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High-performance ultraviolet radiation sensors based on zinc oxide nanorods

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ABSTRACT

The original results of the development of the ultraviolet sensors based on surface acoustic wave resonators with apodized interdigital transducers and reversing multistrip coupler were presented. Zinc oxide nanorods were grown on the resonator surface using the hydrothermal method and were applied as a sensing layer. The surface characterization was performed by the scanning electron microscopy and X-ray diffraction. The nanorods revealed excellent sensitivity for ultraviolet radiation: the frequency shift was above 140 kHz for radiating power of 46.52 μW/cm^2 and the wavelength of 365 nm. The sensors demonstrated good repeatability and full recovery after 45 s of gas-discharge plasma impact process.

1. Introduction

Ultraviolet (UV) radiation sensors have been widely used for solving different practical problems: pollution monitoring, water sterilization, flame sensing, defects detection etc. [1]. All these applications require sensitive devices with high signal-to-noise ratio and high response rate. Silicon-based photodetectors and photomultipliers can be highly sensitive in UV region with low noise and quick response. However, they have significant limitations relating to spectral filtration, degradation and low efficiency. Besides, their application requires ultra-high vacuum environment and voltage supply [2]. All these disadvantages significantly restrict their application area. To avoid these deficiencies, the UV sensors based on wide-bandgap semiconductors (SiC, InGaN/GaN–SiC, GaN/AlN–SiC) have been developed [3].

Zinc oxide (ZnO) is prospective material for UV sensors development. Such properties as excellent radiation hardness, high electron mobility, bandgap of 3.37 eV and large exciton binding energy of 60 meV at room temperature determine the promising potential for their application in electronic and optoelectronic devices [4]. The asymmetric wurtzite structure of ZnO ensures large electromechanical coupling and results in strong piezoelectric properties. In addition, ZnO is biochemically stable and biocompatible material. The surface area of a sensing element has to be increased for sensitivity enhancement. It has been found that ZnO nanowire is more sensitive to UV light than multi-crystal ZnO at almost the same bandgap [5]. Bai et al. [6] reported high-performance integrated ZnO nanowire sensors for which large photoresponse current arises even at weak UV light signals.

Different layers and the doping technique are used for improvement of UV sensors sensitivity. Fabrication of metal-semiconductor-metal UV sensors are performed using novel nanostructured thin films of nanohole-enhanced aligned Al-doped ZnO nanorods arrays [7].

Improvement of the sensitivity of surface acoustic wave (SAW) UV sensor with an ultrathin gold layer and the ZnO nanoparticles layer deposited by a simple spin coating process on a SH-mode 41°YX $LiNbO_3$ piezoelectric substrate have been demonstrated in [8].

Compared with typical UV detectors, the SAW UV sensors are very sensitive to any slight perturbations at the substrate surface and have significant advantages such as the possibility of being combined with the integrated circuits and portability. The SAW sensors with apodized interdigital transducers (IDT-s) and multistrip coupler (MSC) have been reported previously [5]. They have shown an absorbing ability for UV light of 365 nm with the central frequency shift of 65 kHz for radiating power of 150 μW/cm^2.

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To decrease recovery process time addition layers such as poly(3,4-ethylenedioxythiophene) have been applied [9]. The reversing multistrip coupler (RMSC) has been proposed after MSC, it combines features of MSC and Bragg reflector. MSC transfers the wave into another channel, RMSC also transfers the wave into another channel, but simultaneously turns the propagation direction into the opposite.

RMSC are often applied in band pass, notch and low-loss filters, filter-banks, narrow-band spectrum analyzer with 'fan-shaped' strips, contrary-propagating SAW beams acousto-optic correlators and so on due to stopband behavior [10].

In this article, we described the highly sensitive SAW UV sensors with RMSC and ZnO nanorods grown using the hydrothermal method as a sensing layer. The study of sensitivity and repeatability of ZnO sensing coating was performed. The influence of gas-discharge plasma impact process was proposed as an approach to reduce the sensor recovery time.

2. SAW UV sensor design

2.1. SAW resonator modeling and fabrication

The design of the UV sensor was based on SAW resonator structure which consisted of two IDT-s and RMSC. Lithium niobate 128°YX-cut (128°YX-$LiNbO_3$) was chosen as a substrate material to obtain the high performance SAW resonator due to its high electromechanical coupling coefficient. The frequency access method, using an approximation of the frequency characteristics by finite complex Fourier series was applied to calculate the frequency response of IDT-s. It gives the possibility to express the lengths of each electrode by the weighting coefficients with a term of the Fourier series. The change of the periodicity of the structure was accounted by means of Legendre polynomials of 8 degree. Thus, the coordinates of the end point of the apodized metal electrodes of IDT-s were calculated using the formulas:

$$y_n = \frac{W}{2}[1\text{-}\,1(-1)^n \mathrm{h}(f)], \tag{1}$$

$$\mathrm{h(f)} = \sum_{\mathrm{m}=1}^{\mathrm{Ninit}} \left[\mathrm{a_m} \cos\left[2\pi \frac{\mathrm{f}}{\mathrm{Pinit}} \left(\mathrm{m} - \frac{\mathrm{Ninit}+1}{2} \right) \right] + i\mathrm{b_m} \sin\left[2\pi \frac{\mathrm{f}}{\mathrm{Pinit}} \left(\mathrm{m} - \frac{\mathrm{Ninit}+1}{2} \right) \right] \right], \tag{2}$$

where: $\mathrm{a_m} = \frac{\mathrm{Cj+Cinit\text{-}j+1}}{2}$, $\mathrm{b_m} = \frac{\mathrm{Cj-Cinit+1\text{-}j}}{2}$, Cj and Cinit+1-j are coefficients of the electrode of IDT, W is the aperture, Ninit is a number of IDT electrodes.

