



Step-and-terrace surface formation on (-112) β -Ga₂O₃ via thermal annealing

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Step-and-terrace formation on a (-112) β -Ga₂O₃ substrate subjected to chemical mechanical polishing was investigated by thermal annealing and subsequent wet-chemical treatments. Atomic force microscopy revealed that annealing at approximately 1000 °C produced a well-defined stepped morphology with predominantly monolayer steps about 0.2 nm high, consistent with the 0.210 nm spacing between adjacent (-112) planes. In contrast, treatments with hydrofluoric acid, a sulfuric acid–hydrogen peroxide mixture, and tetramethylammonium hydroxide degraded the thermally formed stepped morphology. These results demonstrate that thermal annealing is effective for preparing ordered (-112) β -Ga₂O₃ surfaces and provide guidance for future pre- and post-treatment processes. © 2026 The Author(s). Published on behalf of The Japan Society of Applied Physics by IOP Publishing Ltd

β -Ga₂O₃ has attracted considerable attention as an ultrawide-bandgap semiconductor because of its wide bandgap, high critical electric field, and availability of large bulk single crystals.¹⁾ Various epitaxial growth and device-processing techniques have therefore been developed.¹⁾ However, β -Ga₂O₃ has a monoclinic crystal structure, and its growth, etching, and surface properties strongly depend on the crystallographic orientation.^{2–5)} The selection and control of the substrate surface are therefore important for establishing reliable material-processing methods.

Surface morphology is particularly important because atomically smooth surfaces with ordered steps and terraces are desirable for epitaxial growth and subsequent device fabrication. Detailed surface structures, including steps and facets, can influence surface reaction kinetics, adatom incorporation, and impurity incorporation. In addition, the actual surface after epitaxial growth and material processing may consist of crystallographic facets different from the nominal substrate plane.^{6–8)} Surface preparation has therefore been investigated particularly for the commonly used (100), (010), (001), and $(\bar{2}01)$ β -Ga₂O₃ substrates. Thermal annealing at approximately 1000 °C has been reported to produce a distinct step-and-terrace surface morphology on the (100) substrate.⁹⁾ In contrast, annealing of the (010), (001), and $(\bar{2}01)$ substrates has been reported to result in the development of facets other than the nominal substrate planes.^{1,10,11)} Wet-chemical treatments have been reported to produce well-defined step-and-terrace morphologies on the (001) and $(\bar{2}01)$ surfaces,^{10,12)} whereas facets other than the nominal plane develop on the (010) surface.³⁾

Recently, the $(\bar{1}12)$ plane has been proposed as a fundamental orientation of β -Ga₂O₃ based on its pseudo-cubic oxygen sublattice framework.^{13,14)} In this framework, the $(\bar{1}12)$ and (100) planes belong to the same {100} family of the pseudo-cubic cell,¹³⁾ suggesting that thermal annealing may also produce an ordered step-and-terrace morphology on the $(\bar{1}12)$ surface, similar to that observed on the (100) surface as described above. Furthermore, our previous study demonstrated twin-free homoepitaxy on a $(\bar{1}12)$ substrate, producing an atomically smooth step-and-terrace surface, indicating the

potential of this orientation for epitaxial growth and subsequent device processing.¹⁴⁾

However, suitable surface-preparation methods for $(\bar{1}12)$ β -Ga₂O₃ substrates have not yet been established. In particular, the effects of thermal annealing and commonly used wet-chemical treatments on the surface morphology remain unclear. Therefore, in this study, we investigated step-and-terrace formation on a $(\bar{1}12)$ β -Ga₂O₃ substrate by thermal annealing and evaluated the effects of subsequent wet-chemical treatments.

We used a custom-made Sn-doped $(\bar{1}12)$ β -Ga₂O₃ substrate with a nominal carrier concentration of $5 \times 10^{18} \text{ cm}^{-3}$, supplied by Novel Crystal Technology. Note that, after chemical mechanical polishing (CMP), the substrate was cleaned with organic solvents (acetone, isopropanol, and methanol), hydrofluoric acid (HF), and a sulfuric acid–hydrogen peroxide mixture (SPM).

