

Ultraviolet-Produced Lead-Free and Composite Nanomaterials for SAW Devices

Abstract

We report the specific interaction of various gases on the modified surface of acoustic wave devices for gas sensor applications using the piezoelectric ceramic material BaSrTiO₃ (BST) and various Sr concentrations. To increase the sensor's sensitivity, the conductive polymer polyethylenimine (PEI) was deposited on top of BST thin films. BST thin films were deposited using pulsed laser deposition (PLD), and PEI thin films were deposited using matrix assisted pulsed laser evaporation (MAPLE), which were then integrated into an interdigital Au electrode (IDT) test heterostructure. The layered heterostructures were further integrated into surface acoustic wave (SAW) devices in order to measure the frequency response to a variety of gases, including oxygen, carbon dioxide, and nitrogen. There were differences in the frequency shifts between the sensors when the layered structures of PEI/BST and the frequency responses of sensors based on thin films of the piezoelectric material deposited at various pressures were compared. The results of the SAW tests that were carried out at room temperature were influenced by the conditions of deposition—oxygen pressure and the proportion of strontium in the BaTiO₃ structure. Frequency shift responses were obtained for each and every one of the gases under investigation at a concentration of Sr x = 0.75. Out of all the ones that were looked at, the BST50 polymer CO₂ detection sensor had the best frequency shifts.

Keywords: Barium strontium titanate • Polyethylenimine • PLD • MAPLE • SAW device • Gas sensor

Introduction

Poor air quality is now a pressing concern due to the detrimental effects that a variety of air pollutants, such as greenhouse gases (CO₂, CH₄), volatile organic compounds (VOCs), and toxic gases (CO, H₂S, NO_x, and SO_x), have on health. Because of their numerous applications in environmental science, medicine, the automotive industry, defense, indoor and outdoor air quality measurement systems, and other fields, gas sensors are one of an air monitoring system's most crucial components. A transducer and an active layer make up gas sensors, which convert chemical reactions into measurable quantities like resistance, voltage, frequency, or current. There are many different kinds of gas sensors, each with its own set of advantages and disadvantages in terms of detection [1]. Catalytic gas sensors, for instance, are inexpensive and simple, but they require air or oxygen to function and may be affected by lead or chlorine; robust and simple-to-use thermal gas sensors, but heating wire is what causes the reaction; The electrochemical sensor measures low concentrations of toxic gases, but its failure modes are unknown; IR sensors rely solely on physical response, even though not all gases exhibit absorption in the IR domain; and sensors that are based on how sound moves. Acoustic transduction-based gas sensors can be made in submillimeter sizes, have high awareness, and can be made efficiently. Acoustic sensors are also used in a lot of different fields because they are easy to integrate with wireless communication systems [2]. A piezoelectric material is used to generate acoustic waves based on piezoelectricity in acoustic devices. When high-frequency vibrations travel through these devices, either in bulk or on the material's surface, they produce an acoustic wave that travels through the material. The resulting elastic deformation and perturbation are the result of this. When it

Yashfeen Khan*

Department of Material Science and Nano Material, India

*Author for correspondence:

ykhan123alig@gmail.com

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comes to gas detecting, two types of devices are frequently used: more precise mass acoustic wave devices (BAW) and surface acoustic wave devices (SAW) [3]. The majority of the material in BAW is traversed by the wave, including the film bulk acoustic resonator (FBAR) and the quartz crystal microbalance (QCM). In the second type, surface acoustic wave devices (SAW), acoustic waves are contained to the surface of piezoelectric materials. There are a lot of papers in the literature, including review papers, that compare these two types of sensors (BAW and SAW) and discuss their differences as well as the issues they face. Surface acoustic waves are used in SAW sensors, whereas bulk or volume acoustic waves are used in BAW sensors. In BAW devices, acoustic waves travel through the bulk structure of the active layer. Acoustic waves travel through the bulk structure of the device active layer as the piezoelectric thin film is sandwiched between two metal electrodes in the BAW structure [4]. A thickness shear mode (TSM) resonator is one of the BAW devices that is utilized the most frequently. Due to their susceptibility to additional mass deposited on the surface, ease of fabrication, and temperature stability, they are utilized extensively. Due to their ability to estimate and distinguish fluids, these devices can also be used as biosensors. One of their drawbacks is the low mass sensitivity of TSMs, despite the fact that their operating frequency range is 5–30 MHz. Sensors could be made more sensitive by using thinner devices, but this would make them fragile and hard to work with. A sensor's sensitivity increases with the amount of energy altered in the propagation path. BAW sensors have a low surface energy density because energy is dispersed across their bulk and to the opposite side. By concentrating energy on the surface, surface acoustic wave sensors (SAW) increase their sensitivity. The SAW sensor typically consists of a thin piezoelectric film with two metal electrodes on the same side of the piezoelectric substrate. An acoustic wave travels across the surface of the piezoelectric between these two electrodes, which are the input and output [5].

