


Conference materials

UDC 54.052

DOI: <https://doi.org/10.18721/JPM.183.156>

Effect of short-term heating on the morphology of AlF_3 microstructures

A.M. Dautov^{1, 2} , V.V. Fedorov^{3, 2}, K.P. Kotlyar², V.V. Lendyashova²,


T. Shugabaev^{1, 2}, V.O. Gridchin^{2, 4}, I.V. Shtrom^{4, 1}, N.V. Sibirev¹

¹ St. Petersburg State University, St. Petersburg, Russia;

² Alferov University, St. Petersburg, Russia;

³ Peter the Great St. Petersburg Polytechnic University, St. Petersburg, Russia;

⁴ Institute for Analytical Instrumentation RAS, St. Petersburg, Russia

amdautov24@gmail.com

Abstract. This work explores thermally modified AlF_3 microstructures synthesized via aluminum-zinc reaction in hydrofluoric acid. Brief heating produces cushion-like morphologies, while room-temperature growths results in cracked crystals. The thermally treated samples exhibit anisotropic brittleness and easy delamination, suggesting weak interlayer bonding. SEM-EDS and XRD analyses reveal structure-property relationships, offering insights for tailored AlF_3 microstructures in electronics and nanomaterials.

Keywords: aluminum fluoride, rosenbergit, XRD analyze, microstructures

Funding: The synthesis of AlF_3 microstructures were performed under the support of the Ministry of Science and Higher Education of the Russian Federation (state task No. 0791-2023-0004). A study of the structural properties was funded by «Tailored III-V semiconductor nanostructures with tunable morphology and broadened composition for optoelectronic applications: Phase 1 (2025)» grant number (129360164).

Citation: Dautov A.M., Fedorov V.V., Kotlyar K.P., Lendyashova V.V., Shugabaev T., Gridchin V.O., Shtrom I.V., Sibirev N.V., Effect of short-term heating on the morphology of AlF_3 microstructures, St. Petersburg State Polytechnical University Journal. Physics and Mathematics. 18 (3.1) (2025) 283–286. DOI: <https://doi.org/10.18721/JPM.183.156>


This is an open access article under the CC BY-NC 4.0 license (<https://creativecommons.org/licenses/by-nc/4.0/>)

Материалы конференции

УДК 54.052

DOI: <https://doi.org/10.18721/JPM.183.156>

Влияние кратковременного нагрева на морфологию микроструктур AlF_3

А.М. Даутов^{1, 2} , В.В. Федоров^{3, 2}, К.П. Котляр², В.В. Лендяшова²,


Т. Шугабаев^{1, 2}, В.О. Гридчин^{2, 4}, И.В. Штром^{4, 1}, Н.В. Сибирев¹

¹ Санкт-Петербургский государственный университет, Санкт-Петербург, Россия;

² Академический университет им. Ж.И. Алфёрова РАН, Санкт-Петербург, Россия;

³ Санкт-Петербургский политехнический университет Петра Великого,
Санкт-Петербург, Россия;

⁴ Институт аналитического приборостроения РАН, Санкт-Петербург, Россия

amdautov24@gmail.com

Аннотация. В данной работе приводятся результаты исследования влияния кратковременного нагрева раствора $\text{HF:H}_2\text{O-Al-Zn}$ на морфологию и фазовый состав микроструктур AlF_3 . Исследование показало, что кратковременный нагрев раствора

HF-Al-Zn способствует формированию подушкообразной микроструктуры AlF_3 . Для получившихся структур приведены результаты исследований методами растровой электронной микроскопии и рентгенодифракционного анализа. Полученные результаты расширяют возможности управления формой наноструктур на основе AlF_3 .