The as-received substrate was thermally annealed in a custom-built horizontal quartz furnace under flowing N₂ (>99.99995%) at a flow rate of 2.00 slm and atmospheric pressure for 1 h at 800, 900, or 1000 °C. Details of the furnace, including a schematic illustration, were reported in our previous study.¹⁰⁾ Wet-chemical treatments were also performed on the stepped surface obtained after annealing at 1000 °C. The substrate was cut into three pieces along the (100) cleavage plane. The three pieces were treated separately for 1 h with 46–48 wt% HF at room temperature, SPM consisting of 96 wt% H₂SO₄ and 30 wt% H₂O₂ mixed at a volume ratio of 3:1 without external heating, or 25 wt% tetramethylammonium hydroxide (TMAH) at room temperature. The surface morphologies of the as-received, annealed, and chemically treated substrates were examined by tapping-mode atomic force microscopy (AFM; Jupiter XR, Oxford Instruments).

We first investigated the annealing-temperature dependence of the $(\bar{1}12)$ surface morphology. Figure 1 shows the evolution of the surface morphology of the $(\bar{1}12)$ substrate from the as-received state through sequential annealing at progressively increasing temperatures of 800, 900, and 1000 °C for 1 h at each temperature. The as-received substrate exhibited a flat surface without discernible steps or terraces [Fig. 1(a)]. The root-mean-square (RMS) roughness



