


NANOSTRUCTURED ELECTRODE MADE WITH SILVER NANOWIRES COVERED WITH TiO₂ NANOCRYSTALS

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ABSTRACT: Optoelectronic devices rely heavily on transparent conducting electrodes (TCE) made of indium oxide (ITO). However, indium scarcity, lack of flexibility, and high costs have driven the search for nanostructured materials, such as silver nanowires (AgNWs), which offer optical and electrical properties comparable to ITO. This research develops a TCE using AgNW and titanium oxide (TiO₂) nanocrystals, nanomaterials synthesized by the Polyol and Sol-Gel methods respectively, deposited on glass by centrifugation. The deposition of the thin layer of TiO₂ nanocrystals on the AgNWs improves the

adhesion of these nanostructures to the glass. Furthermore, it provides excellent thermal and chemical stability, prolonging the useful life of the electrode. The nanomaterials were characterized by UV-vis spectroscopy, electron microscopy, and the four-point probe method, ensuring their morphological, optical, and electrical properties, essential to obtain an excellent optoelectronic device.

KEYWORDS: Nanostructured, AgNWs, TiO₂ nanocrystals and electrode

INTRODUCTION

There is currently a great interest in the field of nanotechnology through nanostructured materials; in particular, metallic nanostructures, especially those made of copper (Cu), silver (Ag), or gold (Au), as they show great potential in their electrical, optical and mechanical properties, for future applications in optoelectronic devices; as is the case of TCE, An essential device for various electronic devices, used in everyday life, from flat screens to smart windows and in next-generation electronic components

such as transparent transistors and solar cells [1]. Considering that ITO is the most commercial electrode on the market due to its high transmittance ($\geq 80\%$) and its resistance of $15 \Omega/\text{sq}$ [2], however, their flexibility is limited, which restricts their applications on highly flexible devices [3]. In addition, its manufacture requires high temperatures and toxic compounds such as indium, which makes it a relatively expensive technology. Due to these limitations and the emergence of new transparent electrode technologies, various nanomaterials have been developed, such as metallic nanowires, particularly AgNWs, which can be produced on a large scale and deposited under ambient conditions. In addition, they offer very high conductivity and transmittance, these being the two essential properties for a TCE, surpassing the optoelectronic properties of ITO. Therefore, this research aims to study and develop a TCE with nanostructured materials that serve as alternatives to ITO, such as AgNWs. This nanomaterial does not require high temperatures for its deposition, allowing its application in flexible devices, and reducing its weight and manufacturing costs, since it requires very little nanomaterial to achieve high-efficiency optoelectronic properties. However, the useful life of these electrodes made with AgNWs is shorter than that of ITO, since this nanomaterial, when in contact with the environment, changes its surface, leading to the formation of silver sulfate nanoparticles (Ag_2SO_4) due to the sulfidation of silver [4]; which contributes to the degradation of the AgNWs and the electrode itself, a problem that can be overcome by depositing a thin film of TiO_2 nanocrystals on the AgNWs, to protect the nanowires already deposited from the environment and preventing their oxidation, providing good stability and durability to both the nanomaterial and the future electrode. In addition, the thin film of TiO_2 nanocrystals improves surface homogeneity and increases the adhesion of these nanowires to the glass, preventing their detachment; also, through a post-deposition thermal process, it improves the conductivity and transmittance of the device, since organic residues such as polyvinylpyrrolidone (PVP), an insulating polymer that remains on the surface of the nanowires after the entire synthesis and purification process, are evaporated. Apart from the fact that the contact between the nanowires and therefore their degree of conductivity increases.

EXPERIMENTAL

Materials

Silver nitrate (AgNO_3 , 99.8 %), ethylene glycol (EG), polyvinylpyrrolidone (PVP), sodium chloride (NaCl , 97 %), titanium isopropoxide ($\text{C}_{12}\text{H}_{28}\text{O}_4\text{Ti}$, 97 %), isopropyl alcohol ($\text{C}_3\text{H}_8\text{O}$, 99.84 %), nitric acid (HNO_3) and ethyl alcohol, were obtained from Aldrich Chemical, Ltd.

