

Effects of High-Temperature Annealing on the Microstructure and Mechanical Properties of β -Ga₂O₃:Fe Crystals

D.A. Kalganov , D.I. Panov , V.A. Spiridonov , A.Yu. Ivanov ,
I.G. Smirnova , D.A. Bauman 

Institute of Advanced Data Transfer Systems, ITMO University, Kronverkskiy pr., 49, lit. A, St. Petersburg, 197101, Russia

Received: March 06, 2026

Corresponding author: [D.A. Kalganov](mailto:D.A.Kalganov@itmo.ru)

Abstract. In this work, the microstructure and mechanical properties of bulk β -Ga₂O₃:Fe (0.01 mol.%) crystals were investigated using X-ray diffraction, optical transmission spectroscopy, and the ultrasonic composite oscillator technique at successive stages of two-step annealing at 1400 °C in air. Rocking curves revealed a substantial microstructural evolution, with a more complex profile envelope after 6 hours of annealing and a significant narrowing accompanied by increased reflection intensity after 12 hours. Optical transmission spectra showed an increase in transmittance below 650 nm and a decrease in the long-wavelength region. The amplitude dependences of internal friction and the effective elastic modulus were obtained and characterized. Changes in the microplastic yield point under oscillatory deformation and in the background values of these quantities were observed, indicating a reduction in the concentration of mobile defects and microstructural ordering. The effective elastic modulus measured in β -Ga₂O₃:Fe (0.01 mol.%) crystal after annealing along the [100] direction was found to be 339.8 GPa.

Acknowledgements. The authors are grateful to Professor Alexey Romanov for insightful discussion and valuable suggestions.

Funding. This work was supported by the Ministry of Science and Higher Education of the Russian Federation, project no. FSER-2025-0005.

