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Influence of Thickness and Number of Silver Layers in the Electrical and Optical Properties of ZnO/Ag/ZnO/Ag/ZnO ultra-Thin Films Deposited on the Glass for Low-Emissivity Applications

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ABSTRACT

We report on transparent ZnO/Ag/ZnO and ZnO/Ag/ZnO/Ag/ZnO thin-films were deposited on the glass substrate by RF and DC sputtering for ZnO and Ag targets, respectively. The electrical and optical properties of the single and double Low Emissivity coatings were investigated with respect to the deposition time of Ag mid layer. The visible transmittance remains about 65% for single and 45% for double Low-E coatings as the Ag deposition time increases. Besides, the decrement of sheet resistance makes lower thermal emissivity of the coatings. In agreement with the optical performance, lower thermal emissivity can strongly increase NIR reflectance as the Ag layer thickness increases. The carrier concentration increases gradually as the sheet resistance decreases and the increase of the free carriers and conductivity of the coatings lead to a sharp rise of the NIR reflectance. The results show that the characteristics of Low-E coatings depend on both the continuity of the metal layer and the metal thickness. Prog. Color Colorants Coat. 12 (2019), 83-91© Institute for Color Science and Technology.

1. Introduction

The Low Emissivity (Low-E) coatings are ultra-thin layers of metals and metal oxides that are deposited on the glass. These coatings are used for selecting the transmitted spectrum. Low-E coatings transmit the visible spectrum wavelengths, but reflect the infrared spectrum wavelengths simultaneously. Low-E coatings are first used in the manufacture of IGUs (Insulating Glass Units) for buildings. Because of the electrical conductivity of these films, many other applications such as transparent conductors in organic or color-sensitive solar cells [1, 2], smart switches [3], gas sensors [4-6], and LEDs [7] prioritize them for super technology investigations.

Two groups of materials are considered for use as Low-E coatings [8]: first group, doped metal oxides with a thickness of above 100 nm. These layers are hard, compact and have good adhesion to glass, relatively high visible transmittance, and low NIR absorption. The thickness of these coatings has a slight effect on the electrical resistance of the layer. The Low-E coatings based on this group include a host network (usually In_2O_3, SnO_2, or ZnO) that is doped with metal or halide atoms. The most common representatives of this group are In_2O_3: Sn (ITO), SnO_2: F(FTO), and ZnO: Ga(GZO), and the other coatings are multi-layers of metal with the 10 nm thickness. These layers are soft, porous, and have little

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adhesion to glass. In this group of coatings, the electrical resistance strongly depends on the thickness of the film. Usually, high-refractive metal oxides such as TiO_2, ZnO, ZnS, SnO_2, Bi_2O_3, and In_2O_3 with suitable metals (Ag, Au, Cu, and Al) are used for this application.

The investigation of the silver and anti-reflex metal oxide as Low-E coatings on transparent substrates has been reported [9]. In this patent, a silver layer with a thickness of less than 30 nm was sputtered. Then, another thin metal layer (less than 10 nm thickness) was deposited on the silver. The presence of this additional layer on the silver coating caused low emissivity of silver and high visible transmittance of the sample remaining in the optimum level.

Optimum thermal emissivity value not exceeding 0.06 was investigated in another study [10]. Coated layer comprised a transparent dielectric substrate with a refractive index of 2.5 - 2.6 (at 550 nm).

In another patent, 25 nm thickness of SnO_2 was deposited between the infrared reflector and 15 nm thickness of zinc oxide layer to develop mechanical properties of the film [11]. Sometimes, other layers were used between reflective and metal layers to protect and reduce oxidation of the metal layer during the heat treatment (e.g., tempering, thermal bending, etc.) [12]. All these studies show that the dielectric layers as the pre-coat have a positive effect on the visible transmittance, while the dielectric layers on the metal layer as an overcoat are added to increase durability.

The effect of the silver layers on the Low-E coatings was examined in another patent [13]. The emissivity was controlled by changing the thickness of the upper silver layer. Although the lower silver layer played a minor role in the film's emissivity, the influence of both layers were great in achieving the desired transmittance, reflectance, and color parameters. In this study, it was revealed that the thickness of the metal layer should be diminished to the minimum possible value to achieve a high visible transmittance.