Synthesis of silver nanowires

To perform this synthesis silver nitrate (AgNO_3) and polyvinylpyrrolidone (PVP) are dissolved in ethylene glycol (EG), considering their boiling point at $197\text{ }^\circ\text{C}$, allowing synthesis at high temperatures. Another agent is sodium chloride (NaCl), which directly affects the morphology of metal nanoparticles, decreasing the concentration of silver chloride (AgCl). These reactions facilitate the formation and growth of nanowires, influencing their size and shape. The synthesis was developed using the Poliol method [5], following the following steps. In the first step, 0.094 moles of AgNO_3 were dissolved in 15 ml of EG until a homogeneous solution was obtained. In the second step, the solution obtained was mixed, with another solution containing 0.110 moles of PVP and 0.100 moles of NaCl, previously dissolved in 15 ml of EG. The latter solution was kept under magnetic stirring at $58\text{ }^\circ\text{C}$ until a creamy solution was obtained. In the third step, the creamy suspension is placed in a preheated oven at $100\text{ }^\circ\text{C}$ for 2 hours. After this time, the AgNWs obtained are purified through a washing process with ethyl alcohol, followed by centrifugation at 2000 rpm for 45 minutes. This process is repeated three times, eliminating excess liquid containing EG, PVP, and nanoparticles not formed as nanowires. In the end, approximately 450 mg of AgNWs with a diameter of 72 nm and a length of $27\text{ }\mu\text{m}$, were obtained by storing them in ethanol at $4\text{ }^\circ\text{C}$.

Synthesis of titanium oxide nanocrystals

Through the Sol-Gel method [6], the synthesis of TiO_2 nanocrystals carried out, and the precursor solution (solution 1), was prepared by mixing 3.13 ml of titanium isopropoxide ($\text{C}_{12}\text{H}_{28}\text{O}_4\text{Ti}$) with 0.5 ml of isopropyl alcohol ($\text{C}_3\text{H}_8\text{O}$); a mixture that magnetically stirred for 10 minutes. In a separate container, 37.5 ml of ultrapure deionized water was mixed with 0.5 ml of nitric acid (HNO_3) (solution 2), adjusting the pH to 6, achieving a good morphology in the nanocrystals, shaking vigorously at $65\text{ }^\circ\text{C}$ for 10 minutes. Then (solution 1) was slowly added to (solution 2), forming a milky solution and initiating the hydrolysis process, which continues to be stirred at the same temperature for 1 hour and 30 minutes after this time is allowed to cool to room temperature at which time the TiO_2 nanocrystals begin to precipitate. Finally, it is centrifuged at 2000 rpm for 5 minutes to extract the liquids, preserving the white powder (TiO_2 nanocrystals). The powder was transferred, to an open container inside a range hood to evaporate the remaining liquids; Approximately 400 mg of TiO_2 nanocrystals, with diameters of 15 nm, were obtained by storing them in a hermetically sealed flask.

Deposition of nanostructures AgNWs / TiO₂ nanocrystals

The synthesized AgNWs nanostructures were dispersed in 15 ml of ethanol at a concentration of 1.5 %, depositing 90 μ l of this nanomaterial using the Spin Coating method [7] on glass substrates. She performed four depositions at room temperature at rotation frequencies of (1000, 2000, 3000, and 4000) rpm for 30 seconds respectively, repeating this procedure four times with intervals of 1 minute on each of the substrates. This process is performed to consider the most convenient rotation frequency, establishing a balance between the complete coverage of the sample and the position in which the nanowires are randomly arranged to form the film. Once the deposition completed, a thin layer of TiO₂ nanocrystals was superimposed on each of the AgNWs films already deposited; material that was synthesized and dispersed in 10 ml of ethanol at a concentration of 1 %, depositing 90 μ l of the solution on the nanowires, by the Spin Coating method at a rotation frequency of 2000 rpm for 30 seconds at room temperature, a process performed only once on each of the four deposited samples.