Citation: Rev. Adv. Mater. Technol., 2026, vol. 8, no. 2, pp. 73–80

View online: <https://doi.org/10.17586/2687-0568-2026-8-2-73-80>

REFERENCES

1. Y. Du, C. Zhang, S. Cui, G. Li, N. Tang, B. Shen, J. Xu, H. Tang, L. Zhao. Iron-doped β - Ga_2O_3 single crystal: the iron occupying site and optical properties. *CrystEngComm*, 2025, vol. 27, no. 15, pp. 2392–2400.
2. K.M. Dowling, B. Chatterjee, S. Ghandiparsi, Q. Shao, J. Varley, J.D. Schneider, C. Chapin, M.S. Gottlieb, L. Leos, M. Sword, S. Harrison, L. Voss. Evaluation of Fe- $\beta\text{Ga}_2\text{O}_3$ for photoconductive semiconductor switching. *IEEE Trans. Electron Devices*, 2024, vol. 71, no. 3, pp. 1535–1540.
3. R.A. Babunts, A.S. Gurin, E.V. Edinach, Yu.A. Uspenskaya, P.G. Baranov. Features of the iron charge states in semi-insulating $\beta\text{-Ga}_2\text{O}_3\text{:Fe}$ identified by high-frequency electron paramagnetic resonance. *Tech. Phys. Lett.*, 2023, vol. 49, no. 11, pp. 12–14.
4. M. Higashiwaki, K. Sasaki, H. Murakami, Y. Kumagai, A. Koukitu, A. Kuramata, T. Masui, S. Yamakoshi. Recent progress in Ga_2O_3 power devices. *Semicond. Sci. Technol.*, 2016, vol. 31, no. 3, art. no. 034001.
5. R.A. Babunts, A.S. Gurin, E.V. Edinach, H.-J. Drouhin, V.I. Safarov, P.G. Baranov. Non-Kramers iron $S=2$ ions in $\beta\text{-Ga}_2\text{O}_3$ crystals: High-frequency low-temperature EPR study. *J. Appl. Phys.*, 2022, vol. 132, no. 15, art. no. 155703.
6. A. Kuramata, K. Koshi, S. Watanabe, Y. Yamaoka, T. Masui, S. Yamakoshi. Bulk crystal growth of Ga_2O_3 . *Proc. SPIE 10533, Oxide-based Materials and Devices IX*, 2018, art. no. 105330E.
7. J.P. Remeika. GaFeO_3 : a ferromagnetic-piezoelectric compound. *J. Appl. Phys.*, 1960, vol. 31, no. 5, pp. S263–S264.
8. N. Zhang, H. Liu, Q. Sai, C. Shao, C. Xia, L. Wan, Z.C. Feng, H.F. Mohamed. Structural and electronic characteristics of Fe-doped $\beta\text{-Ga}_2\text{O}_3$ single crystals and the annealing effects. *J. Mater. Sci.*, 2021, vol. 56, no. 23, pp. 13178–13189.
9. Y. Wang, J. Su, Z. Lin, J. Zhang, J. Chang, Y. Hao. Recent progress on the effects of impurities and defects on the properties of Ga_2O_3 . *J. Mater. Chem. C*, 2022, vol. 10, no. 37, pp. 13395–13436.
10. M. Higashiwaki, A. Kuramata, H. Murakami, Y. Kumagai. State-of-the-art technologies of gallium oxide power devices. *J. Phys. D: Appl. Phys.*, 2017, vol. 50, no. 33, art. no. 333002.
11. N.A. Mahadik, M.J. Tadjer, P.L. Bonanno, K.D. Hobart, R.E. Stahlbush, T.J. Anderson, A. Kuramata. High-resolution dislocation imaging and micro-structural analysis of HVPE- $\beta\text{-Ga}_2\text{O}_3$ films using monochromatic synchrotron topography. *APL Mater.*, 2019, vol. 7, no. 2, art. no. 022513.
12. K. Adachi, H. Ogi, N. Takeuchi, N. Nakamura, H. Watanabe, T. Ito, Y. Ozaki. Unusual elasticity of monoclinic $\beta\text{-Ga}_2\text{O}_3$. *J. Appl. Phys.*, 2018, vol. 124, no. 8, art. no. 085102.
13. X.Q. Zheng, H. Zhao, Z. Jia, X. Tao, P.X. Feng. Young's modulus and corresponding orientation in $\beta\text{-Ga}_2\text{O}_3$ thin films resolved by nanomechanical resonators. *Appl. Phys. Lett.*, 2021, vol. 119, no. 1, art. no. 013505.
14. J.Q. Zhang, H. Zhou, W. Jiang, S.X. Hu, X.P. Ren, C.W. Zhang. Investigation of dynamic mechanical properties and constitutive model of (010) plane of gallium oxide crystals under shock loading. *Ceram. Int.*, 2022, vol. 48, no. 19, pp. 27823–27835.
15. Y. Yao, Y. Sugawara, K. Sasaki, A. Kuramata, Y. Ishikawa. Anisotropic mechanical properties of $\beta\text{-Ga}_2\text{O}_3$ single-crystal measured via angle-dependent nanoindentation using a Berkovich indenter. *J. Appl. Phys.*, 2023, vol. 134, no. 21, art. no. 215106.
16. V.V. Kaminskii, D.I. Panov, V.A. Spiridonov, D.A. Bauman, D.A. Kalganov, M.P. Scheglov, A.E. Romanov. Effect of high-temperature annealing on the internal friction and optical transmittance of single crystal gallium oxide. *Mater. Phys. Mech.*, 2024, vol. 52, no. 5, pp. 48–54.
17. S. Kustov, S. Golyandin, A. Ichino, G. Gremaud. A new design of automated piezoelectric composite oscillator technique. *Mater. Sci. Eng. A*, 2006, vol. 442, no. 1–2, pp. 532–537.
18. G. Gremaud, S. Kustov. Theory of dislocation-solute atom interactions in solid solutions and related nonlinear anelasticity. *Phys. Rev. B*, 1999, vol. 60, no. 13, pp. 9353–9366.
19. A. Wolfenden, M.R. Harmouche. Elastic constants of silver as a function of temperature. *J. Mater. Sci.*, 1993, vol. 28, no. 4, pp. 1015–1018.

