

Current Status of Bulk β -Ga₂O₃ and β -(Al_xGa_{1-x})₂O₃ Crystal Growth

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Keywords: β -Ga₂O₃, β -(Al_xGa_{1-x})₂O₃, bulk crystals, doping, electrical properties

Abstract

β -Ga₂O₃ and its solid solutions β -(Al_xGa_{1-x})₂O₃ are considered as potential candidate materials for next generation power devices due to their ultra-wide bandgaps (4.85–5.6 eV) and large breakdown electric field (≥ 8 MVcm⁻¹). Wafers prepared from bulk crystals constitute a foundation in a device fabrication chain. The present report briefly discusses challenges in growing bulk crystals by different methods, crystal structural quality, wafers, and electrical properties.

INTRODUCTION

β -Ga₂O₃ as an n-type oxide semiconductor was already known since the 60s of the 20th century, but its re-discovery and scientific study started roughly two decades ago, with rapid technological development after reporting a first β -Ga₂O₃-based transistor in 2012 [1]. Great physical properties, including an ultra-wide bandgap of 4.85 eV, large breakdown electric field of 8 MVcm⁻¹, wide doping range with free electrons between $\sim 10^{15}$ - 10^{20} cm⁻³, and good electron mobility approaching 200 cm²V⁻¹s⁻¹, made β -Ga₂O₃ a really attractive material for power devices aiming for switching high voltages (kV range). Indeed, intensive material investigation (theoretical and experimental), film growth by different epitaxial methods, and fabrication of power devices in various configurations [2, 3] demanded the development of bulk β -Ga₂O₃ crystals for substrates. Among a diversity of crystal growth methods from the melt utilized for β -Ga₂O₃, those enabling at least 2 inch diameter wafers include the Edge-Defined Film-Fed Growth (EFG) [4], Czochralski (CZ) [5], Vertical Bridgman (VB) [6], direct solidification (DS) or “casting” [7], and possible Cold Crucible (CC) [8]. As a result, 4-inch diameter β -Ga₂O₃ wafers became commercially available [9], while 6-inch diameter wafers have been demonstrated [3]. To enlarge the bandgap, Ga₂O₃-Al₂O₃ solid solution system has recently been investigated, also bulk β -(Al_xGa_{1-x})₂O₃ crystals with [Al] \leq 40 mol.% being the physical limit for the crystals, have been grown [10, 11].

THERMODYNAMICS

Ga₂O₃ is thermally unstable at high temperatures and has high melting point ($\sim 1800^\circ\text{C}$) leading to a thermal decomposition independent on the crystal growth method from the melt. To get a liquid phase, some oxygen partial pressure, $p(\text{O}_2) \geq 10^{-5}$ bar, is required. During crystal growth,

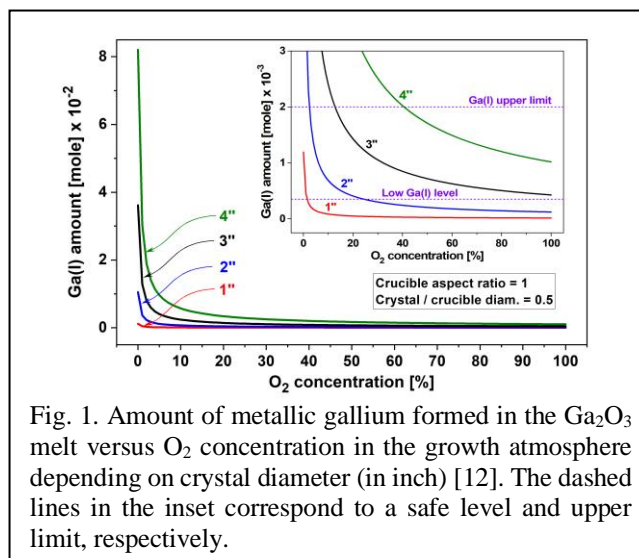


Fig. 1. Amount of metallic gallium formed in the Ga₂O₃ melt versus O₂ concentration in the growth atmosphere depending on crystal diameter (in inch) [12]. The dashed lines in the inset correspond to a safe level and upper limit, respectively.

