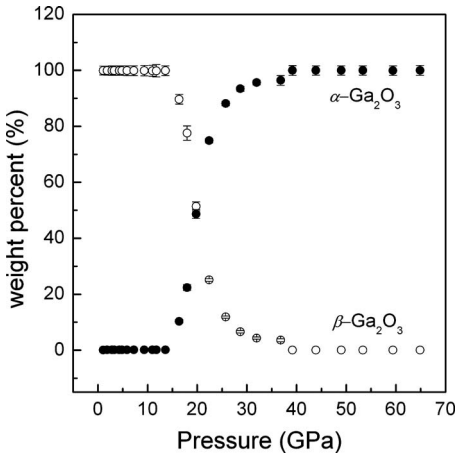

FIG. 4. The evolution of wight percent for β-Ga_2O_3 and α-Ga_2O_3 with pressure during compression.

lines toward lower 2θ angles indicates a progressive relaxation of the α-Ga_2O_3 structure, as shown in Fig. 3(b). That demonstrates the pressure-driven β- to α-Ga_2O_3 phase transition is irreversible within the decompression time scale used here. Figure 5 illustrates Rietveld full profile refinements of the diffraction patterns of α-Ga_2O_3 at 64.9 GPa and ambient pressure. The refinements yielded the following lattice parameters of α-Ga_2O_3: a=4.7807(9) Å, c =12.4743(29) Å at 64.9 GPa and a=4.9723(6) Å, c =13.4031(17) Å at ambient pressure, respectively. The ambient lattice parameters of α-Ga_2O_3 correspond well with the results of Lipinska-Kalita *et al.*[16] [a=4.979(10) Å and c =13.432(28) Å].

Table I presents onset pressures of β- to α-Ga_2O_3 phase

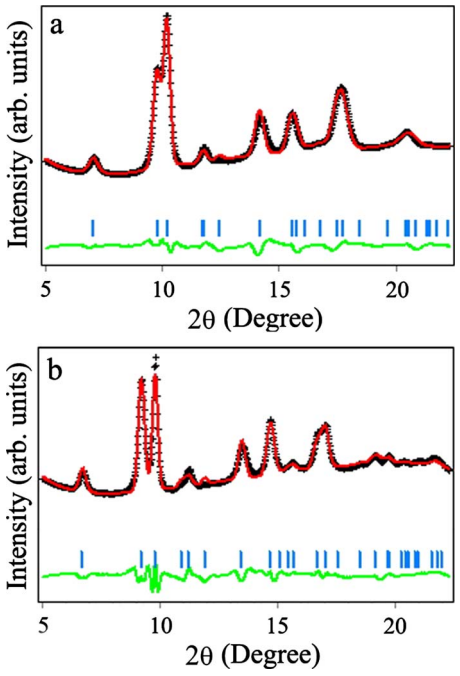

FIG. 5. (Color online) Full profile Rietveld refinements of diffraction patterns for α-Ga_2O_3: (a) at 64.9 GPa and (b) ambient pressure. Asterisks, upper, and lower solid lines represent experimental, calculated, and difference pattern, respectively. Bars are marked at the positions of diffraction peaks.

transition in our work and others. We found that the pressure-induced phase transition for freestanding β-Ga_2O_3 nanocrystal began within 13.6–16.4 GPa, higher than the previous result of 6 GPa for nanocrystalline β-Ga_2O_3 embedded in amorphous matrix.[21] It indicated that the host glass matrix could promote the occurrence of the β- to α-Ga_2O_3 phase transition. The glass matrix would introduce the anisotropic stress on the nanocrystalline β-Ga_2O_3 embedded in it. The anisotropic stress is supposed to play an important role in the β- to α-Ga_2O_3 phase transition. It was also proved by the Lipinska-Kalita *et al.*,[16] who found that the transition pressure was 3 GPa without transmitting medium, lower than 6.5–7 GPa with nitrogen as a pressure medium. In addition, the pressure-induced coordination change and densification of the host glass matrix could also influence the phase transition, as mentioned in Ref. 21.