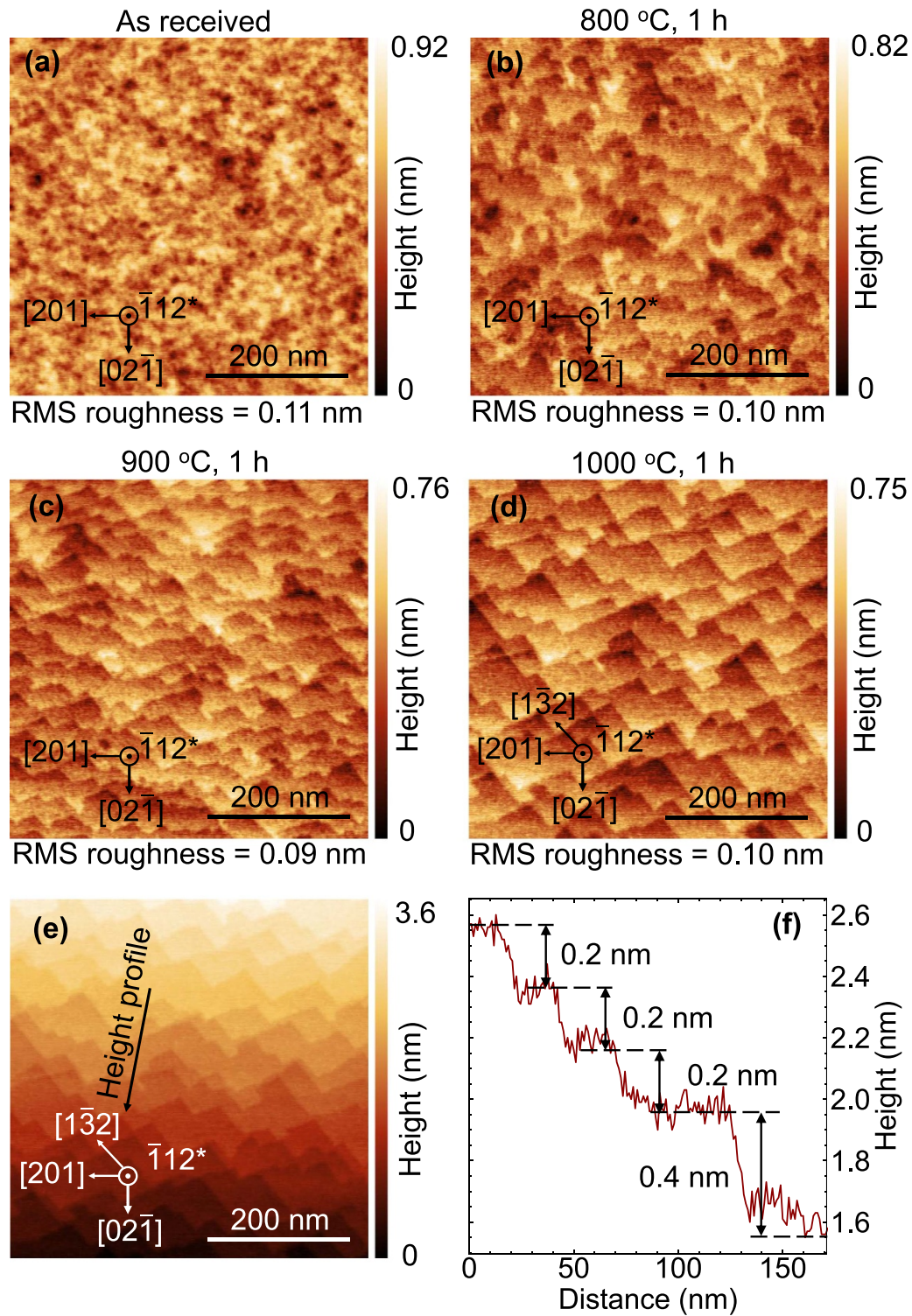


Fig. 1. AFM images of a $(\bar{1}12)$ β - Ga_2O_3 substrate showing the evolution of the surface morphology in (a) the as-received state and after thermal annealing at (b) 800 °C, (c) 900 °C, and (d) 1000 °C. (e) AFM image of the surface in (d) after terrace-by-terrace flattening. The arrow indicates the miscut direction of the substrate surface. (f) Height profile taken along the arrow in (e). Note that hkl^* denotes the direction normal to the (hkl) plane.

was 0.11 nm. It should be noted that this surface morphology was essentially the result of the wet-cleaning process performed after CMP. The initially featureless surface gradually evolved into a distinct stepped morphology as the annealing temperature increased from 800 to 1000 °C. At 800 °C, steps and terraces appeared on the surface; however, the step edges were poorly defined, and numerous pits were present on the terraces [Fig. 1(b)]. These surface irregularities became less pronounced at 900 °C [Fig. 1(c)], and a well-defined

step-and-terrace structure was obtained at 1000 °C [Fig. 1(d)]. The corresponding RMS roughness values were as small as 0.10, 0.09, and 0.10 nm at 800, 900, and 1000 °C, respectively.

The results indicate that the $(\bar{1}12)$ plane of β - Ga_2O_3 can develop an ordered step-and-terrace morphology upon thermal annealing at approximately 1000 °C. As noted above, such a highly ordered arrangement of surface steps formed by thermal annealing has previously been observed only for

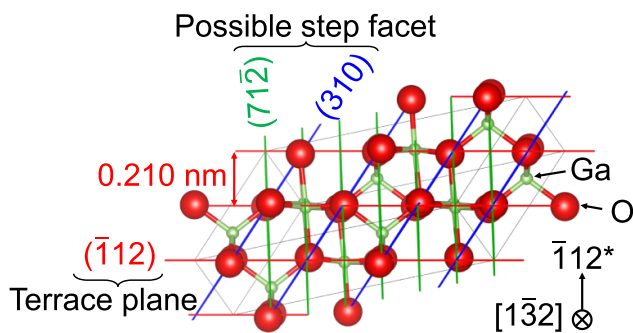


Fig. 2. Crystal structure of β -Ga₂O₃ projected along the $[1\bar{3}2]$ direction.

the (100) plane among the conventionally used substrate orientations. This similarity may arise from the common arrangements of oxygen atoms on the $(\bar{1}12)$ and (100) surfaces.¹³⁾ Notably, homoepitaxial growth by metal-organic chemical vapor deposition (MOCVD) and halide vapor phase epitaxy,^{15–17)} dopant-activation annealing for homoepitaxial layers grown by MOCVD,¹⁸⁾ and post-implantation recovery annealing^{19,20)} are generally performed at temperatures around 1000 °C, suggesting that step-and-terrace formation can be achieved concurrently with these processes.