$p(\text{O}_2) \approx 10^{-2}$ bar is sufficient to bring partial pressures of the most volatile species of O₂ and Ga₂O down to a level to enable obtaining a single crystal. Surely, the higher $p(\text{O}_2)$ the better crystal structural quality can be achieved. Although the volatile species can be easily handled by even small O₂ concentration in a growth atmosphere (0.5 vol.%), another issue becomes more critical when scaling-up the crystal size. This is the formation of elemental Ga that partly remains in the melt in the liquid phase leading to damage of a noble metal crucible (Ir and Pt) by forming eutectic, and prevents a single crystal from being obtained [12]. As shown in Fig. 1, the Ga amount strongly depends on the melt volume, from which a crystal of a pre-defined diameter can be grown, and $p(\text{O}_2)$. At small crystal diameter ≤ 1 inch, the Ga amount is negligibly small, but it significantly increases with crystal diameter (melt volume). To keep a small and safe Ga amount in the melt as in the case of 1 inch crystal diameter, 2 and 3 inch diameter crystals would require 25 and 100% O₂, respectively, while in the case of 4 inch diameter crystal 100% O₂ under overpressure would be needed. Large crystal diameter still can be grown at lower O₂ concentrations, at min. 5, 12, and 40 % for 2, 3, and 4 inch diameter crystals, respectively, but the crystal quality and noble metal crucibles may deteriorate.

To avoid the formation of solid IrO₂ at elevated temperatures when using Ir or Ir-alloyed crucibles, a specific scheme of oxygen supply to the growth atmosphere has been

developed and applied [13]. To decrease the melt volume, a crucible with the height smaller than the diameter is preferred [5]. In the case of Pt-alloyed crucibles, a constant oxygen concentration can be used.

BULK CRYSTAL GROWTH

Among different growth methods, such as from the gas phase, solutions, and the melt, only the latter enable large (≥ 2 inch diameter) β -Ga₂O₃ single crystals to be obtained. Large crystal / wafer diameter was achieved by the EFG, CZ, VB, and DS methods.

The **EFG** method has been developed in Japan (Novel Crystal Technology, NCT) and produces crystal slabs grown along the [010] direction with (-201) and (001)-oriented principal planes for large wafer diameter up to 6 inch [4, 9]. Both the crucibles and dies (shapers) are made of Ir. The EFG is also utilized in the US (Luxium Solutions) with the (100) principal planes, and in China (Shandong University) with the (010) principal planes by using the [001] growth directions [14]. Typically, the EFG method requires smaller melt volumes as compared to cylindrical crystals (CZ, VB) even to grow large slabs, which can be thin (ca. 1 cm for 4 inch wafers). Nonetheless, the melt is transported to the die surface via capillary forces from lower part of the crucible, which is less saturated with O₂ than the growth atmosphere. Additionally, the crystal entirely covers the surface area of the die, so O₂ access to the growth interface from the growth atmosphere is also limited (through the melt meniscus only). As a result, there is a decomposition of Ga₂O₃ at the growth interface and the gaseous species are entrapped leading to void-type defects (nanopipes or line-shaped defects), which gains current leakage in electronic devices (depending on orientation and device architecture), and also affects the growth of thick films. The dislocation density is at a level of 10³-10⁵ cm⁻², while the void-type defects are on the order of 10² cm⁻². Typically, Sn or Si are used as shallow donors, while Fe is a compensating acceptor. Differently oriented wafers from NCT are widely used for homoepitaxy.

The **CZ** method for β -Ga₂O₃ has been developed at Leibniz-Institut für Kristallzüchtung (IKZ) in Germany using Ir crucibles and above-mentioned specific scheme of O₂ supply to the growth atmosphere [5, 12, 13]. The CZ method is also utilized in the US (Northrop-Grumman Synoptics) [15]. Because the crystals are grown from the exposed melt surface being in direct contact with O₂ from the growth atmosphere, and due to buoyancy-driven melt flow along the growth interface, the interface is saturated with O₂ and no void-type defects are formed (IKZ crystals) [5]. With large crystal diameter it is very important to use low temperature gradients, as high gradients enhance thermal decomposition. The crystals are grown along the [010] direction enabling 2 inch diameter (010) oriented wafers. Also, other wafer orientations (100) and (001) have been prepared. In particular, IKZ has developed the homoepitaxial growth of β -Ga₂O₃ layers by MOVPE on off-oriented (100) wafers providing the smoothest surface compared to other orientations, and