It should be noted that the optical properties are due to the film's proper thickness. The thicker layer of the metal leads to the increase of the electronic conductivity of the coating and the decrease of its thermal emissivity.

As another related study [14], the ZnS/Ag/ZnS coating emissivity with various silver thicknesses was investigated. In this work, a sudden decrease in the emissivity was observed by a 10-15 nm increase in the silver thickness, and then the emissivity reduction continued at a lower rate. They also added ultra-thin layers, less than 5 nm, of copper or aluminum on the silver to improve the thermal stability of these coatings. The results showed that the visible transmittance did not change significantly by adding an ultra-thin layer of copper, while the visible transmittance reduced sharply by the addition of an aluminum layer.

In this study, we investigate the effect of silver thickness on the optical and electrical properties of the single (ZnO/Ag/ZnO) and double Low-E coatings (ZnO/Ag/ZnO/Ag/ZnO) prepared by the sputtering method at room temperature. We show that the increasing conductivity due to free carriers of Ag layer can effectively cause the decrease of thermal emissivity and the increase of NIR reflectance for single Low-E coatings. But free carriers of Ag layers aren't sufficient to inject free electrons to top ZnO layer for double Low-E coatings. Hence, the increasing NIR reflectance can be suppressed by a sheet resistance increasing.

2. Materials and methods

2.1. Materials

In this paper, the ZnO/Ag/ZnO ultra-thin films were deposited on the glass substrate by 2-targets RF /DC magnetron sputtering equipment at room temperature. In this method, a ZnO ceramic with 99.999% purity, 50 mm diameter and 5 mm thickness was used by RF magnetron sputtering. The Ag target with 99.99% purity, 50 mm diameter and 3 mm thickness as a second target was used by a DC magnetron sputtering.

2.2. Experimental methods

Substrates were cleaned with ethanol and acetone in the ultrasonic bath for 15 minutes and rinsed by distilled water. Afterward, substrates were dried by air flow before the deposition process. The sputtering was performed in Argon (99.9995%) atmosphere within a distance of 75 mm between target and substrate. After placing substrates on the holders, the sputtering chamber was evacuated to a base pressure 2.7×10^{-5} Torr by a turbo molecular pump, and the working pressure was maintained at 1.0×10^{-2} Torr. We adjusted the Argon flow rate 20 sccm. Before the beginning of the sputtering deposition process, the chamber was flashed three times by Argon gas to ensure no oxygen exists in the

sputtering chamber. Besides, pre-sputtering was performed for 5 minutes to discard any impurities on the Ag and ZnO target surfaces. For first samples of ZnO/Ag/ZnO ultra-thin layers, ZnO thin films were deposited with 103 Watt RF power for 300 seconds time set. After the sputtering of the bottom ZnO layer, Ag mid-layer was deposited on the ZnO sub-layer without vacuum breaking of the chamber. It was sputtered by 34 Watt DC power (340 V, 0.1 A). Then, ZnO top layer was sputtered on the Ag ultra-thin films with the same conditions as the ZnO bottom layer without vacuum breaking. In the second stage to produce ZnO/Ag/ZnO/Ag/ZnO ultra-thin layers, another layer of silver was deposited on the ZnO third layer with the same conditions of the second layer. Afterward, another layer of ZnO was sputtered to prevent the oxidation of silver. This stage was designed to prepare double Low-E coatings for comparison with the single Low-E coatings. The above-mentioned process has been selected to achieve appropriate conductive transparent layers for Low-E glazing application.

The optical properties of the samples were measured with Perkin Elmer Lambda 1050-model spectrophotometer. The transmittance and reflectance coefficients were measured for 250 to 2500 nm wavelengths, for UV-Vis-NIR regions. For electrical analyses, sheet resistance and Hall mobility of the films were assessed by Jandel four-probe and the Van der Pauw method measuring, respectively. All the measurements were performed at room temperature. Subsequently, the thermal emissivity value of coatings was computed.