As an alternative to the harmful lead-based titanium compounds (PbZrTiO₃-PZT) that are used in gas.

Sensing SAW devices, barium titanate has been the subject of extensive research. Barium titanate possesses dielectric, photoelectric, ferroelectric, and piezoelectric properties as the first ferroelectric perovskite oxide material

discovered. In order, the four polymorph phases of BaTiO₃—cubic, tetragonal, orthorhombic, and rhombohedral—change with temperature [6]. However, at room temperature, its structure is tetragonal. The ferroelectric phase is formed by a number of phase transitions as the temperature drops: the cubic (centrosymmetric) phase to the tetragonal, orthorhombic, and rhombohedral phases. Ferroelectric properties are present in all of the phases mentioned earlier, with the exception of the cubic phase [7]. Due to the Ti⁴⁺ cation displacement, the formed spontaneous polarizations are oriented in the directions, respectively. Due to its improved thermal and chemical stability, BaTiO₃ can be used in a wide range of applications in addition to its electric properties. BaTiO₃ has a cubic design that is stable up to 1460 °C at its Curie temperature of 120 °C, which places it in a paraelectric state. Solid solutions formed when barium is replaced by other elements increase these materials' potential for use in a variety of fields by altering their dielectric properties. One of the requirements for applications in the optical, RF, and microwave fields is that the electrical permittivity perform in a paraelectric state due to its low losses and lack of thermal hysteresis. Because Sr²⁺ is one of the barium substituents, replacing the Ba²⁺ ions with Sr²⁺ ions decreases the transition temperature between the ferroelectric and para-electric phases monotonically [8]. The tetragonal-orthorhombic transition remained unchanged, but the orthorhombic-rhombohedral temperature transition decreased linearly as the Sr molar fraction increased for Ba_{1-x}Sr_xTiO₃ compositions with $x = 0.95$. The substance that has received the most research attention over the past two decades is barium strontium and titanate, also known as Ba_{1-x}Sr_xTiO₃ or simply BST. It is utilized across nearly the entire frequency range in low-frequency electronic circuits and microwaves [9].

BST is an important material for tuneable microwave devices because of its high dielectric constant, large electric field turnabilities, low dielectric loss, and variable Curie temperature (from 30 to 400 K depending on the strontium doping). Stage shifters, tuneable channels, defer lines, and tuneable oscillators are only a couple of instances of microwave gadget applications that have profited from its utilization. BST thin films have also emerged as a viable platform for multimodal sensing functions like pressure, force, temperature, humidity, gas detection, and infrared radiation because of their high

capacitance, switchable spontaneous polarization, pyroelectric, piezoelectric, photovoltaic, and electro-optic effects [10].

Materials and Methods

Deposition Using IDT and thin films

The $\text{Ba}_{1-x}\text{Sr}_x\text{TiO}_3$ (BST) thin films were deposited using pulsed laser deposition (PLD) in two distinct Sr concentration compositions: $x = 0.5$ and $x = 0.75$ to see how the composition affects the gas sensing response. The BST $_x$ targets were prepared using the standard sintering method. Deposition was carried out on corundum-crystalline Al_2O_3 substrates with lattice constants of $c = 12.991$ and $a = 4.785$. A Nd: was used to make the samples of BST and Al_2O_3 . The laser used was a YAG (266 nm) one with a fluence esteem of 1.65 J/cm^2 . For each sample, a repetition rate of 10 Hz was used to select the number of laser pulses, which was set at 36000 [11]. Between RT and the temperature of the deposition, there was a cooling rate of $10 \text{ }^\circ\text{C/min}$ and a heating rate of $50 \text{ }^\circ\text{C/min}$. The substrate was set to $700 \text{ }^\circ\text{C}$ in temperature. Prior to beginning the PLD interaction, the gas pressure reached $1 \text{ } 101 \text{ mbar}$ or $2 \text{ } 101 \text{ mbar}$, and the oxygen progression during the removal process was set at 35 sccm. Substrates were cooled in oxygen stream at 200 sccm after the removal technique was done. Prior to saving gold cathodes and taking gas response gauges, we put away four tests for every oxygen pressure and BST association in progression, completely looking at their morphology and basic properties [12].

