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Research and development of a deposition synthesis technology for piezoelectric $BaTiO_3$ thin films for use in sensors for dynamic and static pressures

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Abstract. The prospects for creating heterostructures based on ferroelectric materials have been studied. The most suitable technological parameters for forming these structures have been identified. A detailed analysis of the crystalline structure of the obtained ferroelectric films has been carried out. It has been established that at the moment of deposition, when creating thin-film multilayer systems with a temperature of the sensitive element (SE) above 700 °C, the perovskite layer loses its ferroelectric characteristics.

Keywords: sensing element, piezoelectric thin films, magnetron sputtering, stoichiometric composition, pressure sensor

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Исследование и разработка технологии синтеза напыления пьезоэлектрических тонких пленок $BaTiO_3$ для датчика быстропеременных и статических давлений

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Аннотация. Изучены перспективы создания гетероструктур, на основе сегнетоэлектрических материалов. Выявлены наиболее подходящие технологические параметры для формирования данных структур. Проведен детальный анализ кристаллического строения полученных сегнетоэлектрических пленок. Установлено, что в момент напыления, при создании тонкопленочных многослойных систем с температурой чувствительного элемента (ЧЭ) выше 700 °С, перовскитный слой теряет свои сегнетоэлектрические характеристики.

Ключевые слова: чувствительный элемент, пьезоэлектрические тонкие пленки, магнетронное напыление, стехиометрический состав, датчик давления

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Introduction

Currently, the application of methods for registering rapidly varying pressures is very broad, covering a wide range of tasks from fundamental research to practical engineering developments. In this regard, an active search for new functional materials and the development of synthesis technology for existing compounds are underway. Ferroelectrics in thin-film form are of particular interest, as piezoelectric pressure sensors are produced on their basis. The properties of piezoelectric films are determined by the technological deposition regimes, therefore it is important to determine the optimal conditions for forming layered heterogeneous structures for reliable and accurate operation of the pressure sensor.

Materials and Methods

Perovskite ferroelectric films were fabricated using radio frequency (RF) reactive sputtering of a perovskite target with a composition of $Ba_{0.8}Sr_{0.2}TiO_3$ (BST) in an oxygen atmosphere. A key advantage of this method is the ability to deposit structurally perfect metal oxide films while maintaining oxygen stoichiometry. The method offers a wide range of possibilities for varying growth parameters, including temperature, oxygen pressure, growth rate, and the substrate geometry relative to the RF electrode.

A schematic diagram of the sputtering system is shown in Fig. 1. The vacuum chamber for sputtering *1* is a stainless steel cylinder, on the base of which the external RF electrode is mounted. The vacuum system is connected to a pumping system *8*. The working gas, argon (Ar) or oxygen (O_2), is fed from a gas cylinder through inlet *2* into the chamber *1*. The ceramic target *3* is glued to the dielectric window *7* using K 400 adhesive. The K 400 adhesive contains boron nitride filler, which

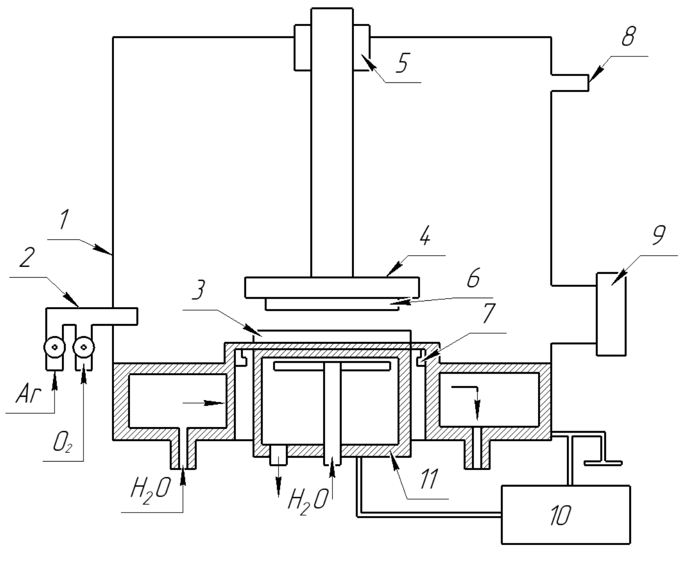

Fig. 1. Schematic of the experimental setup for ferroelectric thin film deposition

provides high thermal conductivity between the cathode and the target *3*. This protects the target from overheating and cracking. The ceramic target *3* with a composition of BST has a diameter of 47 mm and a thickness of 3.5 mm. The dielectric window *7* consists of beryllium oxide and has a diameter of 60 mm. A quartz window *9* is used for optical monitoring of the RF discharge plasma in the reactive gas atmosphere inside the working chamber *1* and for visualizing the sputtering process.

