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Effect of Al₂O₃ coatings on microstructural and optoelectronic properties of porous Si/ SnO₂ composites.

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Abstract:

In this work, Al₂O₃ coating effect on morphology, structure and optoelectronic properties of Si/SnO₂/Al₂O₃ porous matrix composites (PMCs) were investigated. A three-staked thin layers deposited on a <100> oriented silicon substrate made these composites. First, porous silicon layers were achieved by electrochemical etching method. Then Al₂O₃ and SnO₂ layers were successively deposited by physical and chemical vapor deposition, respectively. Morphological and micro-structural properties of the as prepared composites were evaluated by Scanning electron microscope, energy dispersive X-ray spectroscopy and X-ray diffraction. Results proved that Al₂O₃ concentration alters notably the porosity of the PMCs. Variable angle spectroscopic ellipsometry (SE) revealed a high correlation between the optical constants (n, k) and the PMC microstructure. Impedance spectroscopy revealed a semiconductor-metallic transition at high frequency in the temperature range between 340 to 410°C.

Keywords: pSi/SnO₂/Al₂O₃, Microstructure, Optical and electrical properties

1. Introduction

Metal oxides include compounds like TiO₂, ZrO₂, Cr₂O₃, SiO₂, Al₂O₃ and SnO₂. Several compositions and properties of this family have been widely investigated for a

wide range of industrial and technological applications [1-7]. They are used as insulators, semiconductors, conductors and superconductors. These binary oxides are currently being investigated as potential SiO_2 substitutes in the next generations of semiconductor devices [1]. Among these materials, Tin oxide (SnO_2) thin films have been proved useful for a variety of applications including gas sensors, batteries, fuel cells, photovoltaic cells, photodetectors, transparent electronics, and thin film transistors (TFT) due to their excellent characteristics including chemical stability, high electrical conductivity and optical transparency. The properties of tin dioxide and alumina layers deposited on porous Si (pSi) depend on several factors such as the method of preparation, variety of phase states and degree of dispersity [8-12]. The SnO_2 thin films are n-type semiconductors with direct optical band-gap of 3.9 eV [13, 14]. Whereas SnO is a metastable material at room temperature and becomes more stable at higher temperatures. Various methods have been used to prepare SnO_2 film such as electron beam evaporation, thermal evaporation of oxide powders, magnetron sputtering, spray deposition, chemical vapor deposition (CVD) [15-17] and sol-gel. In this work, two deposition techniques, physical vapor deposition (PVD) and CVD, were respectively used to deposit Al_2O_3 and SnO_2 nano-crystals due to many advantages such as large production area and uniform distribution. Both techniques, metal oxide nanocrystals growth in the cavities of porous silicon permits an easy control of material compositions [18]. Appropriate porosity and high specific surface area of oxide supports are required for catalyst or ion exchange applications. In fact, ion exchange in the nanopores of the Al_2O_3 / SnO_2 binary system for the catalytic reaction is not widely recognized. Interestingly, the Alumina Tin oxide catalysts remains an urgent need to increase the catalytic activity and decrease the abrasive properties of the porous matrix composites (PMCs) as the average pore diameter of support materials corresponds to their particle size. Thus, the pore diameter is significant, as catalysts resist sintering highly particularly in the case of a metal catalyst [19]. Such properties encourage the use of Alumina Tin oxide as a tunnel barrier and dielectric gate [20-22]. Spectroscopic ellipsometry (SE) [23-24] is highly appropriate to analyze thin layers such as pSi/ SnO_2 / Al_2O_3 . In this work, pSi/ SnO_2 / Al_2O_3 samples were modeled as alternately overlapping five layers, each of them having their intrinsic properties. The main objective is to depict the effect of the Al_2O_3 content on pSi / SnO_2 microstructural and opto-electronic properties.