3. Results and discussion

In this paper, we used ZnO (energy gap less than 3.5 eV) as a dielectric, because of the transparent behavior of ZnO in the visible and infrared regions [15] to investigate the effect of silver thickness on the optical properties of ZnO/Ag/ZnO/Ag/ZnO and ZnO/Ag/ZnO coatings in the visible and infrared spectra. In all layers, the ZnO deposition time was set to 300 seconds.

3.1. Optical performance

Figure 1, displays the transmittance and reflectance spectra of a ZnO single layer and the ZnO/Ag/ZnO multilayer films (single Low-E coatings) as a function of Ag deposition time 10, 15, 20, and 30 seconds. The results indicate ZnO thin film transmits over 85% of wavelengths above 330 nm as expected. Then, the scattering of photons increases in the visible region for low Ag mid layer deposition time (10 and 15 s). It is attributed to the presence of agglomerated silver islands in thinner Ag thickness. It leads to a decrease of visible transmittance. The transmittance increases in lower energies (longer wavelengths). Increasing the Ag layer thickness causes silver islands deformation to a uniform and continuous film. On the other hand, visible transmittance remains about 65%, and no noticeable progress is made in the visible range. However, increasing of the free carriers leads to a sharp rise of the NIR reflectance for 20 and 30 s Ag deposition time, and NIR transmittance diminishes significantly. It can be attributed to the increase of conductivity in the thicker Ag thickness.

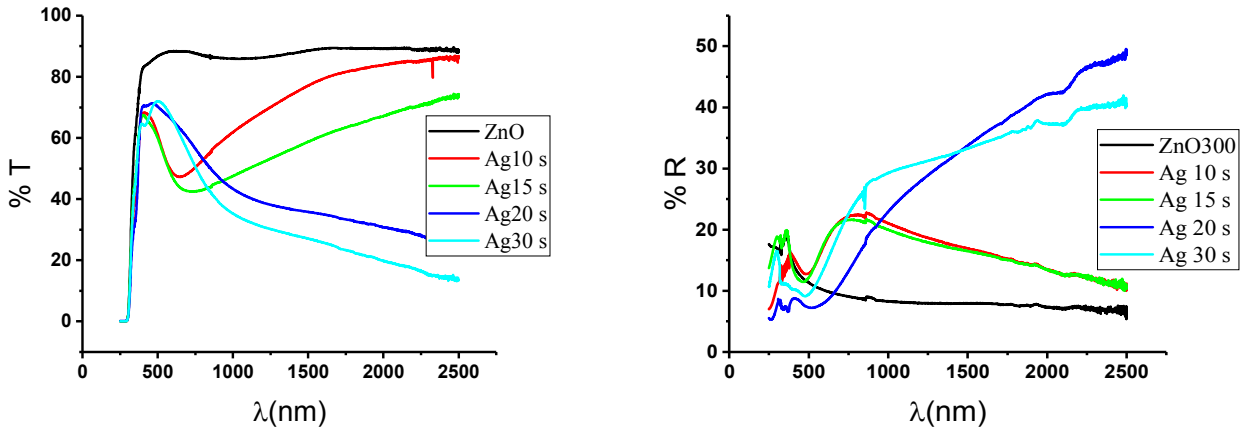

Figure 1: Transmittance (left) and reflectance (right) spectra of ZnO/Ag/ZnO layers as a function of Ag deposition time.

Increasing the Ag mid-layer thickness results in an increase in the NIR reflectance, as shown in the Figure 1. Figure shows the average visible reflectance values change slightly with increasing Ag deposition time. In this study, two schematics for multi layers are considered. Schematic of single and double Low-E coatings has been illustrated in Figure 2.

In the second stage, ZnO/Ag/ZnO/Ag/ZnO layers (double Low-E coatings) were produced with the same deposition time for the Ag layers without vacuum breaking. In these samples, ZnO deposition time was 300 s and the Ag deposition time in each layer was set 10, 15, and 30 s.