enabling high quality thick films (4-6 μ m) while maintaining great electrical properties. The RMS roughness of epi-ready wafers is typically about 200 pm, FWHM of the rocking curves of 20-30 arcsec, and the dislocation density below 10³ cm⁻². To get semiconducting or degenerate semiconducting state β -Ga₂O₃ is typically doped with Si or Sn, while for semi-insulating state with Mg, Co, or Ni [16] (IKZ) or with Fe (Synoptics) [15]. Examples of CZ-grown crystals at IKZ are shown in Fig. 2. The challenge for the Czochralski method is to grow long and highly conducting crystals due to free carriers absorbing infrared radiation (heat) being transported through a growing crystal [5]. By applying at IKZ several measures, such as dynamic growth rate, insulation limiting radiation being reflected back to the growing crystal, and/or gas of high conductivity (He) in the growth atmosphere, IKZ has been able to obtain 2 inch diameter crystals with a length of 30 mm [17, 18] (Fig. 2).

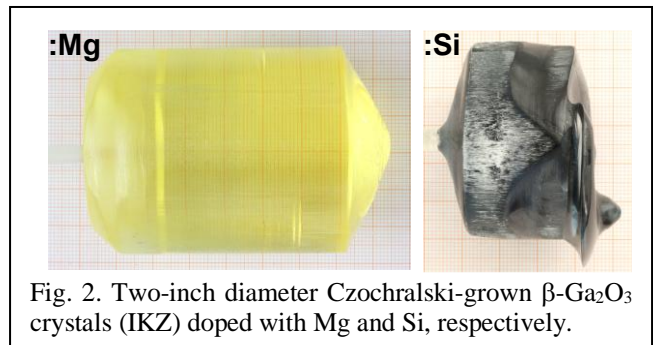


Fig. 2. Two-inch diameter Czochralski-grown β -Ga₂O₃ crystals (IKZ) doped with Mg and Si, respectively.

The **VB** method has been developed in Japan (Shinshu University and NCT) using Pt-Rh alloyed crucibles which are of single use [6]. The crystals are of 2 inch diameter with 6 inch demonstrated (short, 1 cm only) [19]. As the crystallization takes place on the top of the seed (in contrast to EFG and CZ), any seed orientation can be used, such as [100], [010], and [001] [20], enabling large, differently oriented wafers. Here, the growth interface is distant from the growth atmosphere (separated by the melt bulk), therefore, the access of O₂ to the growth interface is always limited, even if high O₂ concentration in the growth atmosphere is used. As a result, the obtained crystals contain void-type defects, similar to those grown by the EFG. The reported structural quality of the wafers is similar to those from EFG crystals in terms of dislocation density and void-type defect density. Additionally, the crystals are contaminated with Rh from the crucibles. Sn is used for n-type doping, and Fe for semi-insulating state. There are no published reports on homoepitaxy using VB wafers so far.

The **DS** method, called "casting", has been recently developed in China (Zhejiang University/ZJU-Hangzhou Global Scientific and Technological Innovation Center, and Garen Semiconductor) [21]. This is just melting and solidification within an Ir crucible without using any seed. As this is a spontaneous crystallization, a high density of structural defects is expected, also the yield may not be high due to easy formation of twins, low-angle grain boundaries,

and polycrystalline regions, especially contacting the crucible wall. To minimize melt volume and segregation of residual impurities only small amount of the starting material was used, yielding crystal discs with a thickness ≤ 20 mm and diameter 4 and 6 inch [22]. 8 inch diameter crystal discs were also demonstrated [23]. The crystals are either semi-insulating or semiconducting. The dislocation density is at the level of $5 \times 10^4 \text{ cm}^{-2}$. The crystals contain void-type defects as in the case of EFG and VB, as well as Ir particles. Also, in this case there no reports on homoepitaxial growth of layers.

The **CC** method is being developed in Japan (Tohoku University and C&A Corporation) [8]. The melting of the starting material proceeds through a direct coupling of the electromagnetic field with the starting material loaded into a water-cooled crucible consisting of separate fingers (typically made of copper). The starting material adjacent to the cold crucible does not melt and forms a shell containing the melt. No noble metal crucible is used. This variant of the CC method involves pulling a crystal from the melt surface. The same temperature at the growth interface and at the shell formed by the starting material, as well as changes of the melt resistivity driven by changes of the melt temperature and thickness of the shell containing the melt, make the melt flow and crystal diameter control really challenging. Although it is possible to grow cylindrical crystals of about 1 inch diameter, trials in growing 2 inch diameter crystals resulted in thin disks only, limiting preparation of large wafers, however, there is still room for the development. So far, there are no reports on homoepitaxial growth using wafers prepared from CC-grown crystals.