2. Experimental detail

Electrochemical anodization was used to prepare the porous Si (pSi) layer. A current density of 10 mA/cm² was maintained for a silicon substrate immersed in a hydrofluoric acid (HF) Solution. The obtained pSi layer was etched in an acid mixture solution (HNO₃: 10%, HF: 20%, H₂O₂: 70%) followed then rinsed with distilled water and dried under oxygen to yield a pSi model with an ordered pore structure. The CVD and PVD methods served to deposit on the pSi substrate a thin layer of tin dioxide (SnO₂) using SnI₂ and O₂ respectively as reagents. SnO₂ in the first place and therefore Al₂O₃ at different concentrations.

The thin film of SnO₂ was grown by CVD on pSi. A vertical CVD configuration was designed to lay the films [25]. Before depositing the film, the chamber was placed under vacuum at 0.1 mbar. The tin precursor was a powdered tin (II) iodide (SnI₂, 99%, from Alfa Aesar Company) for the SnO₂ deposition. The SnI₂ evaporation rate was kept between 0.004 and 0.6 g / h by setting the SnI₂ reservoir temperature in the range of 28 to 460 °C. A flow of argon gas is used to carry the SnI₂ vapor to the reaction zone in a separate quartz tube. The substrates were sited on a quartz glass sample holder, about 10 cm away from the orifice of this separate tube. The sample holder rotation was set at 19 rpm during deposition to assure uniform deposition. The O₂ gas flow rate was varied from 10 to 200 sccm. The carrier gas (Ar) and the reactive gas (O₂) flows were monitored by mass flow regulators. The chamber pressure was kept at 46 mbar during the deposition. The deposition temperature was set to 550°C. The film thickness is estimated to 190 nm for 37 min deposition time. The Al₂O₃ films were deposited with different concentrations: (a) 0% (b) 3.7% (c) 7,4% (d) 11,1% (e) 14,8% (f) 19% relative to the concentration of SnO₂ by a RF reactive magnetron sputtering at a deposition temperature of 660°C.

The obtained pSi/ SnO₂/Al₂O₃ structure was then thermally treated under oxygen and introduced into an oven under oxygen flow at a temperature equal to 1500°C.

3. Characterizations

The crystallographic structures of pSi /SnO₂ / Al₂O₃ were determined by X-ray diffraction (XRD) using a Bruker D8 advance X-ray diffractometer equipped with CuK α radiation ($\lambda_{\text{CuK}\alpha} = 1.5406 \text{ \AA}$). To fully recognize the distribution of crystals on the surface and the correlation with optoelectronic properties the surface microstructure and morphology were analyzed using the scanning electron microscope (SEM) and the atomic force microscope (AFM) techniques. The optical properties of pSi /SnO₂ / Al₂O₃ were investigated using Spectroscopic Ellipsometry (SE) analysis were recorded with a GES5 SOPRA equipped with a rotating polarizer SE, in the wavelength interval from 300 to 1200 nm with a step of 1 nm under incidence angle of 78°. Data acquisition and analysis were performed using the Winelli II Software (version 2.0.0.0). SE served to obtain an accurate determination of film thickness, the percent of each component, including vacuum, refractive index (n) and extinction coefficient (k) Optical dispersion coefficients 'n' and 'k' were calculated for the pSi /SnO₂ / Al₂O₃ thin films structure using the BEMA model. The electrical measurements configuration was carried using a Hewlett-Packard HP 4192 impedance analyzer. For this purpose, two silver electrodes that were painted on both ends of the sample. The electrical tests were carried out over a large interval of temperature from 190 to 370 K and a frequency varying in 5 Hz -13 MHz range.

4. Results and discussion

4.1 Morphology and structure of pSi /SnO₂/Al₂O₃

4.1.1 Energy dispersive X-ray (EDX) analysis

The EDX results of film composition were obtained. Figure 1 displays an EDX spectral representation of the pSi/ SnO₂/Al₂O₃ thin films and the relative elementary composition. Mainly Si, O, Al, and Sn were present in the samples. The atomic percent of these elements were 69.3%, 20.5%, 7.5%, and 1.4%, respectively. EDX surface mapping (Fig. 2) of pSi/ SnO₂/Al₂O₃ clearly shows a uniform distribution of all the elements throwing the whole surface.