The substrate *6* in the chamber *1* (Fig. 1) is mounted on a resistive heater *4* with a platinum coil. The heater with the substrate can be moved coaxially relative to the target by means of a manipulator equipped with a vacuum seal *5*. A UV-1 high-frequency generator *10* is used as the RF discharge source. An alternating voltage with a frequency of 13.56 MHz is applied to the coaxial RF electrode *11*. The master crystal oscillator with the first amplification stage is housed in a separate enclosure. The buffer amplifier is assembled with four GU-50 tubes and the output stage with two GU-80 tubes. A “Pi” matching network with adjustable inductance and capacitances was used as the matching unit. Control of the output power supplied to the RF electrode is achieved by changing the signal at the master crystal oscillator.

Initially, the deposition chamber is hermetically sealed and evacuated to a base pressure of approximately $1\cdot10^{-3}$ Pa. Subsequently, high-purity oxygen is introduced into the chamber at a controlled flow rate of 0.4 ± 0.01 L/min to achieve a residual oxygen partial pressure of approximately $1.5\cdot10^{-2}$ Pa. Upon reaching the target pressure, the substrate is temperature-controlled and stabilized within the temperature range of 400–450 °C for approximately 5 minutes. Subsequently, an RF generator is activated, forming a high-frequency plasma discharge between the sputtering target and the heated substrate, with a target-to-substrate distance of approximately 60 mm, in the oxygen atmosphere. Throughout the perovskite film deposition process, the substrate thermocouple maintains the substrate temperature in the vicinity of approximately 600 °C. As a variation of this technology, a similar deposition process was also performed with a substrate temperature of approximately 700 °C.

Results and Discussion

The study of the crystalline structure, phase composition, and structural quality of the obtained ferroelectric films was carried out using X-ray diffraction analysis. This allowed for the identification of structural components in the stoichiometric composition of the obtained perovskite films based on the set of interplanar spacings in their structure, according to the relative intensities of the corresponding lines on the X-ray diffractogram.

X-ray diffractograms obtained for films synthesized by magnetron sputtering are presented in Fig. 2. Analysis of the diffractogram of the ferroelectric film with stoichiometric composition (Fig. 2, *a*) confirms its crystalline structure. However, the diffractogram of the film deposited at a higher temperature (700°C) (Fig. 2, *b*) indicates a disruption of the crystallographic orientation of the resulting structure, which negatively affects the stability of the ferroelectric properties of the material.

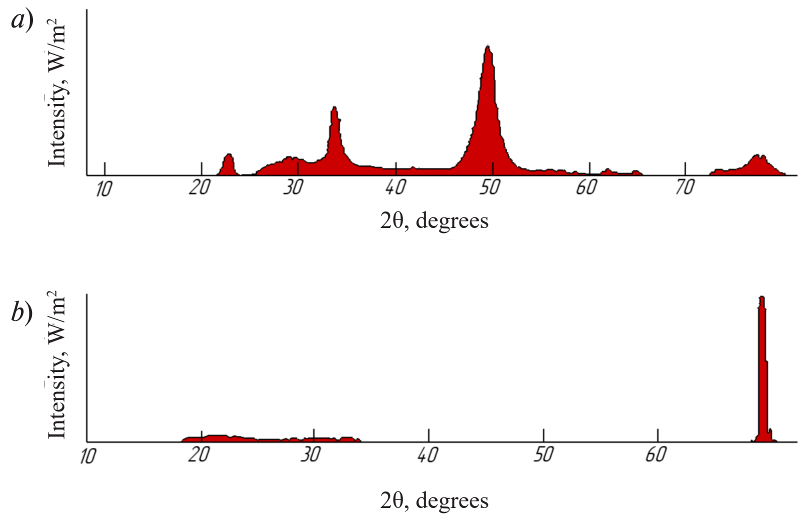

Fig. 2. X-ray diffractogram of a ferroelectric $BaTiO_3$ film deposited at a temperature of 600 °C (*a*) and 700 °C (*b*)

Research aimed at creating $BaTiO_3$ films with a perovskite structure exhibiting ferroelectric properties using radio frequency (RF) plasma sputtering has revealed optimal deposition process parameters. It was established that to achieve the best characteristics, it is necessary to maintain a residual oxygen pressure in the reaction chamber at a level of $\sim 1.5 \cdot 10^{-2}$ Pa, heat the substrate to a temperature of ~600 °C, and set the cathode-anode voltage to 245 V. Ferroelectric films obtained in this manner are characterized by a dielectric constant of ~700 and provide a capacitance tuning coefficient of ~1.5.

Conclusion

A magnetron sputtering process has been developed, based on a comprehensive research program, to enable the deposition of barium titanate ($BaTiO_3$) thin films exhibiting enhanced structural and functional stability. The optimized process parameters, derived from rigorous experimental investigation and theoretical modeling, facilitate the formation of crystalline $BaTiO_3$ films with reduced defect density and improved compositional homogeneity. These modifications contribute to a significant enhancement in the long-term operational stability of the resulting films, particularly under elevated temperatures and electrical field stresses. The specific parameters of the developed technological regime, including the target power, substrate temperature, working pressure, and gas composition, are crucial for achieving the desired film properties.

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