BULK $\beta\text{-(Al}_x\text{Ga}_{1-x})_2\text{O}_3$ SOLID-SOLUTION CRYSTALS

Undoped, Si-doped, and Mg-doped $\beta\text{-(Al}_x\text{Ga}_{1-x})_2\text{O}_3$ single crystals with $x=0.05\text{-}0.35$ have been grown at IKZ [10, 11], including 2 inch diameter crystals with $x \leq 0.25$ (Fig. 3). It has been found that the limit for Al incorporation while keeping a crystalline phase is 40 mol.%. Main physical properties (structural, optical, electrical, and thermal) have been measured as a function of Al content with the steps of $\Delta[\text{Al}]=5$ mol.%. The bandgap reaches a value of 5.6 eV at $[\text{Al}]=30$ mol.%, however, the electrical properties, thermal conductivity, and the dielectric constant deteriorate with $[\text{Al}]$. The surface smoothness and structural quality of the $\beta\text{-}$

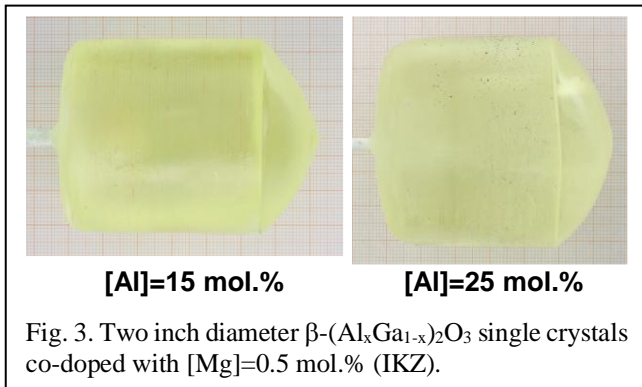


Fig. 3. Two inch diameter $\beta\text{-(Al}_x\text{Ga}_{1-x})_2\text{O}_3$ single crystals co-doped with $[\text{Mg}]=0.5$ mol.% (IKZ).

$(\text{Al}_x\text{Ga}_{1-x})_2\text{O}_3$ wafers are comparable with those of $\beta\text{-Ga}_2\text{O}_3$ for $x \leq 0.2$. MOVPE homoepitaxy on $\beta\text{-(Al}_x\text{Ga}_{1-x})_2\text{O}_3$ wafers is ongoing at IKZ.

ELECTRICAL PROPERTIES

Typically, undoped $\beta\text{-Ga}_2\text{O}_3$ crystals grown from the melt are semiconducting due to residual impurities (mainly Si) and hydrogen, if compensation is not high enough (i.e. O_2 partial pressure and compensating impurities). To get higher conductivity, the crystals are intentionally doped mainly with Si or Sn (also with Hf, Zr, and Nb). Fig. 4 shows a relation between electron mobility (μ_e) and free electron concentration (n_e) for the CZ, VB, and EFG methods. In the case of $\beta\text{-Ga}_2\text{O}_3$, μ_e gradually decreases with n_e due to dominating scattering at phonons (low n_e) and impurities (high n_e). EFG μ_e data are close to those of CZ data, except low n_e values due to extra scattering. VB μ_e data are on average lower than those of CZ and EFG. As the crystals are used for electronic devices either as semi-insulating (lateral configuration) or highly conducting (vertical architecture) state, crystals grown by all discussed methods might be suitable for devices from electrical properties point of view. However, structural defects may play a crucial role.

In the case of $\beta\text{-(Al}_x\text{Ga}_{1-x})_2\text{O}_3$ crystals, both μ_e and n_e decrease with $[\text{Al}]$, and vanish at $[\text{Al}]=25$ mol.% independent on the Si doping level. This is due to extra scattering at phonons caused by Al incorporation into the crystal lattice [10], and by strong compensation by cation vacancies, the density of which increases with $[\text{Al}]$ [24].