Post-deposition heat treatment thin films of AgNWs / TiO₂ nanocrystals

The electrical resistance of AgNW networks can be high due to the low contact between the nanowires, so several post-deposition techniques have been developed to increase their adhesion. Techniques include thermal annealing [8], mechanical pressing [9], chemical treatments [10] or laser annealing [11]. Thermal annealing has been shown to significantly improve transparent nanowire-based metal electrodes by decreasing contact resistance between them [12]. For this reason, each of the four thin samples of AgNWs/TiO₂ nanocrystals obtained was heat-treated using a horizontal quartz tube furnace that can reach high temperatures. First, to evaporate the remaining solvents present, the temperature was initially set at 160 °C for 10 min. Second, the films were subjected to optoelectronic characterization, selecting the sample that obtained the highest conductivity and transmittance; which will establish the frequency of rotation to which the following six samples must be subjected. Performing the same deposition process mentioned above these six new samples were exposed to six different temperatures: (120, 140, 160, 180, 200, and 220) °C for 10 minutes respectively; after this time of thermal exposure, the electrical resistance in some samples is reduced, in others, due to their excess temperature, the nanostructures can be affected or destroyed in the worst case and due to their low temperature, the liquid residue does not evaporate, increasing its electrical resistivity. Once the thermal process is completed, a new optoelectronic characterization is carried out for each of the six films obtained, selecting again the new sample that presents the highest transmittance and conductivity, which will determine the temperature to which the electrode should be exposed. Finally, to obtain greater precision in the optoelectronic response of the device, the last five samples are deposited, with the rotation frequency and temperature previously selected, but in this case, exposed to different times, which are (10, 12, 15, 18 20) minutes respectively; to determining the exact time of the thermal process; thus obtaining the most efficient TCE and most suitable for its application in optoelectronic devices.

Characterization techniques

The nanomaterials of AgNWs and TiO₂ nanocrystals after being deposited were characterized by the four-point technique and UV-vis spectroscopy, to corroborate whether they meet the optoelectronic conditions for their application as TCE.

RESULTS AND DISCUSSION

Analysis of results of films manufactured at (1000, 2000, 3000, and 4000) rpm

The AgNWs were deposited, on four glass substrates, varying their rotation frequency at (1000, 2000, 3000, and 4000) rpm for 30 seconds. A thin film of TiO₂ nanocrystals was then deposited on each of them. These samples were subjected to heat treatment, initially set at 160 °C for 10 minutes to evaporate the solvents present in these films; once the thermal process was completed, the films were optically and electrically characterized, allowing the optimal rotation frequency for the electrode manufacturing process to be determined. The average results obtained for transmittance and resistivity as a function of rotation frequency over 30 seconds are presented in Table 1.

Deposition AgNWs				Deposition of TiO ₂ nanocrystals				Heat treatment		Film resistance	Transmittance (550 nm)
Deposition	N° of depositions	Rotation frequency (rpm)	Time (s)	Deposition	N° of depositions	Rotation frequency (rpm)	Time (s)	Temperature (°C)	Time (min)	Ω/sq	%
90	4	1000	30	90	1	2000	30	160	10	8 ± 2	68.9
90	4	2000	30	90	1	2000	30	160	10	8.2 ± 0.6	73.6
90	4	3000	30	90	1	2000	30	160	10	14 ± 2	71.1
90	4	4000	30	90	1	2000	30	160	10	23 ± 2	72.2

Table 1: Manufacturing parameters and optical properties. Transmittance at 550 nm wavelength and electrical measurements.

Figure 1(a) shows the resistances obtained for the four depositions at different rotation frequencies, obtaining less resistance at lower rotation frequencies, increasing as its rotation frequency increases.

Among the values obtained, the recommended one for the application in the electrode corresponds to the film centrifuged at 2000 rpm for 30 seconds, obtaining a resistance of (8.2 ± 0.6) Ω/sq, demonstrating a value lower than the commercial ITO (15 Ω/sq). In Figure 1 (b), transmittance data at a wavelength of 550 nm show an increasing relationship in the optical properties of prepared films with rotational frequencies of 3000 and 4000 rpm compared to those at 1000 rpm. This increased centrifugation speed, leaving less nanomaterial on the surface of the substrate, resulting in an increase in its transmittance and lower conductivity. On the other hand, the film deposited at 2000 rpm reaches a transmittance of 73.6 %, a value lower than that of the ITO, recorded by 86.5 %, making it

essential to carry out a process of temperature variations in this film to obtain an increase in its transmittance. In addition, the glass used as a substrate has a transmittance of 89 % at a wavelength of 550 nm, being highly transparent in both the near and visible infrared, with an optical behavior similar to that obtained by AgNWs/ TiO₂ nanocrystals. However, the deposited films show a reduction in their transmittance, reaching a minimum value at 430 nm, marked by the green circle with dashed lines, as shown in Figure 1(b). This decrease is due to the absorption of Ag [13].