As to onset pressure of bulk β- to α-Ga_2O_3 transition, experimental data reported in recent years can be divided into two ranges of 3.3–7 and 20–22 GPa, respectively (Table I). Our result just exists between these two ranges. Thus it is difficult to judge the influence of grain size on the transition pressure. Jamieson[26] founded a relationship between transition pressure P_{tr} and energy band gap at ambient pressure E_g for semiconductors: $P_{tr}\Delta V \propto E_g$, where ΔV is the volume deviation between two phases at P_{tr}. From transmission spectrum collected at ambient condition (Fig. 6), we obtained the band gap of 14 nm β-Ga_2O_3 as 5.9 eV, which is higher than 4.9 eV for bulk β-Ga_2O_3. By substituting E_g and ΔV of bulk and nanocrystalline β-Ga_2O_3 into Jamieson's function, it is deduced that the transition pressure P_{tr} for the nanocrystalline sample is higher than that for the bulk one. Based on this analysis as well as theoretical calculation results of 2.6–9 GPa for the phase transition pressure of β- to α-Ga_2O_3 (Table I), it is more reasonable to consider the onset pressure of phase transition for bulk sample in the range of 3.3–7 GPa. Therefore, we can conclude that the phase transition pressure of freestanding nanocrystalline β-Ga_2O_3 is higher than that of bulk material, consistent with the results of other nanocrystalline materials.[27–30]

The evolution of the lattice parameters with pressure for β-Ga_2O_3 and α-Ga_2O_3 is presented in Fig. 7, and the linear compressibilities are summarized in Table II. The lattice compression of both gallium oxide phases is anisotropic, with the a and b axes more compressible than the c axis in the monoclinic β-Ga_2O_3 structure, and with the c axis more compressible than the a axis in the rhombohedral α-Ga_2O_3 structure. The monoclinic angle decreased with increasing pressure, in agreement with the tendency in Ref. 15. The compressibility values along the b and c axes of β-Ga_2O_3 in our study are comparable with those obtained previously for bulk β-Ga_2O_3 (Ref. 15) and for nanocrystalline samples embedded in amorphous silica.[21] However, the a axis compression is only comparable with that of bulk β-Ga_2O_3,[15] while twice as large as that of nanocrystalline samples embedded in amorphous silica. Such difference in compressibility along the a axis might be explained by the possibility that host glass matrix surrounding the β-Ga_2O_3 nanocrystal could accommodate and relax the strains. The ratio of c/a for the

TABLE I. Comparison of bulk modulus B_0 and unit-cell volume V_0 of β-Ga_2O_3 and α-Ga_2O_3 at ambient conditions, and onset pressure of β-,to α-Ga_2O_3 transition in our study with those reported.

		β-Ga_2O_3			α-Ga_2O_3			
Sample	Beginning of phase transition	B_0 (GPa)	B_0'	V_0 (Å^3)	B_0 (GPa)	B_0'	V_0 (Å^3)	Method
14 nm[a]	13.6–16.4 GPa	193(6)	9.8(9)	207.0(8)	354(22)	3.0(4)	287.0(7)	ADX
		228(9)	4	207.0(8)	333(19)	4	287.0(7)	
14.8 nm[b]	6 GPa	191(5)	8.3(9)	209.5(3)				EDX
Bulk[c]	6.5–7 GPa	199(6)	3.1(4)	209.1(2)	220(9)	5.9(6)	288(1)	ADX
		184(3)	4	209.4(2)	252(14)	4	288(1.5)	
	3.3GPa	255(16)	7.63(90)	209.9(4)	261(20)	7.93(71)	288.8(1)	
Bulk[d]	7.6 GPa, 1500 K	134(12)	4	209.24	223(2)	4	288.96(24)	ADX
		142	4.1	204.56	243	4.0	283.44	DFT
Bulk[e]	20–22 GPa	202(7)	2.4(6)					ADX
Bulk[f]	9.5	174	3.79		210	4.95		DFT
Bulk[g]	2.6 or 6 GPa							DFT

[a]This study.
[b]Reference 21.
[c]Reference 16.
[d]Reference 17.
[e]Reference 15.
[f]Reference 19.
[g]Reference 18.

α-Ga_2O_3 linearly decreased during compression and then increased from 2.609 at 64.9 GPa to 2.696 at ambient pressure during decompression.