The calculation of the optimal operating characteristics with central frequency in the range of 38 MHz was performed in MATLAB as well as the generation of IDTs and RMSC topology. The simulated frequency response of the designed resonator is presented in Fig. 1(a) and experimental frequency response of the proposed SAW resonator is given in Fig. 1(b).

The microelectronic processing was carried out for resonator fabrication. Initially the 3-inch 128°YX-$LiNbO_3$ wafer was cleaned with hydrogen peroxide at 30 °C during 30 min. The aluminum film was formed by magnetron sputtering deposition process and conventional contact ultraviolet photolithography was used to obtain the topology of resonators (Fig. 2(b)). Single package (Sip 6 M) was used for placing the sensor (Fig. 2(c)).

2.2. ZnO sensing layer synthesis

One dimensional ZnO nanostructures and nanostructured films can be synthesized by numerous methods: vapor phase techniques [11] and solution-based methods [12]. Vapor phase growth methods such as chemical vapor deposition [13], metal-organic chemical vapor deposition [14], physical vapor deposition [15] and molecular beam epitaxy [16] are well developed and often applied for the synthesis of high quality metal and metal oxide nanomaterials. Solution based methods provide the formation of nanostructures with different morphologies and structures by self-organization at the low-temperature chemical [17,18] and electrochemical [19,20] processes without expensive vacuum and other microelectronic technologies. While electrochemical growth allows obtaining well-aligned ZnO nanorods on the conductive films the low-temperature hydrothermal synthesis is suitable for synthesis of nanostructures with different shapes almost on any substrates. The hydrothermal method has following advantages: low-cost, easy handling, scalability, opportunity to form various structures depending on the process parameters and integration with other classical technologies such as lithography, epitaxy or diffusion [21].

ZnO nanorods were synthesized as sensing layers on the fabricated SAW resonator structures by the low-temperature hydrothermal synthesis. Two main steps in the growth of the ZnO nanostructures by the hydrothermal method are used: the first one is the preparation of a ZnO seed-layer by sol-gel method for providing the crystallization centers and the second one is the growth of nanostructures array in salt solution [18].

For the first step zinc acetate dihydrate (ZnAc) $Zn(COOCH_3)_2{\cdot}2H_2O$ was used as the starting salt material to prepare ZnO seed-layer by sol–gel method. ZnAc was dissolved in isopropanol ($(CH_3)_2CHOH$). Then monoethanolamine (MEA) $HOCH_2CH_2NH_2$ solution was added at room temperature. The concentration of ZnAc was 0.3 mol/l and molar ratio of MEA to ZnAc was kept to 1.0. The mixture was stirred by the magnetic agitator at 65 °C until the clear and homogeneous solution was formed. Prepared sol-gel was cooled to the room temperature and filtered with 0.22 μm membrane. Film deposition was carried out in air at room temperature. The precursor solution was spin coated at 3000 rpm for 30 s on the substrate. After each coating the obtained film was dried at 100 °C for 30 min at the sintering furnace. The preheat-treatment temperature of 100 °C is required for the complete evaporation of organics and the initiation of formation and crystallization of the ZnO film. After deposition of the third layer, the resulting thin films were annealed at 400 °C in air during 1 h to obtain the stable film with crystallization centers.

Second step was carried out in zinc nitrate based solution. Analytically pure zinc nitrate ($Zn(NO_3)_2{\cdot}6H_2O$) and hexamethylenetetramine ($C_6H_{12}N_4$) were used in equimolar concentration. The zinc nitrate concentration was 0.05 mol/l. The chemicals were solved in deionized water, resulting in a transparent solution under magnet stirring during 5 min at room temperature. The as-pretreated substrate with the formed seed-layer was immersed and suspended in the mixed solution and the growth of ZnO was carried out by heating the reaction solution from room temperature to 95 °C and then stayed during 30 min without any stirring at the sintering furnace. The as-grown pattern was rinsed with deionized water for several times and dried in air at 60 °C before characterization.

The Al-doped ZnO nanorods were synthesized on the fabricated SAW resonator structure by the similar process. The aluminum nitrate ($Al(NO_3)_3$) was added to the solution for the hydrothermal synthesis. The aluminum concentration of 0.2 at.% was measured in resulting structures by energy-dispersive X-ray spectroscopy (EDX, MIRA3, TESCAN).

2.3. Surface characterization

Parameters of the chemical synthesis process as well as substrate preconditioning and seed layer formation significantly affect the quality and the shape of the obtained nanostructures. Deposition of the seed layer was carried out to provide the growing