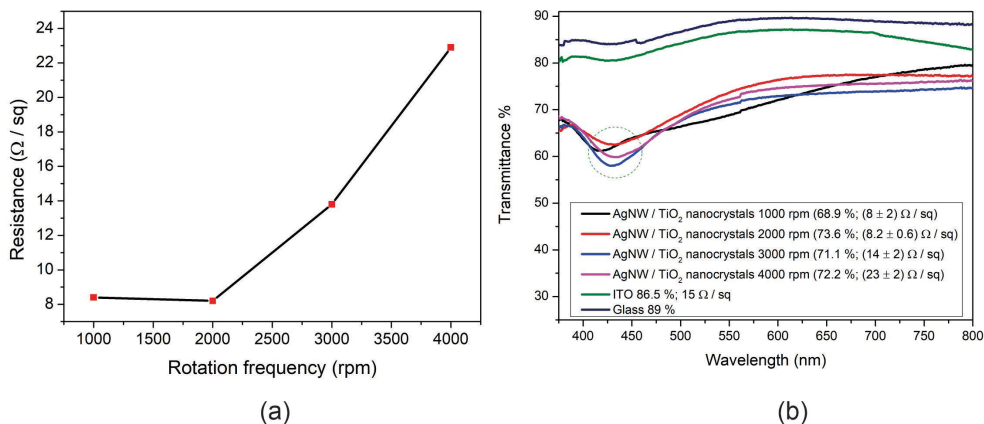


Figure 1: (a) Film resistance as a function of rotational frequency, (b) Spectrum of transmittance of films manufactured at (1000, 2000, 3000, and 4000) rpm for 30 seconds.

Temperature variation for films at 2000 rpm

Four depositions of AgNWs, followed by a deposition of TiO₂ nanocrystals, were made on each of the six previously washed glass substrates, centrifuging them at 2000 rpm for 30 seconds; each of the deposited samples exposed to a heat treatment, whose temperatures were (120, 140, 160, 180, 200 and 220) °C for 10 minutes respectively. Once the thermal process was completed, the samples were optically and electrically characterized to determine the optimal temperature, which obtained the highest conductivity and transmittance. The temperature that will be considered, for the following samples will determine the exact annealing time and thus get the most efficient electrode for future optoelectronic applications. The average results obtained both for transmittance and resistance as a function of temperature for a time of 10 minutes are shown in Table 2.

Deposition AgNWs				Deposition of TiO ₂ nanocrystals				Heat treatment		Film resistance	Transmittance (550 nm)
Deposition	N° of depositions	Rotation frequency (rpm)	Time (s)	Deposition	N° of depositions	Rotation frequency (rpm)	Time (s)	Temperature (°C)	Time (min)	Ω/sq	%
90	4	2000	30	90	1	2000	30	120	10	22 ± 2	73
90	4	2000	30	90	1	2000	30	140	10	16 ± 2	73.5
90	4	2000	30	90	1	2000	30	160	10	8.2 ± 0.6	73.6
90	4	2000	30	90	1	2000	30	180	10	17.5 ± 0.9	74.1
90	4	2000	30	90	1	2000	30	200	10	24 ± 2	76
90	4	2000	30	90	1	2000	30	220	10	29 ± 2	78.7

Table 2: Manufacturing parameters and optical properties. Transmittance at 550 nm wave-length and electrical measurements.

The results obtained in Figure 2(a), mark three critical temperature ranges in the thermal process. The first is located between temperatures [120 – 160] °C, where an increase in resistance is obtained due to the presence of liquids and organic residues in the film. The second range is located between temperatures (160 – 220] °C, presenting small deteriorations in its nanostructures due to high temperatures, influencing its conductivity. Finally, the third range is located at the temperature of 160 °C, this being the ideal temperature achieving the lowest resistance in the film due to the increase in contact between the AgNWs and their greater surface homogeneity.