The V/V_0 relations as a function of pressure for nanocrystalline β-Ga_2O_3 and α-Ga_2O_3 during compression are shown in Fig. 8. At 16.4 GPa, the volume collapse across the phase transition was about 6.5%. The P-V data were fitted using a third-order Birch–Murnaghan equation of state

$$P = 1.5B_0[(V/V_0)^{-7/3} - (V/V_0)^{-5/3}] \times \{1 + 0.75(B_0' - 4) \times [(V/V_0)^{-2/3} - 1]\},$$

where V_0 is the volume at ambient pressure, B_0 is the bulk modulus at ambient pressure, and B_0' is the pressure derivative at ambient pressure. The fitting parameters B_0 and B_0' are summarized in Table I, where reported data are also presented. Since the value of B_0' differs largely between the two phases, we fitted again the compression data by fixing B_0' =4 to give a better idea of the relative compressibility between β-Ga_2O_3 and α-Ga_2O_3 phases. It should be pointed out that the data of minor phase while two phases coexist, α-Ga_2O_3 phase at low pressure and β-Ga_2O_3 phase at high pressure, were not fitted because of their large uncertainty.

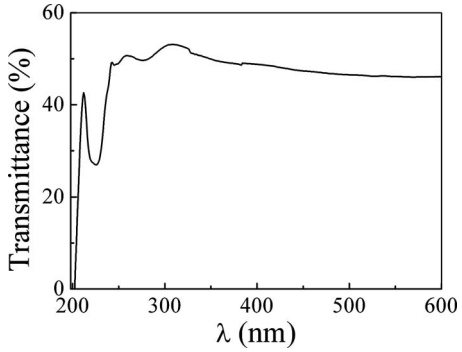

FIG. 6. The optical transmission spectrum of 14 nm β-Ga_2O_3 at ambient condition.

As shown in Table I, the bulk modulus of 14 nm β-Ga_2O_3 in our work is close to that of nanocrystalline sample embedded in glass matrix and slightly higher than

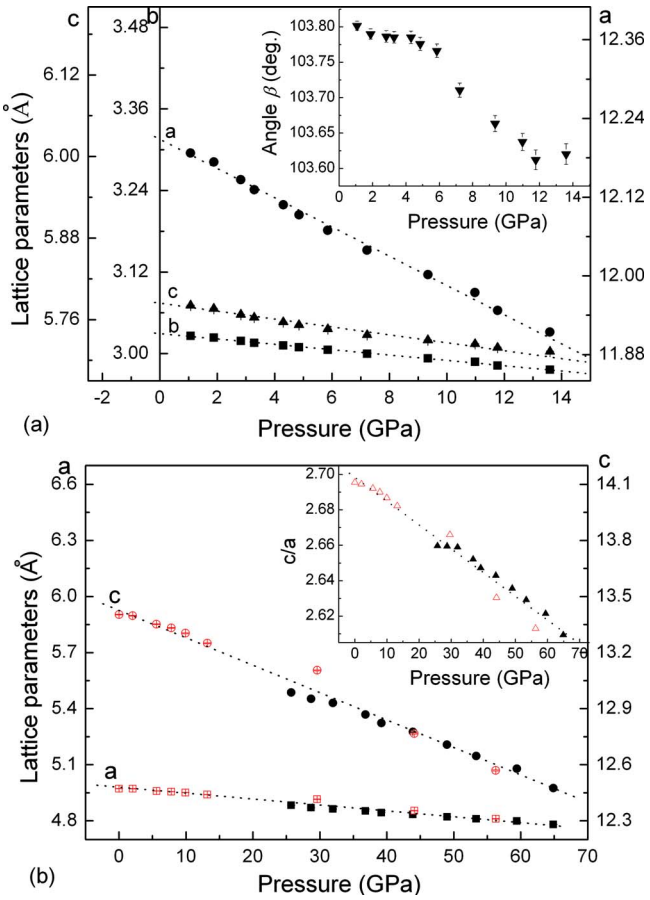

FIG. 7. (Color online) Pressure evolution of lattice parameters: (a) of the β-Ga_2O_3, and (b) of the α-Ga_2O_3. Solid and open symbols refer to the compression and decompression data, respectively. The differential (in angstrom) of the vertical scales of each graph is the same, in order to facilitate comparison between the evolutions of lattice parameters. Dot lines are drawn as a guide for the eyes.

