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Optimization of porous anodic alumina nanostructure for ultra high sensitive humidity sensor

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ABSTRACT

In this report, we are going to present the development and optimization of high-performance porous alumina growth having a large surface to volume ratio for ultra-high sensitive humidity sensing. The control of the structure of pores, such as the diameter of pores, depth of porous layer, etc., has shown crucial implications for the sensitivity of sensor; and there are precisely fine-tuned with the anodization process parameters. The sensitivity of the prepared sensors reaches upto 793.02% while reported sensitivity for conventional capacitive humidity sensors is much less. Besides, the prepared sensor maintains its fair responsivity as well as sensitivity even up to 100 KHz unlike 10 KHz reported by other researchers so far; and it gives an added advantage for its applications where humidity measurement at high frequencies are required The samples are characterized by Field Emission Scanning Electron Microscopy (FESEM, FEI NNS 450) to support the results and impedance spectroscopy were employed for the electrical characterization of the sensor. An equivalent electrical circuit is obtained to represent the electrical performance at different humidity level and it is in good agreement with the observed results.

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1. Introduction

Now-a-days, humidity measurement is a significant factor to be considered in most industries, laboratories, in the preservation of archeological monuments of historical importance, meteorological studies and in many areas of our lives [1–6]. Humidity sensors based on different materials and detection range are available over the years [7]; with certain advantages and limitations. There are numerous studies which have been focused on the use of polymers due to their excellent performance [8]. Ceramic based humidity sensors, on the other hand, can be manufactured with great reliability and are therefore available commercially [9]. They offer great advantages owing to thermal stability, mechanical strength, high resistance to chemical attack and fast response [10–12]. However, their usage is restricted due to hysteresis, aging and hence require calibration.

There has been a renewed interest in the Porous Anodic Alumina (PAA) film for humidity sensing applications. This is because of the highly ordered pores with tunable diameters, periodicity, and their high density distribution. The self-assembled nano pores are highly uniform so that the surface area of the sensor is increased by large quantity [13–18]. The high yield ratio significantly reduce the overall cost of the sensor and therefore ensuing reliable sensor fabrication on the commercial scale [19].The average sensitivity of PAA-based humidity sensors is 0.2-0.5 pF/RH% while relative humidity sensors based on porous alumina fabricated by Micro Electro-Mechanical System (MEMS), shows the sensitivity of the order of 15 pF/RH% [20].

In present work, PAA based humidity sensor was reported with much higher sensitivity than the value reported in the earlier studies. PAA film was fabricated using two-step anodization method in 0.30 M oxalic acid solution [21]. The purpose of the present investigation is to optimize the fabrication of PAA layer, with controllable periodicity, pore diameter and thickness of porous layer for humidity sensing applications. The quality of aluminum sheets and their surface pre-processing impact on the nanostructuring, electro-polishing time was investigated. The sensing response was investigated as a function of frequency to optimize the best condition for humidity sensing of PAA layer.

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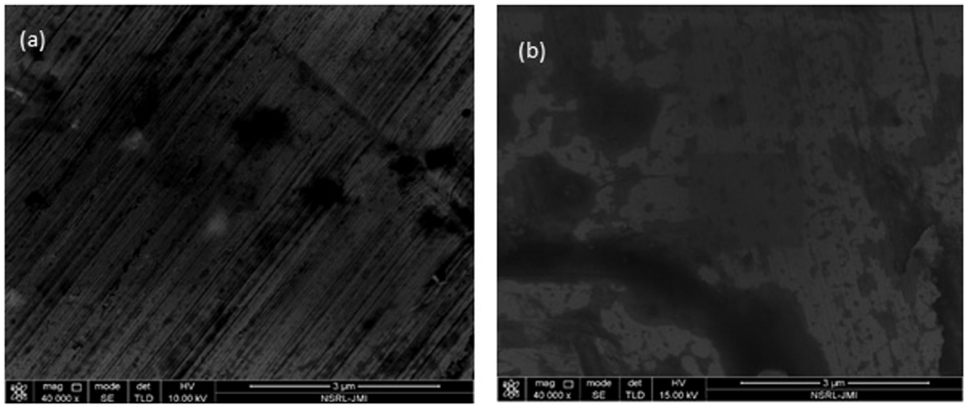

Fig. 1. FESEM micrographs (a) aluminum sheet, and (b) annealed aluminum sheet at 500 °C.