20. S. Kustov, J. Miguez Obrero, X. Wang, D. Damjanovic, E.K. Salje. Phase transitions in the ferroelectric relaxor $(1-x)\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_{3-x}\text{PbTiO}_3$ close to the morphotropic phase boundary. *Phys. Rev. Mater.*, 2022, vol. 6, no. 12, art. no. 124414.
21. M. Xie, Q. Huan, F. Li. Quick and repeatable shear modulus measurement based on torsional resonance using a piezoelectric torsional transducer. *Ultrasonics*, 2020, vol. 103, art. no. 106101.
22. Z.H. Wu, X.P. He, H. Zheng, X.M. Zhu. An analysis of high-frequency dynamic mechanical parameters based on the design of transducer. *J. Yunnan Univ. Nat. Sci. Ed.*, 2022, vol. 44, no. 3, pp. 569–575 [in Chinese].
23. D.A. Kalganov, D.A. Bauman, D.Yu. Panov, V.A. Spiridonov, A.Yu. Ivanov, A.E. Romanov. Amplitude-dependent internal friction and modulus of elasticity in single crystal of $\text{Ga}_2\text{O}_3\text{-Al}_2\text{O}_3$ solid solution. *Phys. Solid State*, 2025, vol. 67, no. 10, pp. 1930–1935.
24. H. Nishinaka, Y. Kajita, S. Hosaka, H. Miyake. Composition analysis of $\beta\text{-(In}_x\text{Ga}_{1-x})_2\text{O}_3$ thin films coherently grown on (010) $\beta\text{-Ga}_2\text{O}_3$ via mist CVD. *Sci. Technol. Adv. Mater.*, 2024, vol. 25, no. 1, art. no. 2414733.
25. J. Furthmüller, F. Bechstedt. Quasiparticle bands and spectra of Ga_2O_3 polymorphs. *Phys. Rev. B*, 2016, vol. 93, no. 11, art. no. 115204.
26. S. Poncé, F. Giustino. Structural, electronic, elastic, power, and transport properties of $\beta\text{-Ga}_2\text{O}_3$ from first principles. *Phys. Rev. Research*, 2020, vol. 2, no. 3, art. no. 033102.
27. E. Welch, P. Borges, L. Scolfaro. Hybrid density functional theory study of substitutional Gd in $\beta\text{-Ga}_2\text{O}_3$. *Physica B*, 2023, vol. 651, art. no. 414558.
28. M.D. Ganji, H. Ko. Engineering multifunctionality in gallium oxide: unveiling novel structural, electronic, and opto-mechanical attributes of gadolinium-doped $\beta\text{-Ga}_2\text{O}_3$ through advanced first-principles design. *RSC Adv.*, 2025, vol. 15, no. 49, pp. 41677–41690.
29. G. Chen, Z. Ni, D. Zheng, W. Lv. Micromechanical properties of $\beta\text{-Ga}_2\text{O}_3$ single crystal by instrumented indentation and scratch tests. *ECS J. Solid State Sci. Technol.*, 2026, vol. 15, no. 3, art. no. 034002.
30. D.A. Bauman, D.I. Panov, V.A. Spiridonov, A.Yu. Ivanov, P.A. Bogdanov, W.V. Lundin, E.Yu. Lundina, A.F. Tsatsulnikov, M.V. Tokarev, B.Y. Ber, S.S. Rachkov, D.Yu. Kazantsev, P.N. Brunkov, A.E. Romanov. Control of iron doping during the growth of bulk gallium oxide crystals by the Czochralski method. *J. Vac. Sci. Technol. A*, 2025, vol. 43, no. 4, art. no. 042804.
31. D.I. Panov, V.A. Spiridonov, O.S. Vasilev, P.A. Bogdanov, D.A. Bauman, A.E. Romanov. Laser processing of gallium oxide crystals in the preparation of samples for microelectronics. *Rev. Adv. Mater. Technol.*, 2025, vol. 7, no. 3, pp. 198–202.
32. Y. Yuan, W. Hao, W. Mu, Z. Wang, X. Chen, Q. Liu, G. Xu, C. Wang, H. Zhou, Y. Zou, X. Zhao, Z. Jia, J. Ye, J. Zhang, S. Long, X. Tao, R. Zhang, Y. Hao. Toward emerging gallium oxide semiconductors: A roadmap. *Fundam. Res.*, 2021, vol. 1, no. 6, pp. 697–716.
33. B.K. Tanner, D.K. Bowen (Eds.). *Characterization of Crystal Growth Defects by X-Ray Methods*. Springer New York, NY, 2013, 589 p.
34. V.A. Spiridonov, D.I. Panov, A.Yu. Ivanov, D.A. Bauman, A.E. Romanov. The effect of high-temperature annealing on the properties of bulk $\beta\text{-Ga}_2\text{O}_3$ obtained in different growth atmospheres. *Mater. Phys. Mech.*, 2024, vol. 52, no. 3, pp. 80–85.