Figure 2(b) shows, that there are no changes in transmittance, which is present at the 550 nm wavelength. The first range in the thermal process corresponds to temperatures between [120 – 140] °C, while in the second range, corresponding to temperatures between [180 – 220] °C, there is a substantial increase in transmittance due to the small breaks suffered by the nanostructures, caused by high temperatures, allowing light to pass between them. On the other, in hand film exposed to the temperature of 160 °C, a decrease in transmittance is observed in the region near the ultraviolet, due to its multiple connections between AgNWs, generating an increase in both the absorption and scattering of light due to the thickening in these contact areas. The results show variations in its transmittance in the visible range due to the thermal process, an essential aspect since it seeks to reduce resistance without affecting its optical properties. Advisable, to make a variation in time for the sample whose centrifugation of the AgNWs was carried out at 2000 rpm with a thermal process of 160 °C for 10 minutes to obtain the best electrode to replace the ITO.

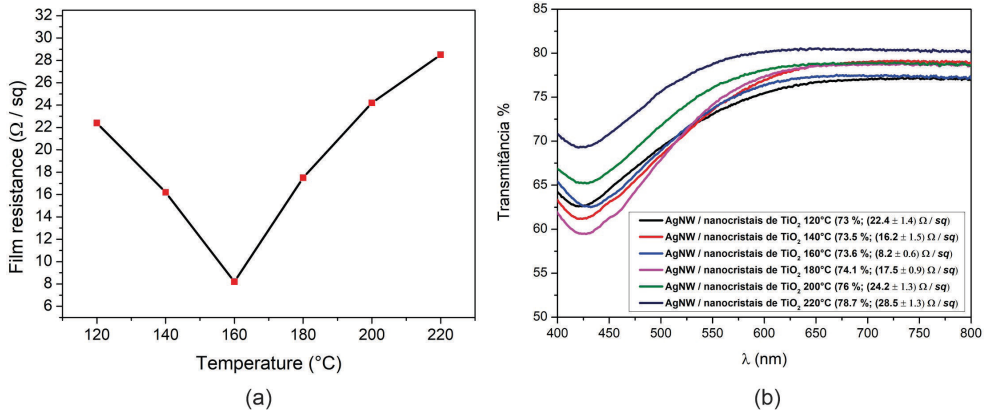


Figure 2: (a) Resistance as a function of temperature, (b) Transmittance spectrum of six films, manufactured at 2000 rpm for 30 seconds, exposed to heat treatment of (120, 140, 160, 180, 200, and 220) $^{\circ}\text{C}$ for 10 minutes, respectively.

Morphological analysis of surfaces

The images obtained by SEM are presented, of the previous samples exposed to different temperatures; these images reveal the morphological changes in the nanostructures due to the variations in temperatures they were exposed to, dividing into three intervals.

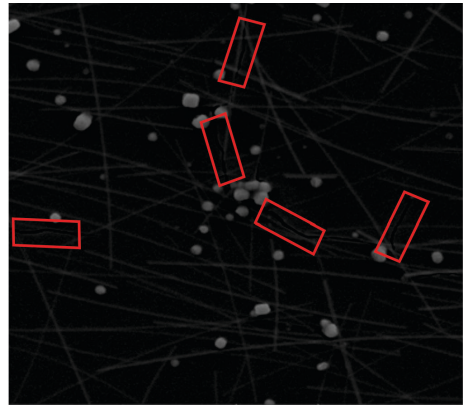
The first interval corresponds to temperatures between [120 – 140] $^{\circ}\text{C}$. The sample exposed to 120 $^{\circ}\text{C}$ presents liquid and organic residues, observed in the image within the white circles as shown in Figure 3(a), which did not evaporate due to the low temperature to which the sample was exposed. In addition, the sample exposed to 140 $^{\circ}\text{C}$ had small grooves on its surface, enclosed by the red rectangles as seen in Figure 3(b), due to the low evaporation of liquids in the sample.