The conductive polymer polyethylenimine (PEI) was deposited by means of matrix assisted pulsed laser evaporation (MAPLE). The use of a variety of natural and inorganic materials without altering their structure or inherent characteristics was the reason this method was chosen. High temperatures are not a problem for polymers. The MAPLE deposition method is used to deposit a target made of the material that will be deposited (one to five weight percent). is created. dissolved in a solvent (matrix) The substance that will be deposited can dissolve in the solvent without causing any chemical interactions, and the solvent, not the substance that will be deposited, absorbs the laser energy. Consequently, a layer is formed from the evaporated polymer molecules while the pump removes the volatile solvent molecules from the deposition chamber. The PEI target was made by dissolving 400 mg of PEI in a 1:1 mixture of deionized water and isopropyl

alcohol after 60 minutes of magnetic stirring. The target was then irradiated with the fourth harmonic of a Nd (266 nm) after being frozen in liquid nitrogen: YAG laser with a frequency of 10 Hz and 36,000 laser pulses. The remaining films had a roughness of 120 nm and a thickness of 298 nm [13].

Thermal evaporation was used to deposit interdigital electrodes on the surface of thin films for use in surface acoustic wave sensors to measure the gas response of the investigated materials. Mechanical energy is converted from an electrical signal applied to the input interdigital transducers (IDTs) of a SAW sensor into surface acoustic waves. The other pair of IDTs (output) are reached by these waves, which travel across the sensor's surface at a particular frequency. The manner in which the output IDTs convert mechanical energy into an electrical signal is determined by the characteristics of the arriving wave front. On its way to the second pair of IDTs, the wavefront alters its oscillation frequency in response to a variety of troubling conditions. Au metal terminals with thicknesses of approximately 200 nm were stored on the piezoelectrically dynamic thin layers for the IDT using an interdigital cathode veil with a time of 50 m and a digit width of 50 m. One of the troubling factors is the presence of gas molecules at the sensitive film's level. Through mass accumulation or acoustic–electric interactions, these molecules have the potential to change the oscillation frequency of the wave front. A 10–20 nm-thick introductory layer of chrome was utilized to guarantee the gold's great grip, and both warm vanishings were completed all the while. On the spot, the thicknesses of the two deposited layers were measured with a calibrated quartz crystal. Later, gold wires with a 75-meter diameter were adhered to the electrodes using conductive silver (Ag) paste with a minimum Ag concentration of 70%. The sensors were evaluated and characterized for various gases (O_2 , CO_2 , and N_2) at room temperature [14].

Characterization of the sample and saw measurements

AFM (model XE100, Park Systems, Suwon, Republic of Korea) and SEM (JSM-531 Inspect S Electron Scanning Microscope, FEI Company, Prague, Czech Republic), both working in non-contact mode and recording the topography of 20 20 m², were used to characterize the surface morphology of the deposited thin film. This allowed for good sample viewing and positioning.

Using reflection type spectroscopic ellipsometry at a fixed angle of incidence in the spectral range of 1–5 eV, from the near IR to the UV (300–1200 nm), optical properties were measured on a Woollam Variable Angle Spectroscopic Ellipsometer (VASE) system with a high-pressure Xe discharge lamp. We looked into how linearly polarized light was affected by surface reflection. In order to obtain extinction coefficients and refractive indexes for the thin films, the WVASE32 software (VASE, J.A. Woollam Co., Inc., Lincoln, NE, USA) was utilized for fitting and extracting useful data from the complex multilayer response [15].

Conclusions

This study demonstrates that BST and PEI/BST layered structures can be produced using laser deposition techniques (PLD and MAPLE). Strontium concentrations and O₂ deposition pressures were used at two distinct levels. Later, the layered structures were incorporated into surface acoustic wave devices to measure the frequency response to different gases (N₂, CO₂, and O₂). To recognize the sensors' recurrence shifts, slender movies of the piezoelectric material BST and layered PEI/BST structures were contrasted with the recurrence reactions of the sensors. The SAW tests that were conducted at room temperature produced distinct results depending on the deposition conditions (oxygen pressure and the percentage of strontium in the BaTiO₃ structure). The BST 50_0.2 mbar polymer sensor had the highest CO₂ detection frequency shift of all the sensors examined, at 0.3 MHz. In the event of a higher Sr concentration in the films, both with and without polymer, frequency shift responses were obtained for all examined gases at 0.2 mbar oxygen pressure.

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