In Figure 3, transmittance, reflectance and absorption curves for these layers are exposed. The visible transmittance and reflectance of double Low-E coatings are independent of Ag thickness. However, a decrease of transmittance is observed for thicker Ag layers in the infrared wavelengths. This behavior is attributed to the increase of the Ag layer thickness and the increase of the free carrier concentration; hence, absorption of the photons increases due to more free carriers availability [16]. The conductivity of the coatings increases with increasing the Ag deposition time to 30 s in each layer. In consequence, it can be believed that reflectance increases in the infrared region. FESEM image of ZnO/Ag/ZnO/Ag/ZnO multilayer for 30 s Ag deposition time displays that the average thickness of this sample is about 62 nm.

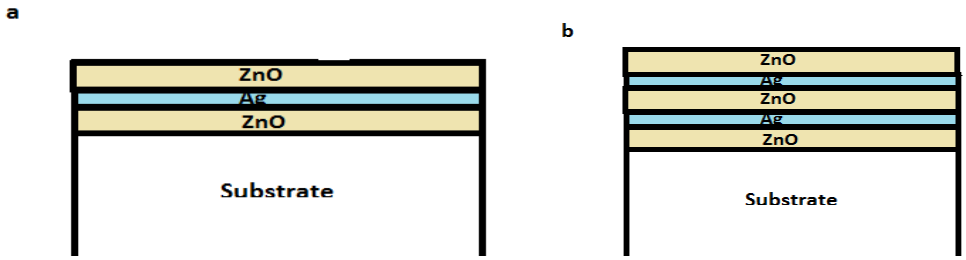

Figure 2: Schematic of (a) single and (b) double Low-E coatings.