The second interval corresponds to the temperature of 160 $^{\circ}\text{C}$, where a very compact surface is shown, with excellent dispersion and good contact between the nanostructures, as shown in Figure 3(c); also without organic residues, which allows good conductivity and transmittance in the film.

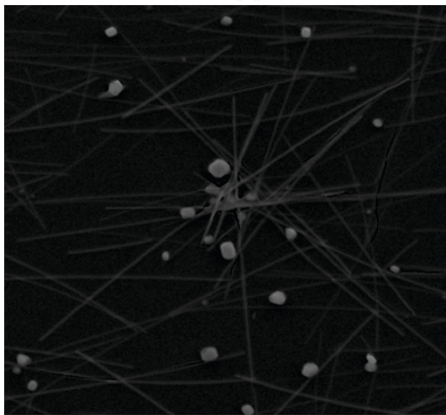
The third interval corresponds to the highest temperatures, between [180 – 220] $^{\circ}\text{C}$. In the sample exposed to 180 $^{\circ}\text{C}$, small breaks are observed in the AgNWs, giving rise to the presence of small nanoparticles, as shown within the green circles in Figure 3(d) these breaks in the nanostructures are due to the exposure of the sample to high temperatures. Finally, in the samples exposed to (200 and 220) $^{\circ}\text{C}$, it is observed how the nanomaterial begins to degrade from the temperature of 200 $^{\circ}\text{C}$, as shown within the orange squares, observed in Figure 3(e) presenting a greater degradation at 220 $^{\circ}\text{C}$ leading to small, breaks in the surfaces, shown in Figure 3(f), drastically interfering with the conductivity of the electrode, decreasing the number of connections between the nanostructures, causing a very low conductivity.



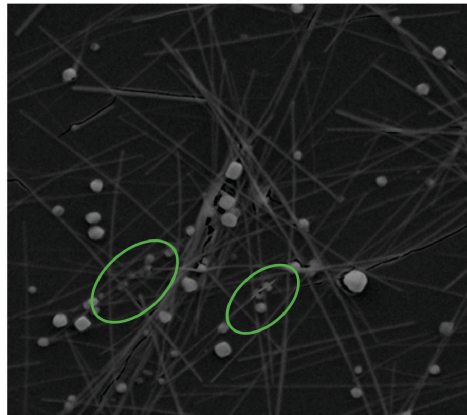
(a)



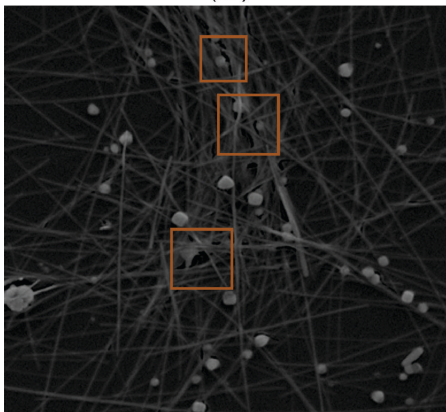
(b)



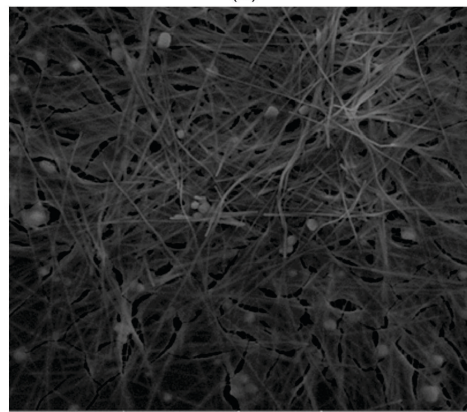
(c)



(d)



(e)



(f)

Figure 3: SEM of AgNWs/nanocrystal TiO_2 films with a thermal pre-fired of (a) 120 °C, (b) 140 °C, (c) 160 °C, (d) 180 °C, (e) 200 °C and (f) 220 °C.

Variation of thermal exposure time to 160 °C for thin films of AgNW / TiO₂ nanocrystals

Once the temperature is determined, the thermal exposure time is established, where the thin films of AgNWs / TiO₂ nanocrystals reach lower resistance and higher transmittance, demonstrating variations in its topography, significantly influencing its optoelectronic characteristics, as detailed in the data recorded in Table 3.