4.1.2 SEM analysis

Figure 3 shows a cross section view of pSi/ SnO₂/Al₂O₃ layers after coating as a

function of alumina concentration: (a) 0%, (b) 3.5%, (c) 7%, (d) 11%, (e) 19%, (f) 24% relative to the concentration of Sn. Notably, the layers begin to grow as the alumina crystals are incorporated in pSi/ SnO₂ pores. Small pseudo -pyramids are thus formed by nucleation [26,27]. The sectional image shows the incorporation of alumina crystals in the pores of the pSi / SnO₂. A surface layer of alumina also emerged. The SEM micrograph of the pSi /SnO₂ / Al₂O₃ structure reveals the coalescence of small nanoparticles on the surface. The rate of voids decreases in accordance with the percent of deposited alumina; which improves the crystallization of the film.

4.1.3 AFM analysis

The surface roughness of the sample and the alteration caused by Al₂O₃ on the surface morphology of pSi / SnO₂ were studied via AFM. According to Figure 4, the surface morphology of the pSi/ SnO₂/Al₂O₃ films is uniform and homogenous. Upon the deposition of Al₂O₃ at different concentrations, the particles undergo an increase of the distribution consistency, a change of the size, the movement of the atoms position and a recrystallization. The minor spherical grains agglomerate depending on the alumina concentration. Table 1 illustrates the particle size, the mean square (RMS) and the roughness as obtained for these films.

4.1.3 X-ray diffraction analysis

Evolution of XRD spectra of pSi/SnO₂/Al₂O₃ samples annealed at 660°C is presented in Fig.5. Eight diffraction peaks corresponding respectively to SiO₂ [101], Al₂O₃ [311], Al₂O₃ [511], SnO₂ [115], and SnO₂ [120] were depicted.

To calculate the mean crystallite size of Al₂O₃, we used Debey Scherrer's equation

$$[30]: \quad D = \frac{0.9 \lambda}{\beta \cos \theta} \quad (2)$$

with D is the average size of crystallite, λ is the wavelength of the X -rays, θ is the Bragg diffraction angle, and β is the adjusted FWHM. The Al₂O₃ crystallites size increases from 2.8 nm to 6.7 nm as the Al₂O₃ concentration increases. The crystallites size increase was attributed to a diffusion of aluminum crystals from the surface towards the interior of the pSi coated with SnO₂, which improved the crystallinity of

the structure of the films.

4.2 Opto-electrical characterization

4.2.1 Optical analysis

The ellipsometric results are interpreted via an optical model which exactly matches the configuration of the studied surface. The samples contained porous layers of different compositions superimposed in parallel, as shown in figure 6. The compositions of each layer are detailed in Table 1. Our proposed model is in congruency with De La et al. [29, 30]. Based on the approximation of the effective medium (EMMA) of Bruggeman [31], Fig. 6 shows a model consisting of 4 vertically superimposed layers of different densities. The proposed structure is composed of vacuum mixtures, SnO₂, Si, and Al₂O₃ layers, each having its intrinsic compositions. The optical model is accepted when the root means squared error (RMSE) is inferior to 9% [32].

$$RMSE = \sqrt{\frac{1}{2N - P - 1} \sum_{j=1}^N [(\tan \psi_j^m - \tan \psi_j^s)^2 + (\cos \Delta_j^m - \cos \Delta_j^s)^2]} \quad (3)$$

