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EFFECT OF SHORT-TERM HEATING ON THE MORPHOLOGY OF ALF_3 MICROSTRUCTURES

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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.

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ВЛИЯНИЕ КРАТКОВРЕМЕННОГО НАГРЕВА НА МОРФОЛОГИЮ МИКРОСТРУКТУР ALF_3

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Аннотация. В данной работе приводятся результаты исследования влияния кратковременного нагрева раствора HF:H_2O-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, рентгенофазный анализ, микроструктуры.

Финансирование: Синтез микроструктур AlF3 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]).

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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 (HF:H_2O~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

HF_{init},%	x	y	Al, g	Zn, mg	Al, mol	C_m,%	C_M, mol/L	ν_{HF}, mol	ν_{HF}/ν_{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.

a) *b*)

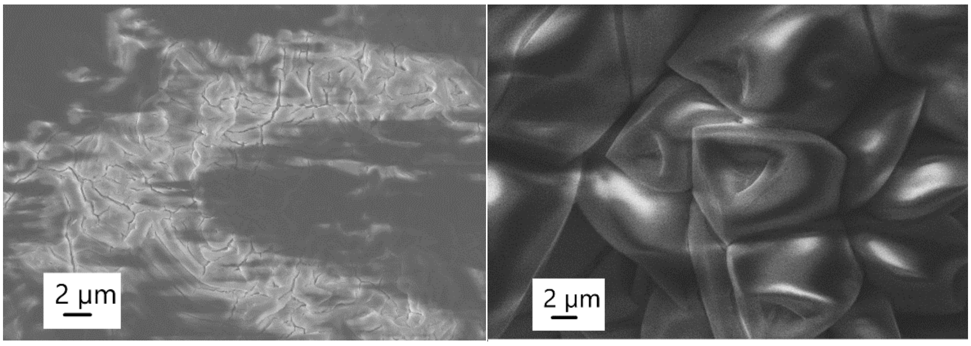

Fig. 1. SEM images show the obtained samples in isometric view: (a) without heating, (b) with heating.

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 ($AlF_3 \cdot 3H_2O$) [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.

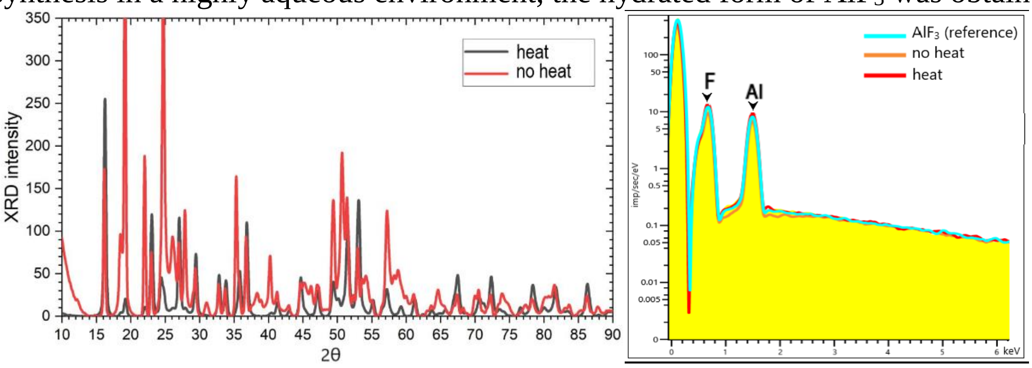

Fig. 2. (a) XRD pattern of the synthesized samples. (b) X-ray diffraction analysis data comparison for the synthesized samples.

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 AlF_3. 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 AlF_3 microstructures, producing cushion-like formations. The rapid thermal treatment promotes crystalline AlF_3 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 AlF_3 microstructures with tailored characteristics for advanced applications in electronics and nanotechnology.

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