We then characterized the stepped surface structure using the sample annealed at 1000 °C [Fig. 1(d)]. Most step edges contained straight segments oriented parallel to the $[1\bar{3}2]$ direction. This direction is parallel to the (310) and $(7\bar{1}2)$ planes, along which the oxygen atoms form well-ordered sublattice planes,¹³⁾ as shown in Fig. 2. Therefore, step edges oriented along this crystallographic direction may have a lower density of dangling bonds and, consequently, a lower step energy. Notably, the (310) plane appears as a facet during HCl gas etching of (001)–, (010)–, and $(\bar{1}02)$ -oriented substrates,^{21–23)} which suggests that this plane has a relatively low surface energy.

To measure the step heights, terrace-by-terrace flattening was applied to the topographic data [Fig. 1(e)]. The observed step heights corresponded predominantly to one monolayer, with some steps having a height of two monolayers. From the height profile, the single-monolayer step height was determined to be approximately 0.2 nm [Fig. 1(f)], corresponding to the spacing between adjacent $(\bar{1}12)$ planes ($S = 0.210$ nm) [Fig. 2], calculated using lattice parameters of β -Ga₂O₃.²⁴⁾ In addition, the miscut angle θ was estimated to be $\approx 0.4^\circ$ using the simple trigonometric relation: $\tan \theta = NS/L$, where L is the length of the arrow along the miscut direction ($L = 172$ nm) and N is the number of monolayer steps counted along the arrow ($N = 5$) [Fig. 1(e)]. This miscut angle was not intentionally introduced but originated from the custom substrate fabrication process. A smaller miscut angle would result in wider terraces and might also suppress step bunching.

Finally, we evaluated the effects of wet-chemical treatments using HF, SPM, and TMAH. HF is commonly used to remove colloidal silica originating from polishing slurries and siloxane-based contaminants deposited upon exposure to ambient air.^{25,26)} SPM is widely used for the oxidative removal of organic contaminants, including photoresists and polymer residues, from semiconductor surfaces.²⁷⁾ TMAH is often used to mitigate dry-etch-induced damage

