TEMPERATURE'S EFFECT ON ALUMINUM FLUORIDE MICROSTRUCTURE

A. M. Dautov [1,2✉], I.V. Shtrom[3], N.V. Sibirev[1,3], V. I. Gridchin [1,2,3]
[1] St. Petersburg State University, Faculty of Physics, **199034** St. Petersburg, Russia;
[2] St. Petersburg Academic University, Lab of epitaxial nanotechnologies, **194021** St. Petersburg, Russia
[3]Institute for Analytical Instrumentation of Russian Academy of Sciences, 198095 St. Petersburg, Russia
✉amdautov24@gmail.com

Abstract. Aluminum fluoride is a promising material. The unique electrophysical properties of its microstructures make it attractive for diverse applications in micro- and nanoelectronics. This paper investigates the effect of heating on the microstructure of aluminum fluoride grown from an aqueous solution of hydrofluoric acid, aluminum, and zinc. We observed that short-term heating of the solution at 60°C promotes the growth of cushion-shaped aluminum fluoride microstructures. The obtained macroscopic crystal formations displayed a brittle consistency and a strong tendency to delaminate. The morphology features of the obtained structures were investigated using scanning electron microscopy.

Keywords: aluminum fluoride, hydrofluoric acid, microstructures

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Introduction

In recent years, compounds based on group III-VII elements of the periodic table have received increasing attention, with aluminum fluoride being of particular interest. Currently, aluminum fluoride finds widespread use as a standalone catalyst and as a support matrix for vanadium-containing catalysts [1]. However, its potential extends beyond this, and aluminum fluoride finds use as an active emitting element in ultraviolet devices, inorganic photolithography resists [2], and functional dielectric layers. It should also be noted that aluminum fluoride exhibits rich polymorphism [3,4], which further expands its potential applications. Under ambient conditions, α- aluminum fluoride adopts a rhombohedral perovskite structure [5], which can be described as a rotation of the fluorine octahedron around one of the cubic perovskite cell axes, while the metastable β- aluminum fluoride possesses a hexagonal structure. Calculations have revealed that the reactivity of α- aluminum fluoride is significantly surface-dependent. However, despite existing theoretical and experimental work, the potential for forming functional microstructures remains largely unexplored, and a reliable fabrication technology is still lacking. Addressing this gap requires expanding the experimental database. In this paper, we report the results of a study on the effect of short-term heating on the reaction products formed by the interaction of aluminum with an aqueous solution of hydrofluoric acid in the presence of zinc.

Materials and Methods

The synthesis of aluminum fluoride solutions was performed in polypropylene ware. The procedure involved initially adding 8 mg of zinc to each ware, which was pre-filled with an aqueous hydrofluoric acid solution ($HF:H_2O$ = 1:10), followed by the addition of 242 mg of granular aluminum. Within 24 hours, complete dissolution of the aluminum was observed; however, a portion of the zinc remained undissolved. The solution was stirred again to ensure homogeneity. Subsequently, 2 milliliters were withdrawn from the solution using a Pasteur pipette and deposited onto two silicon substrates. The solution was split and placed on two silicon substrates. The silicon substrates were cleaned beforehand by dipping them in aqueous hydrofluoric acid for 30 seconds and then rinsing them with deionized water to remove any surface contaminants. One portion of the solution dried to completion at room temperature. The second portion, rather than room temperature, was heated via a heating element. The heating element was set to 60°C, and the substrate was heated for 10 seconds. After this time, the substrate was taken off the heating element and set aside to cool down.

A study of the morphology properties was conducted via a Carl Zeiss Supra 25 scanning electron microscopy (SEM) system equipped with an Ultima 100 energy dispersive X-ray analyzer (EDX).

Results and Discussion

Figure 1 demonstrates SEM images of the obtained sample morphologies. In the case where the solution was dried without heating, SEM revealed that the resulting aluminum fluoride structure exhibited numerous cracks. It is important to note that, upon closer examination, the crystals located between the cracks exhibited a very smooth surface morphology. In contrast, the solution subjected to short-term heating exhibited a distinct cushion-like morphology. 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.

a) *b*)

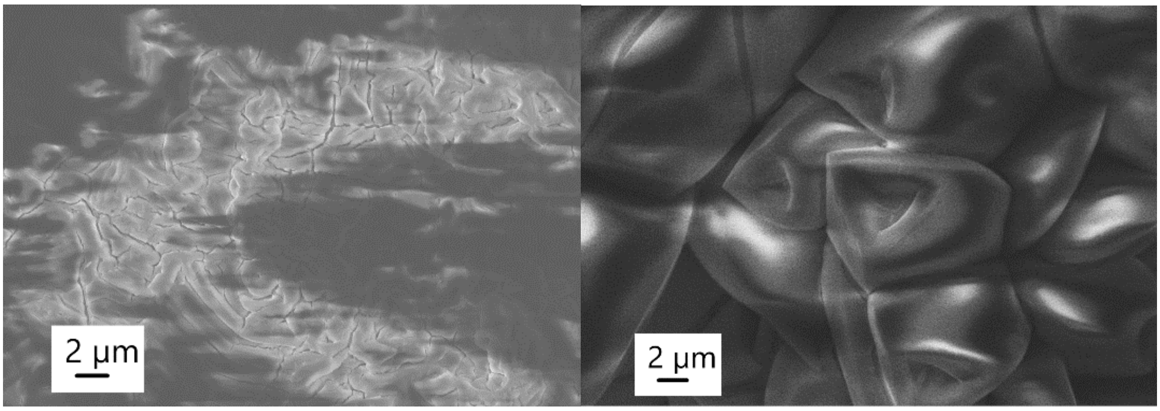

Fig. 1. SEM images of the obtained samples without (a) and with (b) heating.

Conclusion

The growth of aluminum fluoride microstructures from an aqueous solution of hydrofluoric acid, aluminum, and zinc is studied. It is shown that the short-term heating allows us to control the shape of aluminum fluoride microstructures. These findings contribute to a broader understanding of the opportunity for tailoring the microstructure of this material.

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REFERENCES

1. **Scheurell. K.. & Kemnitz. E.** (2005). Amorphous aluminium fluoride as new matrix for vanadium-containing catalysts. Journal of Materials Chemistry. 15(45). 4845-4853.

2. **L.I. Vergara. R. Vidal. J. Ferron.** Electron induced reduction on AlF3 thin film. Appl. Surf. Sci. 229 (2004) 301.

3. **König, R., Scholz, G., Scheurell, K., Heidemann, D., Buchem, I., Unger, W. E. S., & Kemnitz, E.** (2010). Spectroscopic characterization of crystalline AlF3 phases. Journal of fluorine chemistry, 131(1), 91-97.

4. **Daniel, P., Bulou, A., Rousseau, M., Nouet, J., Fourquet, J. L., Leblanc, M., & Burriel, R.** (1990). 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), 5663.

5. **Navarro. Jorge & Albanesi. Eduardo & Vidal. Ricardo & Ferrón. J.** (2016). A study on the structural. electronic and optical properties of the αAlF3 compound. Materials Research Bulletin. 83. 10.1016/j.materresbull.2016.07.007.