TABLE II. Comparison of linear compressibilities of β-Ga_2O_3 and α-Ga_2O_3 in our study with data reported.

Linear compressibility	14 nm[a]		14.8 nm[b]	Bulk[c]
	α-Ga_2O_3	β-Ga_2O_3	β-Ga_2O_3	β-Ga_2O_3
$\beta_a(GPa^{-1})$	$6.21(5)\times10^{-4}$	$1.78(7)\times10^{-3}$	8.2×10^{-4}	$1.99(9)\times10^{-3}$
$\beta_b(GPa^{-1})$		$1.30(6)\times10^{-3}$	1.97×10^{-3}	$1.45(2)\times10^{-3}$
$\beta_c(GPa^{-1})$	$1.10(3)\times10^{-3}$	$9.34(9)\times10^{-4}$	8.8×10^{-4}	$8.8(4)\times10^{-4}$

[a]This study.
[b]Reference 21.
[c]Reference 15.

that of bulk β-Ga_2O_3, while bulk modulus of α-Ga_2O_3 in our work is much higher than that of the bulk material which might be connected with the high surface energy of α-Ga_2O_3. Further investigation is required to get a better understanding of this elevation.

IV. CONCLUSIONS

The high-pressure behavior of 14 nm β-Ga_2O_3 particles was investigated using angle-dispersive synchrotron XRD up to 64.9 GPa. It was found that the onset transition pressure of β- to α-Ga_2O_3 was within 13.6–16.4 GPa, and phase transition was sluggish and irreversible with α-Ga_2O_3, remaining after pressure release. Based on the first principles calculations reported and the relationship between the transition pressure and energy gap, we conclude that the transition pressure of nanocrystalline β-Ga_2O_3 is higher than that of bulk counterpart. According to the hydrostatic compression data, the bulk modulus at a fixed $B_0'=4$ of nanocrystalline β-Ga_2O_3 and α-Ga_2O_3 was estimated at 228(9) GPa and 333(19) GPa, both higher than the bulk materials, respectively.

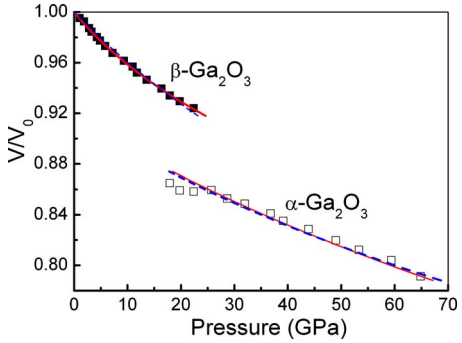

FIG. 8. (Color online) The evolution of V/V_0 for β-Ga_2O_3 and α-Ga_2O_3 with pressure. Solid lines and dashed lines are the Birch–Murnaghan equation of state fits to experimental data with unfixed B_0', and with fixed $B_0'=4$, respectively.

ACKNOWLEDGMENTS

The authors would like to thank the KEK, Japan, for the assistance during the high-pressure x-ray diffraction measurements. Financial support from the National Natural Science Foundation of China (Grant Nos. 50701038, 60776014, 60876002, 10804096, 50920105101, and 10979002), Zhejiang University-Helmholtz cooperation fund, the Ministry of Education of China (Program for Changjiang Scholars and the Research Fund for the Doctoral Program of Higher Education), the Department of Science and Technology of Zhejiang Province, and Zhejiang University is gratefully acknowledged.