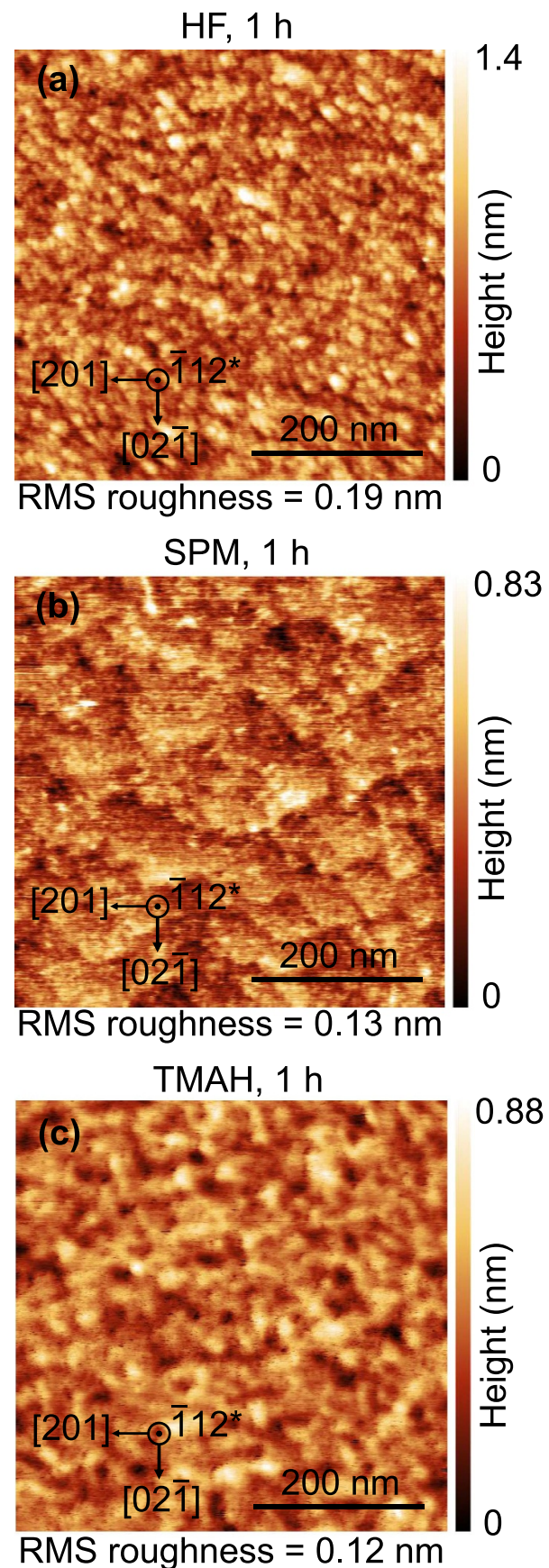


Fig. 3. AFM images of $(\bar{1}12)$ β -Ga₂O₃ substrates after subsequent wet-chemical treatments with (a) HF, (b) SPM, and (c) TMAH.

and reduce surface roughness^{3,28,29)} and is suitable for forming a distinct step-and-terrace structure on (001) and $(\bar{2}01)$ surfaces as described above.^{10,12)} Fig. 3 compares the surface

morphologies after the three wet-chemical treatments. All treatments degraded the thermally formed step-and-terrace morphology, although the degree of degradation depended strongly on the chemical used. After HF and TMAH treatment, the original stepped structure was completely lost, and no distinct terraces remained [Figs. 3(a) and 3(c)]. In contrast, the SPM-treated surface retained a partially preserved stepped morphology, although the step edges became indistinct and pits formed on the terraces [Fig. 3(b)]. The relatively small influence of SPM on the surface can be attributed to its negligible etch rate, consistent with previous wet-etching experiments on (100) substrates.³⁰ These results indicate that thermal annealing is effective for developing an ordered ($\bar{1}12$) surface, whereas wet-chemical treatments examined here disrupted the step-and-terrace morphology. Therefore, wet treatments should be used with caution when the stepped surface needs to be preserved. For example, the treatment time should be kept to the minimum necessary. When organic contaminants need to be removed, ozone treatment may be considered as an alternative.

In conclusion, we demonstrated the formation of a well-defined step-and-terrace surface morphology on a CMP-processed ($\bar{1}12$) β -Ga₂O₃ substrate via thermal annealing at approximately 1000 °C. The observed step height was in good agreement with the spacing between adjacent ($\bar{1}12$) planes (0.210 nm). We also confirmed that commonly used wet-chemical treatments using HF, SPM, and TMAH did not preserve the stepped surface morphology. These findings provide useful guidance for pre- and post-treatment processes of ($\bar{1}12$) β -Ga₂O₃, an orientation that has only recently begun to attract research interest.