Deposition AgNWs				Deposition of TiO ₂ nanocrystals				Heat treatment		Film resistance	Transmittance (550 nm)
Deposition	N° of depositions	Rotation frequency (rpm)	Time (s)	Deposition	N° of depositions	Rotation frequency (rpm)	Time (s)	Temperature (°C)	Time (min)	Ω/sq	%
90	4	2000	30	90	1	2000	30	160	10	8.2 ± 0.6	73.6
90	4	2000	30	90	1	2000	30	160	13	7.8 ± 0.7	78.3
90	4	2000	30	90	1	2000	30	160	15	7.6 ± 0.5	84.7
90	4	2000	30	90	1	2000	30	160	18	7.9 ± 0.8	81
90	4	2000	30	90	1	2000	30	160	20	8 ± 2	75.3

Table 3: Manufacturing parameters and optical properties. Transmittance at 550 nm wavelength and electrical measurements, concerning (10, 13, 15, 18, and 20) minute times.

Figures 4 (a) and 4(b) show the graphs of the results obtained for resistivity and transmittance, respectively; in these graphs, it can be seen that the film exposed to 160 °C for 15 minutes, is the most optimal, later applied as an electrode in various optoelectronic devices.

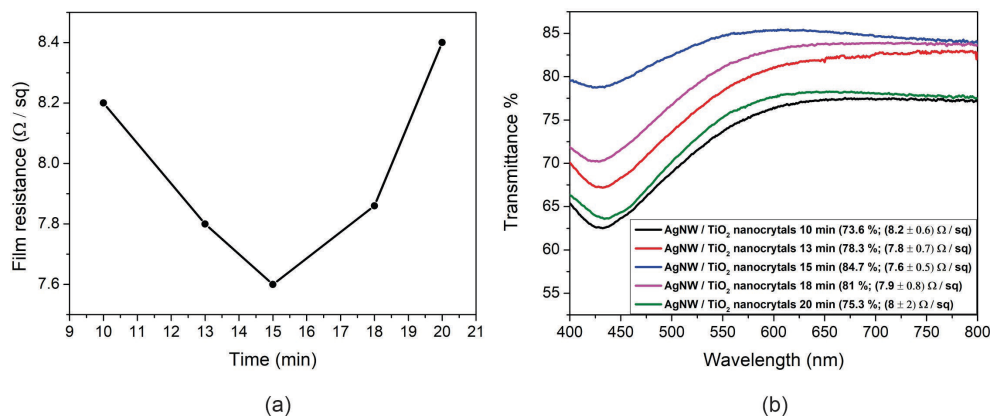


Figure 4: (a) Resistance as a function of time and (b) Transmittance spectrum of five films exposed to a thermal process of 160 °C for (10, 13, 15, 18, and 20) min.

CONCLUSION

The electrode made with AgNWs / TiO₂ nanocrystals on a glass substrate, presents optoelectronic performance with high expectations, after a heat treatment whose objective was the evaporation of liquid residues present in it, reducing the resistance between AgNWs by increasing contact between them. In addition, the coating with the thin film of TiO₂ nanocrystals manages to reduce the resistance between the glass substrate and the nanowires, giving the electrode a good surface homogeneity, thus achieving the optoelectronic optimization of the electrode. It was found that the maximum optimization of the electrode was completed at a temperature of 160 °C for 15 minutes, presenting a resistance of 7.6 Ω/sq and a transmittance of 84.7 %, placing it among the best standards described in the literature. In the same way, thanks to thermal after treatment, the electrodes AgNWs / TiO₂ nanocrystals demonstrate an optoelectronic performance equivalent to that of ITO, which has a resistance of around 15 Ω/sq and a transmittance that reaches up to a value ≥ 80 %. Importantly, unlike the latter electrodes, AgNWs/TiO₂ nanocrystal electrodes are transparent in the near-infrared spectral range which may open doors to new applications such as developing wide spectral range solar cells. In addition, the process of manufacturing nanostructured electrodes does not require specialized equipment, making them more economical.

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