[1]L. Binet and D. Gourier, J. Phys. Chem. Solids **59**, 1241 (1998).
[2]M. Ogita, N. Saika, Y. Nakanishi, and Y. Hatanaka, Appl. Surf. Sci. **142**, 188 (1999).
[3]M. Passlack, E. F. Schubert, W. S. Hobson, M. Hong, N. Moriya, S. N. G. Chu, K. Konstadinidis, J. P. Mannaerts, M. L. Schnoes, and G. J. Zydzik, J. Appl. Phys. **77**, 686 (1995).
[4]T. Miyata, T. Nakatani, and T. Minami, Thin Solid Films **373**, 145 (2000).
[5]Z. Li, C. de Groot, and J. H. Moodera, Appl. Phys. Lett. **77**, 3630 (2000).
[6]Y. C. Choi, W. S. Kim, Y. S. Park, S. M. Lee, D. J. Bae, Y. H. Lee, G. S. Park, W. B. Choi, N. S. Lee, and J. M. Kim, Adv. Mater. **12**, 746 (2000).
[7]R. Rao, A. M. Rao, B. Xu, J. Dong, S. Sharma, and M. K. Sunkara, J. Appl. Phys. **98**, 094312 (2005).
[8]C. H. Liang, G. W. Meng, G. Z. Wang, Y. W. Wang, L. D. Zhang, and S. Y. Zhang, Appl. Phys. Lett. **78**, 3202 (2001).
[9]S. Sharma and M. K. Sunkara, J. Am. Chem. Soc. **124**, 12288 (2002).
[10]J. S. Kim, H. E. Kim, H. L. Park, and G. C. Kim, Solid State Commun. **132**, 459 (2004).
[11]R. Roy, V. G. Hill, and E. F. Osborn, J. Am. Chem. Soc. **74**, 719 (1952).
[12]H. G. Kim and W. T. Kim, J. Appl. Phys. **62**, 2000 (1987).
[13]T. P. Beales, C. H. L. Goodman, and K. Scarrott, Solid State Commun. **73**, 1 (1990).
[14]B. Tu, Q. Cui, P. Xu, X. Wang, W. Gao, C. Wang, J. Liu, and G. Zou, J. Phys.: Condens. Matter **14**, 10627 (2002).
[15]D. Machon, P. F. McMillan, B. Xu, and J. Dong, Phys. Rev. B **73**, 094125 (2006).
[16]K. E. Lipinska-Kalita, P. E. Kalita, O. A. Hemmers, and T. Hartmann, Phys. Rev. B **77**, 094123 (2008).
[17]H. Yusa, T. Tsuchiya, N. Sata, and Y. Ohishi, Phys. Rev. B **77**, 064107 (2008).
[18]P. Kroll, Phys. Rev. B **72**, 144407 (2005).
[19]H. He, R. Orlando, M. A. Blanco, R. Pandey, E. Amzallag, I. Baraille, and M. Rerat, Phys. Rev. B **74**, 195123 (2006).
[20]R. Caracas and R. E. Cohen, Phys. Rev. B **76**, 184101 (2007).
[21]K. E. Lipinska-Kalita, B. Chen, M. B. Kruger, Y. Ohki, J. Murowchick, and E. P. Gogol, Phys. Rev. B **68**, 035209 (2003).
[22]M. Grimsditch, Phys. Rev. Lett. **52**, 2379 (1984).
[23]C. Meade, R. J. Hemley, and H. K. Mao, Phys. Rev. Lett. **69**, 1387 (1992).
[24]H. K. Mao, P. M. Bell, J. W. Shaner, and D. J. Steinberg, J. Appl. Phys. **49**, 3276 (1978).
[25]H. M. Rietveld, J. Appl. Crystallogr. **2**, 65 (1969).
[26]J. C. Jamieson, Science **139**, 845 (1963).
[27]S. H. Tolbert and A. P. Alivisatos, Science **265**, 373 (1994).
[28]S. B. Qadri, J. Yang, B. R. Ratna, E. F. Skelton, and J. Z. Hu, Appl. Phys. Lett. **69**, 2205 (1996).
[29]J. Z. Jiang, L. Gerward, D. Frost, R. Secco, J. Peyronneau, and J. S. Olsen, J. Appl. Phys. **86**, 6608 (1999).
[30]Y. He, J. F. Liu, W. Chen, Y. Wang, H. Wang, Y. W. Zeng, G. Q. Zhang, L. N. Wang, J. Liu, T. D. Hu, H. Hahn, H. Gleiter, and J. Z. Jiang, Phys. Rev. B **72**, 212102 (2005).