The physical parameters (n, k) are adequately determined when the model fits the experimental $\tan \psi$ and $\cos \Delta$ data. Fig. 7 illustrates the used multilayer model. Ψ and Δ are the so-called ellipsometric parameters. They respectively stand for the amplitude ratio and phase difference between the p-polarization and s-polarization components of the polarization state of the incident light. N is the number of points, P the number of parameters, m the measured spectra and s the simulated spectra. Optimization resulted in congruency between the experimental results and theoretical fit. The curves in Fig. 8 exhibit a close agreement throughout the spectral range. This same method is also useful to measure the thickness of the studied layer based on the interference between the reflected rays. Table 1 records the layer thickness and the corresponding RMSE values. The optical constants including the refractive index (n) and the extinction coefficient (k) were extracted and evaluated as a function of the Al₂O₃ concentration (Fig.8). The refractive index n and the extinction coefficient k variation depend on the concentration of Al₂O₃, which is attributed to the diffusion of cations along the grain boundaries of Al₂O₃. An opposite flux of vacancies was thus

created towards the surface of the pSi / SnO₂ layer. The vacancies can condense and then reach the SnO₂/ Al₂O₃ interface to form cavities under the Al₂O₃ grain boundaries.

4.3.1 Conductivity measurements

The complex impedance was measured on all the pSi/SnO₂/Al₂O₃ samples within a frequency range between 100 Hz and 13 MHz, and temperature (190-370°C). The corresponding diagrams are shown in the representation of Nyquist $Z'' = f(Z')$ in Fig.9. In practice, several contributions to a dielectric response of an oxidized materials such as grains, grain boundaries, interface are available. The semicircles relative to pSi/SnO₂/Al₂O₃ display different radii (dissimilar to Debye). This effect is attributed a dipolar system involving multi-relaxation processes [33]. We conclude that all samples are semiconductors. In addition, the resistance R₀ changed as a function of the deposited alumina concentrations. The peak Z'' intensity variation pleads in favor of a low-capacity semiconductor region assigned to the response of the SnO₂ grain embedded in alumina, and the variation of vacuum rate among the samples.

4.4.1 Imaginary part of the impedance

The evolution of the imaginary part Z'' of the pSi/SnO₂/Al₂O₃ sample impedance with frequency at several temperatures is shown in Fig. 10. A maximum of Z'' gives the frequency $f_{\max}$ relaxations as governed by the Arrhenius [34-37] law:

$$f_{\max} = f_0 e^{-\frac{E_a(Z'')}{K_B T}} \quad (4)$$

Where $E_a(Z'')$ is the activation energy, f_0 is the characteristic phonon frequency and K_B is the Boltzmann constant. The activation energy relative to the relaxation process $E_a(Z'')$ was calculated according to Eq. (4) by the plotting $\log(f_{\max})$ vs. reciprocal temperature. Table 2 shows that the increase of activation energy with alumina content to 2.31eV, with an Al₂O₃ concentration equal to 19% and its decrease beyond this rate. This variation is attributed to the incorporation of Al₂O₃ in pSi/SnO₂, and the decrease in the vacuum rate, that facilitates the rate of jump activated thermally. The decrease in $E_a(Z'')$ when [Al₂O₃] is equal to 24% is a possible result of the nucleation of a new surface layer where Al₂O₃ crystals are very far apart; which ceases the

thermally activated jump [38]. This behavior can also be assigned to vacancies in deposited alumina and to interface defects resulting from Tin dioxide vacancies occupied by oxygen atoms as the surface was coated with Al₂O₃. The values of Ea(Z") for pSi/SnO₂/Al₂O₃ (Fig. 9) reveal two activation energy domains with temperature. in the variation of the electrical properties of the prepared thin film is due to the presence of a new oxide formed in the pores of Si . This phenomenon was favored by the transfer of the oxygen supplied during the external oxide dissociation. The oxide dissociation is due to the excessive number of cation created at the grain boundaries, thus diffusing through the layer. Conductivity abides by power law with the pulsation given by the equation below [39]:

$$\sigma_t = \sigma_{dc} + \sigma_{ac}(T, \omega) \quad (5)$$

Where ω is the angular frequency, T is the absolute temperature, σ_{dc} is the independent frequency conductivity or dc conductivity and σ_{ac} is the ac conductivity.