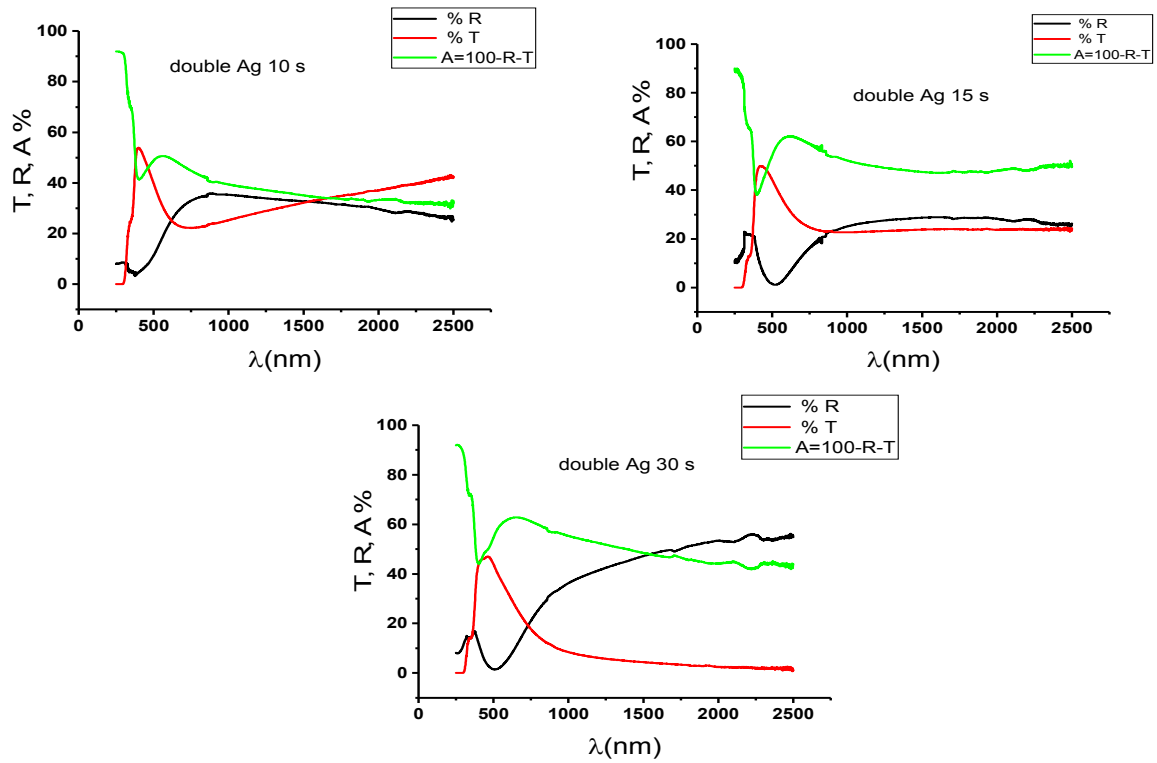

Figure 3: Transmittance, reflectance and absorptance spectra of ZnO/Ag/ZnO/Ag/ZnO multilayers for 10, 15 and 30 s Ag deposition time in each layer.

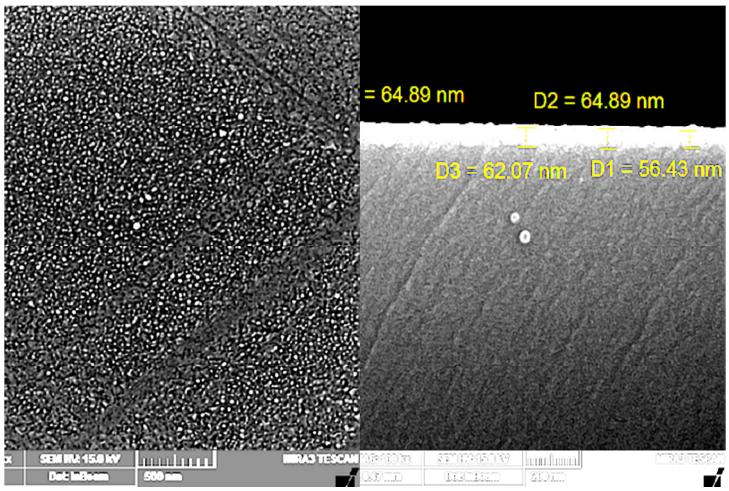

Figure 4: FESEM surface (left) and cross-section (right) images of ZnO/Ag/ZnO/Ag/ZnO multilayer for 30 s Ag deposition time.

The transmittance and reflectance spectra of single and double Low-E coatings can be compared concerning Ag deposition time. It is observed from Figure 5 that the visible transmittance of the double Low-E coatings (red line) is about 20% lower than that of the single Low-E coatings (green line) with same Ag deposition time. However, visible transmittance is almost unchanged for single Low-E coatings (green line) with a deposition time of twice more (black line), and NIR reflectance is twice more in some cases. On the other hand, in the single and double Low-E coatings, transmittance and reflectance spectra have the same course of changes in the visible to NIR spectrum. This result indicates the behavior change of the transmittance and reflectance spectrum depends on the continuity of the Ag layer not just on its thickness. Because of the discontinuous structure of the Ag layer in double Low-E coatings with low Ag deposition time, surface plasmon resonance is considered as one of the main factors of optical absorption of the layer which leads to reduction of the transmittance and reflectance. Also, the surface roughness is decreased with the increasing of the Ag thickness for single Low-E coatings with twice more deposition time that it leads to the reduction of the surface plasmon stimulations, so plasmonic absorption is decreased.

The following table shows, NIR and visible transmittance and reflectance coefficient of the ZnO/Ag/ZnO and ZnO/Ag/ZnO/Ag/ZnO coatings with various Ag deposition times. Transmittance and reflectance coefficient are calculated with the equation 1 [17].

$$T\ or\ R = \frac{\int T\ or\ R\ (\lambda)V(\lambda)d\lambda}{\int V(\lambda)d\lambda} \quad (1)$$

Where V(λ) is the luminous spectral efficiency, and T or R (λ) is the measured transmittance or reflectance of films: T or R $_{visible}$ is calculated for: 380<λ<780 nm and T or R $_{NIR}$ is calculated for: 780<λ<2500 nm.