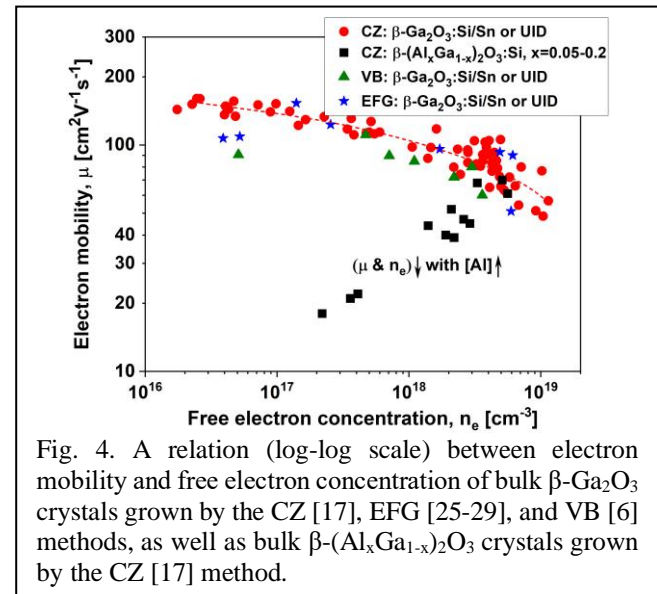


Fig. 4. A relation (log-log scale) between electron mobility and free electron concentration of bulk $\beta\text{-Ga}_2\text{O}_3$ crystals grown by the CZ [17], EFG [25-29], and VB [6] methods, as well as bulk $\beta\text{-(Al}_x\text{Ga}_{1-x})_2\text{O}_3$ crystals grown by the CZ [17] method.

WAFER COSTS

Wafer costs includes costs of growing bulk crystals and wafer fabrication, the latter should be similar for all growth methods, as it is a separate process. Crystal growth costs include: (i) starting material; (ii) re-use of the starting material; (iii) thermal insulation and its re-use; (iv) noble metal crucible and other parts; (v) re-use of the crucible; (vi)

crucible fabrication; (vii) crucible material losses; (viii) gasses; (ix) energy consumption; (x) labor; and (xi) yield. The costs of (i), (iii), (vi), (viii), and (x) are likely similar across the growth methods. In VB and DS whole starting material (ii) is being crystallized in one growth run, while in EFG and CZ the melt is re-used multiple times. Noble metal crucibles and other parts (iv) are made of Ir in the case of EFG, CZ, and DS, and of Pt-Rh alloy in the case of VB. Currently, Pt is cheaper than Ir, while the price of Rh is similar to Ir. Crucibles (v) used for EFG, CZ, and DS are used multiple times, while for VB – only once, generating crucible fabrication costs for each growth run. Due to necessity of using oxidizing atmosphere, there are losses of Ir (EFG, CZ, DS), but not Pt-Rh alloy (VB). The unknown is the yield of crystal growth for different methods, as this has never been reported, but may significantly contribute to overall costs. Due to monoclinic system and 2 easy cleavage planes, structural defects are easily formed and lower the yield in both the crystal growth and wafer fabrication. In the end, the final wafer price depends on the crystal volume and its structural quality. Cylindrical crystals grown by the CZ and VB offer a large number of wafers from one crystal, while crystals slabs / discs grown by the EFG and DS yield small number of wafers from one crystal, when of large diameter. For instance, from CZ-grown crystals in Fig. 2 (IKZ), about 50 and 25 two-inch diameter (010)-oriented wafers can be fabricated from β -Ga₂O₃ crystal doped with Mg and Si, respectively. EFG-grown crystal slabs with 2 and 4 inch principal planes allow about 18 or 10 wafers to be obtained from 2 or 4 inch diameter slabs, respectively. The DS-grown crystals enable 10–20 wafers to be obtained from one crystal.