4.5.1 Frequency dependence of ac conductivity

The conduction mechanism of pSi/SnO₂/Al₂O₃ dispersions was determined by calculating the conductivity values of dispersions over the frequency range [1 Hz, 10 Hz] and at different temperatures as shown in Fig.11. The alternative conductivity σ_{ac} at different temperatures was modelled by Jonscher's universal power law:

$$\sigma_{dc} = A \omega^S \quad (6)$$

A is the constant dependent on temperature, S is the material property which can have any value between 0 and 1, and σ_{ac} is the ac conductivity. The evolution of S with temperature depends on the conduction mechanism. The exponent S expresses the relative reduction in the size of alumina crystals with frequency and is defined as follows:

$$S = \frac{d \ln \sigma_{ac}}{d \ln \omega} \quad (7)$$

The frequency exponent S raised from the slope of ln($\sigma\omega$) [Fig. 12(a)] declines in parallel with temperature. The charge transfer, described as a superposition of the various conduction phenomena, is responsible for these results. pSi/SnO₂/Al₂O₃

morphology contains metal islands surrounded by amorphous alumina regions. Electronic wave functions lie in amorphous regions; but they can be delocalized in metallic regions. Metal conduction takes place in the metal islands while the charge transfer, is done by jumping in the amorphous regions. an inter-fibrillar and an intra-fibrillar conductions occur inside the metal region by hopping loads from one end of a chain to the other. The equation correlated with the model is written as follows [40]:

$$S = 1 - \frac{6 K_{BT}}{W_m} \quad (8)$$

T is the absolute temperature, W_m and K_B are respectively, the maximum height of the barrier and the Boltzmann constant. The value of W_m (Table 2) is determined

from the adaptation of S as $1 - \frac{6 K_{BT}}{W_m} T$

W_m was found to increase slightly in accordance with the concentration of Al₂O₃ up to 1.81eV, then it decreases slightly. Arrhenius law was applied to interpret the experimental results of dc conductivity [41]:

$$\sigma_{dc} = \frac{C}{T} e^{-\frac{E_a(dc)}{K_B T}} \quad (9)$$

Whereby, C is a constant and E_a is the activation energy for the hopping conduction. Figure 12(b) shows the plot of ln(σ_{dc}·T) vs.1000/T for pSi/SnO₂/Al₂O₃ thin films. According to Arrhenius law, the dc conductivity of the films decreases when temperature increases. This evolution shows the thermal activation mechanism of the electrical conduction, which indicates that the pSi/SnO₂/Al₂O₃ hybrid system has a semiconductor behavior. The activation energy E_a (dc) is extracted from the slope of ln(σ_{dc}·T) vs.1000/T. E_a(dc) values are summarized in Table 2. The electrical results prove that, in thermal fluctuation, sufficient energy can be supplied to a dipole hopping across the potential barrier from one position to another equilibrium position [42-44].

5. Conclusion

The opto-electrical properties of a SnO₂/Al₂O₃ deposit in a porous silicon layer were investigated. The structural properties were found to depend significantly on the Al₂O₃

concentration. XRD spectra showed that the concentration of Al_2O_3 deposited on pSi/SnO₂ was vital to improving crystallinity. After the deposition of Al_2O_3 under oxygen on pSi/SnO₂, the structure tends to crystallize for a temperature that reaches 660°C. The ellipsometric study of pSi/SnO₂/ Al_2O_3 shows the improvement of optical properties (refractive index, extinction coefficient) as a function of the Al_2O_3 concentration. Consequently, the refractive index is raised while the extinction coefficient becomes lower with the concentration of Al_2O_3 . This change is attributed to the progressive pores filling as a function of Al_2O_3 concentration and structure alteration. Moreover, according to the impedance measurements, the ac conductivity obeys the universal power law. The transport of charge carriers was using the CBH model. these results exhibits a novel semiconducting behavior that is suitable for thin film engineering and functional coating applications

Author Declarations :

- Ethics approval and consent to participate..... applicable for that section.
- Consent for publication.....applicable for that section.
- Availability of data and materials.....applicable for that section.
- Competing interests.....applicable for that section.
- Funding.....applicable for that section.
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Figure 12

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