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- 1) K. Sasaki, *Appl. Phys. Express* **17**, 090101 (2024).
- 2) K. Goto, H. Murakami, A. Kuramata, S. Yamakoshi, M. Higashiwaki, and Y. Kumagai, *Appl. Phys. Lett.* **120**, 102102 (2022).
- 3) T. Oshima, *AIP Adv.* **16**, 025145 (2026).
- 4) T. Liu, Z. Li, J. Zhao, X. Fei, J. Feng, Y. Zuo, M. Hua, Y. Guo, S. Liu, and Z. Zhang, *Acta Mater.* **300**, 121484 (2025).
- 5) M. Lee, T. Chou, S. B. Anooz, Z. Galazka, A. Popp, and R. L. Peterson, *ACS Nano* **16**, 11988 (2022).
- 6) P. Mazzolini, P. Vogt, R. Schewski, C. Wouters, M. Albrecht, and O. Bierwagen, *APL Mater.* **7**, 022511 (2019).
- 7) C.-H. Lin, K. Ema, S. Masuya, Q. T. Thieu, R. Sakaguchi, K. Sasaki, and A. Kuramata, *Jpn. J. Appl. Phys.* **62**, SF1005 (2023).
- 8) T. Oshima and Y. Oshima, *Jpn. J. Appl. Phys.* **65**, 138001 (2026).
- 9) S. Ohira, N. Arai, T. Oshima, and S. Fujita, *Appl. Surf. Sci.* **254**, 7838 (2008).
- 10) T. Oshima, *Jpn. J. Appl. Phys.* **64**, 088001 (2025).
- 11) A. Okada, M. Nakatani, L. Chen, R. A. Ferreyra, and K. Kadono, *Appl. Surf. Sci.* **574**, 151651 (2022).
- 12) T. Oshima, *Sci. Technol. Adv. Mater.* **27**, 2666988 (2026).
- 13) T. Oshima, *Jpn. J. Appl. Phys.* **65**, 038003 (2026).
- 14) T. Oshima and Y. Oshima, *Sci. Technol. Adv. Mater.* **27**, 2680969 (2026).
- 15) J. Yoshinaga, H. Tozato, T. Okuyama, S. Sasaki, G. Piao, K. Ikenaga, K. Goto, Y. Ban, and Y. Kumagai, *Appl. Phys. Express* **16**, 095504 (2023).
- 16) H. Murakami et al., *Appl. Phys. Express* **8**, 015503 (2015).
- 17) Y. Oshima and T. Oshima, *Sci. Technol. Adv. Mater.* **26**, 2585551 (2025).
- 18) J. Yoshinaga et al., *Appl. Phys. Express* **18**, 055503 (2025).
- 19) K. Sasaki, M. Higashiwaki, A. Kuramata, T. Masui, and S. Yamakoshi, *Appl. Phys. Express* **6**, 086502 (2013).
- 20) M. H. Wong, C.-H. Lin, A. Kuramata, S. Yamakoshi, H. Murakami, Y. Kumagai, and M. Higashiwaki, *Appl. Phys. Lett.* **113**, 102103 (2018).
- 21) T. Oshima and Y. Oshima, *Appl. Phys. Lett.* **122**, 162102 (2023).
- 22) T. Oshima and Y. Oshima, *Appl. Phys. Express* **16**, 066501 (2023).
- 23) T. Oshima and Y. Oshima, *Appl. Phys. Lett.* **124**, 042110 (2024).
- 24) J. Åhman, G. Svensson, and J. Albertsson, *Acta Crystallogr. C* **52**, 1336 (1996).
- 25) C. Huang, W. Mu, H. Zhou, Y. Zhu, X. Xu, Z. Jia, L. Zheng, and X. Tao, *RSC Adv.* **8**, 6544 (2018).
- 26) J. P. McCandless, C. A. Gorsak, V. Protasenko, D. G. Schlom, M. O. Thompson, H. G. Xing, D. Jena, and H. P. Nair, *Appl. Phys. Lett.* **124**, 111601 (2024).
- 27) W. Kern, *J. Electrochem. Soc.* **137**, 1887 (1990).
- 28) F. Zhang, X. F. Zheng, Y. H. Li, Z. J. Yuan, S. Z. Yue, X. C. Wang, Y. L. He, X. L. Lu, X. H. Ma, and Y. Hao, *Appl. Surf. Sci.* **684**, 161569 (2025).
- 29) A. R. Gutierrez et al., *J. Vac. Sci. Technol. A* **43**, 033210 (2025).
- 30) S. Ohira and N. Arai, *Phys. Status Solidi C* **5**, 3116 (2008).