Ключевые слова: фторид алюминия, розенбергит, рентгенодифракционный анализ, микроструктуры

Финансирование: Синтез микроструктур AlF_3 выполнен при поддержке Министерства науки и высшего образования Российской Федерации (государственное задание № 0791-2023-0004). Исследование структурных свойств выполнено в рамках Государственного задания «Наноструктуры полупроводниковых соединений III-V с управляемой морфологией и расширенной областью состава для оптоэлектроники: 2025 г. этап 1» (код темы [129360164]).

Ссылка при цитировании: Даутов А.М., Федоров В.В., Котляр К.П., Лендяшова В.В., Шугабаев Т., Гридчин В.О., Штром И.В., Сибирев Н.В. Влияние кратковременного нагрева на морфологию микроструктур AlF_3 // Научно-технические ведомости СПбГПУ. Физико-математические науки. 2025. Т. 18. № 3.1. С. 283–286. DOI: <https://doi.org/10.18721/JPM.183.156>

Статья открытого доступа, распространяемая по лицензии CC BY-NC 4.0 (<https://creativecommons.org/licenses/by-nc/4.0/>)

Introduction

The attention of many research groups is increasingly drawn to materials based on group III-VII elements of the periodic table, among which aluminum fluoride (AlF_3) stands out prominently [1]. This material serves as an active emitting component in ultraviolet devices, independent catalyst, functions as an inorganic photolithographic resist [2]. The physicochemical properties of AlF_3 present particular interest for applications in advanced micro- and nanostructures.

Despite existing theoretical and experimental investigations [3], the full potential for creating functional microstructures remains unexplored. To explain the fundamental mechanisms governing the formation of distinct AlF_3 microstructures and fully explore their synthesis opportunities, comprehensive experimental investigations are required to systematically evaluate synthesis parameter effects. One of the most fundamental key factors for controlling material synthesis processes is temperature. Building upon previous findings [4], in this work we investigated the influence of short-term heating on the morphological and phase characteristics of AlF_3 -based structures.

Materials and Methods

The experiments were conducted in polypropylene bath. The dissolution reaction of aluminum (Al) was carried out in an aqueous hydrofluoric acid solution ($\text{HF}:\text{H}_2\text{O}\sim x:y$) using zinc (Zn) as a catalyst. The solution volume was 15 mL. Al and Zn were immersed simultaneously in the HF solution and allowed to react until complete dissolution of Al was achieved. Following complete dissolution, the solution was mixed up using an ultrasonic bath to ensure uniformity. Subsequently, 1 mL aliquots were extracted from each reaction mixture and transferred to polypropylene Petri dishes. In the first experiment, the solution was maintained at ambient temperature (25 °C) until precipitation was complete, which took approximately 20 hours. Under the second condition, the solution was heated under controlled conditions at 60°C. Precipitation occurred within 9 seconds. Table 1 summarizes the initial precursor data.

Table 1

Synthesis parameters

$\text{HF}_{\text{init}}, \%$	x	y	Al, g	Zn, mg	Al, mol	$C_m, \%$	$C_M, \text{mol/L}$	$v_{\text{HF}}, \text{mol}$	$v_{\text{HF}}/v_{\text{Al}}$
3.63	1	10	0.257	5	0.01	0.043	2.161	0.032	3.4



In all experiments, high-purity (5N) Al and Zn (Saying, China) were used for analysis. The hydrofluoric acid solution complied with Russian GOST standards (GOST 10484-78) and had a concentration of 47.5%. Sample microstructures were characterized using a SUPRA 25 scanning electron microscope (SEM) (Carl Zeiss, Germany) equipped with an Ultima 100 energy-dispersive X-ray spectrometer (EDS; Oxford Instruments). To characterize the phase composition of the synthesized structures, X-ray diffraction (XRD) analysis was performed using a Bruker Kappa APEX II DUO system equipped with a microfocus source and a two-dimensional CCD detector.

Results and Discussion

Figure 1 demonstrates SEM images of the obtained sample morphologies. When the solution was dried without heating, scanning revealed that the resulting aluminum fluoride structure was characterized by numerous cracks (Fig. 1, *a*). Notably, upon closer examination, the crystals located between cracks exhibited a visually smooth surface morphology.