CONCLUSIONS

Different methods have been applied for growing large bulk β -Ga₂O₃ crystals from the melt. In terms of wafer size (4, 6, and 8 inch), the best suited methods are DS and EFG, with potentials for VB (6 inch) and CZ (4 inch). It seems the CZ method provides the best structural quality (when grown at low temperature gradients), followed by the EFG and VB methods. DS-grown crystals are rather of lower structural quality. From the point of view of electronic devices, electrical properties of crystals from different methods seem to be acceptable, although void-type defects in EFG, VB, and DS may impact the device operation at certain wafer orientations. The largest β -(Al_xGa_{1-x})₂O₃ crystals (2 inch diameter) were grown by the CZ method, with the Al limit of 40 mol.%, but the electrical properties deteriorate with [Al]. Currently, it is difficult to compare crystal growth costs across different methods, as the crystal growth yield remains unknown (not reported).

ACKNOWLEDGEMENTS

The author would like to thank Klaus Imscher, Andreas Fiedler, and Mike Pietsch from IKZ for electrical characterization, as well as Isabelle M. Hanke and Manuela Sündermann from IKZ for supporting crystal growth. The work at IKZ has been financially supported by the German Research Foundation (DFG) project nos. FO 558/3-1, GA 2057/2-1, and GA 2057/5-1; Bundesministerium für Bildung und Forschung (BMBF) projects nos. 03VP03712 and 16ES1084K; and by the Leibniz Association – Leibniz Science Campus GraFOx.

REFERENCES

- [1] M. Higashiwaki, et al.; *Appl. Phys. Lett.* 100 (2012) 013504.
- [2] Eds. S. Pearton, F. Ren, and M. Mastro; “Gallium Oxide: Technology, Devices and Applications”, Elsevier (2018).
- [3] Eds. M. Higashiwaki and S. Fujita; “Gallium Oxide: Crystal Growth, Materials Properties, and Devices”; Springer Nature Switzerland AG (2020).
- [4] A. Kuramata, et al.; *Jpn. J. Appl. Phys.* 55 (2016) 1202A2.
- [5] Z. Galazka; *J. Appl. Phys.* 131 (2022) 031103.
- [6] K. Hoshikawa, et al.; *J. Cryst. Growth* 546 (2020) 125778.
- [7] X. Gao, et al.; *J. Alloys Compd.* 987 (2024) 174162.
- [8] A. Yoshikawa et al.; *Sci. Rep.* 14 (2024) 14881.
- [9] <https://www.novelcrystal.co.jp/eng/>
- [10] Z. Galazka, et al.; *J. Appl. Phys.* 133 (2023) 035702.
- [11] Z. Galazka, et al.; *Adv. Mater. Interfaces* (2024) 2400122.
- [12] Z. Galazka, et al.; *ECS J. Solid State Sci. Technol.* 6 (2017) Q3007-Q3011.
- [13] Z. Galazka, et al.; EP 3242965 B1, US 11028501 B2, KR 101979130 B1.
- [14] X. Dong, et al.; *J. Semicond.* 46 (2025) 062501.
- [15] J. D. Blevins, et al.; *IEEE Trans. Semicond. Manuf.* 32 (2019) 466-472.
- [16] Z. Galazka, et al.; *J. Alloy. Compd.* 818 (2020) 152842.
- [17] Z. Galazka, et al.; PCT/EP2023/051212 (2023).
- [18] Z. Galazka; “Growth of bulk β -Ga₂O₃ single crystals” in “Comprehensive Semiconductor Science and Technology 2nd Ed.”, Ed. R. Fornari, Elsevier (2025).
- [19] T. Igarashi, et al.; *Phys. Status Solidi B* (2025) 2400444.
- [20] E. Ohba, et al.; *J. Cryst. Growth* 556 (2021) 125990.
- [21] N. Xia, et al.; *J. Alloys Compd.* 935 (2023) 168036.
- [22] L. Huang et al.; *Eur. Phys. J. Spec. Top.* (2025) <https://doi.org/10.1140/epjs/s11734-025-01486-2>
- [23] <http://www.garen.cc/newsx.php?lm=14&id=72>
- [24] H. Peelaers; IWSO-11, 2-6 March 202, Tempe, USA.
- [25] T. Onuma, et al.; *Appl. Phys. Lett.* 103 (2013) 041910.
- [26] T. Oishi, et al.; *Appl. Phys. Express* 8 (2015) 031101.
- [27] B. Fu, et al.; *J. Alloy. Compd.* 896 (2022) 162830.
- [28] W. Mu, et al.; *J. Alloy. Compd.* 714 (2017) 453-458.
- [29] S. Zhang, et al.; *J. Semicond.* 39 (2018) 083003.