Table 1
Anodization parameters used for making PAA layer.

Sample Name	Electrolyte Conc.	Anodization voltage (V)	Anodization time (min.)	No. of stages
S1	0.30 M oxalic acid	14.5	30	1
S2	0.30 M oxalic acid	30	30	2

2. Experimental

2.1. Fabrication of PAA layer

The porous anodic alumina layer was produced by two-step anodization method [22]. The aluminum sheets of high purity (99.0%) with thickness 20 μm were annealed at 500 °C for 3 h, to remove mechanical stress from the surface, followed by ultrasonication with acetone for 15 min in order to remove grease from the top surface. Subsequently, the sheets were rinsed thoroughly in 5 wt% of sodium hydrooxide (NaOH) solution for 30 s at 60 °C to peel off the naturally grown aluminum oxide layer. The surface of the sheets were smoothened electrochemically by doing electro-polishing in 1:4 vol. ratio of perchloric acid ($HClO_4$) and ethanol(C_2H_5OH) at 4 °C. The electro-polished samples were then anodized in 0.30 M oxalic acid solution. The porous oxide layer formed in the first anodization was etched out using alumina etchant (mixture of 4.0 wt% *ortho*-phosphoric acid and 2.0 wt% chromic acid) solution at 65 °C for 1 h [23]. The hexagonal indentations were observed on the surface of aluminum sheet after treating with alumina etchant solution. Afterward, second anodization was carried out in the identical constraint as anodization done initially. Finally, pore widening was done in 0.1 M phosphoric acid solution.

3. Results & discussion

3.1. Pretreatment of aluminum sheet

The initial surface condition of aluminum sheet plays an important role on the nano pore formation [24,25]. It is essential to minimize the surface defects and make the surface smoother prior to anodization. Fig. 1 shows the FESEM image of (a) aluminum sheet and (b) aluminum sheet annealed at 500 °C. It can be seen that annealing results in large grain boundaries which are favored sites for development of pores. The annealing procedure reduces mechanical stress from the sheet and also increases the average grain size. Therefore high purity annealed aluminum sheets are the most desirable starting material for the formation of self-organized assembly of nano pores.

3.2. Effect of electro-polishing (EP) time on self organized nano pore formation

Electro-polishing was performed on six samples for different times (40, 80, 120, 160, 200 and 240 s) at 20 V. FESEM images of the aluminum surface after EP are presented in Fig. 2. Electro-polishing is the most important step in the pretreatment of aluminum sheet before anodization. The pretreatment process results in the reduction of surface roughness, controlled and reproducible generation of an array of the nano pore. Our investigation shows that the surface roughness is minimum for the sample electro-polished for 80 s as observed from the FESEM studies. However, more holes will occur on the surface if EP was done for more than 160 s.

Fig. 3 shows the variation of current density with time, where J_a, J_b, J_c, J_d, J_e and J_f are current density of samples a, b, c, d, e and f, respectively. The process is conducted under steady potential, initially current density decreases rapidly with time, and a minimum of current density is quickly achieved. EP follows 3e- dissolution stoichiometry according to the following equation

$$2\mathrm{Al} \rightarrow 2Al^{3+} + 6\mathrm{e}^{-} \quad (1)$$

Subsequently, Al atom removes uniformly in EP and current density remains almost constant.

3.3. Effect of anodization voltage on pore morphology

The growth of self-aligned pores on electro-polished aluminum sheet occurs within a narrow window of voltage, for a given electrolyte. In this study, two samples were anodized in 0.30 M oxalic acid solution as mentioned in Table 1. Fig. 4 shows the FESEM micrographs of samples anodized at (a) 14.5 V (b) 30 V and respective (c) EDS spectra with table. As observed, pore size increase with an increase in applied voltage. This is because the higher voltage leads to the strengthening of the electric field. As a result of a stronger electric field, the oxide layer dissolution is increased. The pore diameter of the formed anodic aluminum oxide (AAO) template depends heavily on the anodization potential, irrespective of the type of electrolyte used [26]. Sample S1 has predominantly ordered structure of pores whereas sample S2 did not have regu-

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