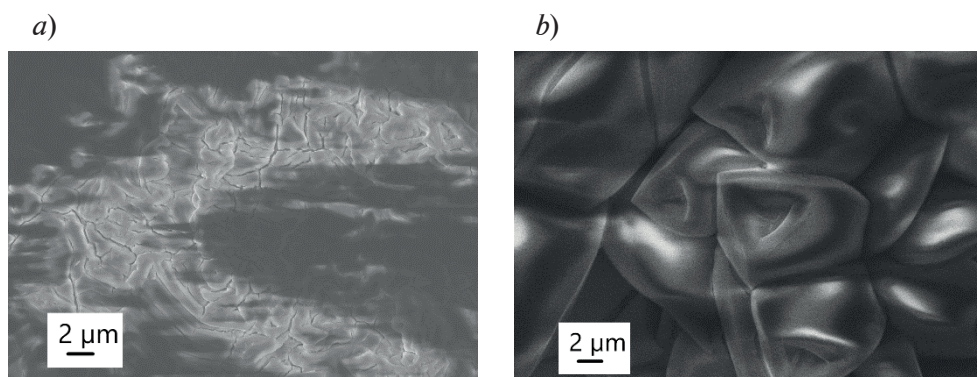


Fig. 1. SEM images show the obtained samples in isometric view: without heating (*a*), with heating (*b*)

In contrast, the solution subjected to short-term heating exhibited a distinct cushion-like morphology (Fig. 2, *a*). At higher magnification, this pillow-like structure was observed across the entire sample. Furthermore, the heat-treated sample was exceptionally brittle and exhibited a strong tendency to delaminate, readily fragmenting into thin flakes upon minimal disturbance.

The formation of cracks is typically caused by stresses in the structure resulting from non-uniform crystallization and solvent evaporation [5]. Short-term heating significantly accelerates synthesis, creating conditions for rapid nucleation and growth of fine-grained structures. Figure 2, *a* shows the XRD patterns of the obtained structures. According to the results, the spectrum of the sample synthesized without heating corresponds to rosenbergite ($\text{AlF}_3 \cdot 3\text{H}_2\text{O}$) [6]. This observation can be explained by the fact that the synthesis solution was highly diluted. Due to prolonged synthesis in a highly aqueous environment, the hydrated form of AlF_3 was obtained.

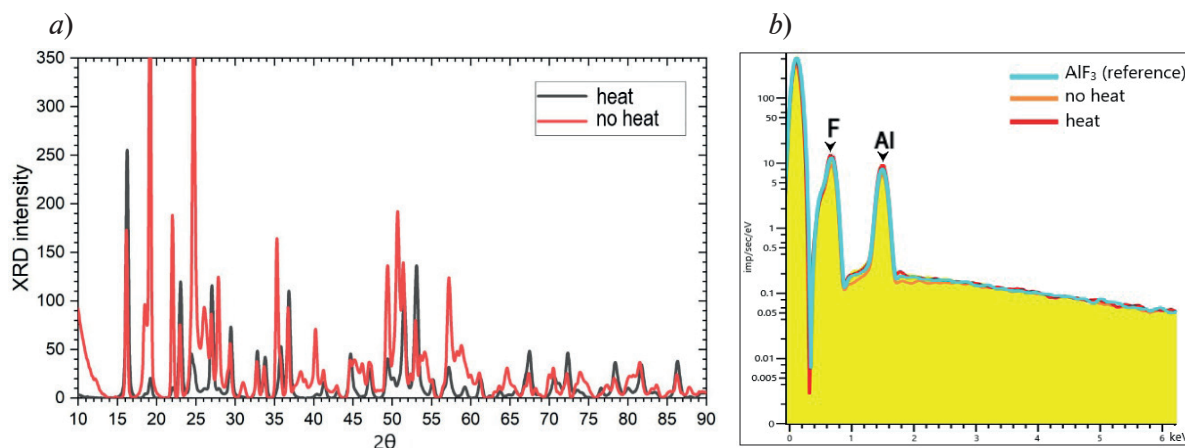


Fig. 2. XRD pattern of the synthesized samples (*a*). X-ray diffraction analysis data comparison for the synthesized samples (*b*)

However, the spectrum of the sample obtained under short-term heating requires further study. The peaks in the region of 20–30° (2θ) correspond to the crystalline phases of AlF₃. Rapid precipitation could have led to amorphous phases, which is reflected in the peak broadening. It is also noteworthy that no significant differences in the Al:F ratio of the obtained samples were detected by X-ray spectroscopy (Fig. 2, *b*).

Conclusion

This work demonstrates that short-term heating during synthesis significantly alters the morphology and crystallinity of AlF₃ microstructures, producing cushion-like formations. The rapid thermal treatment promotes crystalline AlF₃ formation, while room-temperature conditions yield hydrated rosenbergite, as confirmed by XRD analysis. The observed structural differences highlight the crucial role of temperature in controlling nucleation kinetics and material properties. Obtained results provide valuable insights for engineering AlF₃ microstructures with tailored characteristics for advanced applications in electronics and nanotechnology.

REFERENCES

1. Pandharkar R., Becker C., Budau J.H., Kaawar Z., Paulus B., A Computational Study of AlF₃ and ACF Surfaces, *Inorganics* 6.4 (2018) 124.
2. Vergara L.I., Vidal R., Ferron J., Electron induced reduction on AlF₃ thin film. *Appl. Surf. Sci.* 229 (2004) 301.
3. Daniel P., Bulou A., Rousseau M., Nouet J., Fourquet J.L., Leblanc M., Burriel R., A study of the structural phase transitions in AlF₃: X-ray powder diffraction, differential scanning calorimetry (DSC) and Raman scattering investigations of the lattice dynamics and phonon spectrum, *Journal of physics: Condensed matter*. 2 (26) (1990) 5663.
4. Dautov A.M., Lendyashova V., Shugabaev T., Khafizova A.M., Kotlyar K.P., Growth and properties of microstructures based on aluminum fluoride, In 2024 International Conference Laser Optics (ICLO) IEEE. (2024) 352–352.
5. Soshnikov I.P., Kotlyar K.P., Reznik R.R., Gridchin V.O., Lendyashova V.V., Vershinin A.V., Lysak V.V., Kirilenko D.A., Bert N.A., Cirilin G.E., Specific features of structural stresses in InGaN/GaN nanowires, *Semiconductors*. 55 (10) (2021) 795–798.
6. Clos D.P., Nekse P., Johnsen S.G., Aune R.E., Investigation on scale formation in aluminium industry by means of a cold-finger, *Light Metals*, Springer International Publishing. (2019).

THE AUTHORS

DAUTOV Albert M.
amdauto24@gmail.com
ORCID: 0000-0002-0374-7033

SHUGABAEV Talgat
talgashugabaev@mail.ru
ORCID: 0000-0002-4110-1647

FEDOROV Vladimir V.
fedorov_vv@spbstu.ru
ORCID: 0000-0001-5547-9387

GRIDCHIN Vladislav O.
gridchinvo@gmail.com
ORCID: 0000-0002-6522-3673

KOTLYAR Konstantin P.
kopkot95@yandex.ru

SHTROM Igor V.
igorstrohmail@mail.ru
ORCID: 0000-0001-8912-2570

LENDYASHOVA Vera V.
erilerican@gmail.com
ORCID: 0000-0001-8192-7614

SIBIREV Nikolai V.
nicksibirev@yandex.ru
ORCID: 0000-0002-7566-2522

Received 19.08.2025. Approved after reviewing 22.08.2025. Accepted 25.08.2025.