3.2. Electrical performance

Sheet resistance (R_{sh}) of samples is measured by four-point-probe equipment in compare with the conductivity of the layers. Also, we estimate carrier concentration (n) and mobility (μ) by the Van der Pauw method then thermal emissivity (ε) is calculated using the sheet resistance values.

Thermal emissivity (ε) depends on the sheet resistance (R_{sh}) and can be calculated by using the equation 2 [18]:

$$\varepsilon = 0.0129R_{sh} - 6.7 \times 10^{-5}R_{sh}^2 \quad (2)$$

Table 2 denotes the values of sheet resistance, thermal emissivity, carrier concentration, and mobility values for samples. The sheet resistance declines with increasing Ag deposition time. As the free carriers are raised, the conductivity of multi layers increases. This result is consistent with Drude-theory. The conductivity increases with increasing the free carrier concentration according to the equation 3:

$$\sigma = \frac{n.e^2.\tau}{m} \quad (3)$$

Where σ is the conductivity, n is free concentration, τ is time relaxation, e and m are electron charge and mass. Also, the sheet resistance reduces with increasing of conductivity attention to (Eq. 4).

$$R_{sh} = \frac{1}{\sigma.d} \quad (4)$$

Where R_{sh} is sheet resistance, σ is the conductivity and d is thickness.

Table 1: Visible and NIR transmittance or reflectance coefficients (calculated with Eq. 1) of the ZnO/Ag/ZnO and ZnO/Ag/ZnO/Ag/ZnO coatings with various Ag deposition times.

Layers (deposition time)	T_{vis} (%)	T_{NIR} (%)	R_{vis} (%)	R_{NIR} (%)
ZnO (300 s)/Ag (10 s)/ZnO (300s)	56.9	72.6	16.5	17.4
ZnO (300 s)/Ag (10 s)/ZnO (300s)/Ag (10 s)/ZnO (300s)	35.8	31.1	17.1	32.5
ZnO (300 s)/Ag (20 s)/ZnO (300 s)	66.5	37.5	8.7	31.9
ZnO (300 s)/Ag (15 s)/ZnO (300 s)	54.5	57.1	15.8	16.8
ZnO (300 s)/Ag (15 s)/ZnO (300 s)/Ag (15 s)/ZnO (300s)	38.3	23.4	7.7	26.5
ZnO (300 s)/Ag (30 s)/ZnO (300 s)	64.7	28.3	13.2	32.9
ZnO (300 s)/Ag (30 s)/ZnO (300 s)	64.7	28.3	13.2	32.9
ZnO (300 s)/Ag (30 s)/ZnO (300 s)/Ag (30 s)/ZnO (300 s)	35.8	5.8	8.2	32.8
ZnO (300 s)/Ag (60 s)/ZnO (300 s)	53	0.3	37	89.2

Table 2: measured electrical properties of samples.

Layers (deposition time)	R_{sh} (Ω/sq)	ε	n (cm^{-3})	μ ($cm^2/V.s$)
ZnO (300 s)/Ag (10 s)/ZnO (300 s)	5.61×10^6	-	-	-
ZnO (300 s)/Ag (10 s)/ZnO (300 s)/Ag (10 s)/ZnO (300 s)	1.81×10^6	-	-	-
ZnO (300 s)/Ag (20 s)/ZnO (300 s)	52	0.5	-3.49×10^{21}	2.4
ZnO (300 s)/Ag (15 s)/ZnO (300 s)	5.8×10^6	-	-	-
ZnO (300 s)/Ag (15 s)/ZnO (300 s)/Ag (15 s)/ZnO (300 s)	3.4×10^3	-	-	-
ZnO (300 s)/Ag (30 s)/ZnO (300 s)	18.5	0.2	-3.6×10^{21}	6.3
ZnO (300 s)/Ag (30 s)/ZnO (300 s)	18.5	0.2	-3.6×10^{21}	6.3
ZnO (300 s)/Ag (30 s)/ZnO (300 s)/Ag (30 s)/ZnO (300 s)	6.3	0.08	-7.5×10^{21}	12.2
ZnO (300 s)/Ag (60 s)/ZnO (300 s)	3.0	0.04	-7.8×10^{21}	17.1

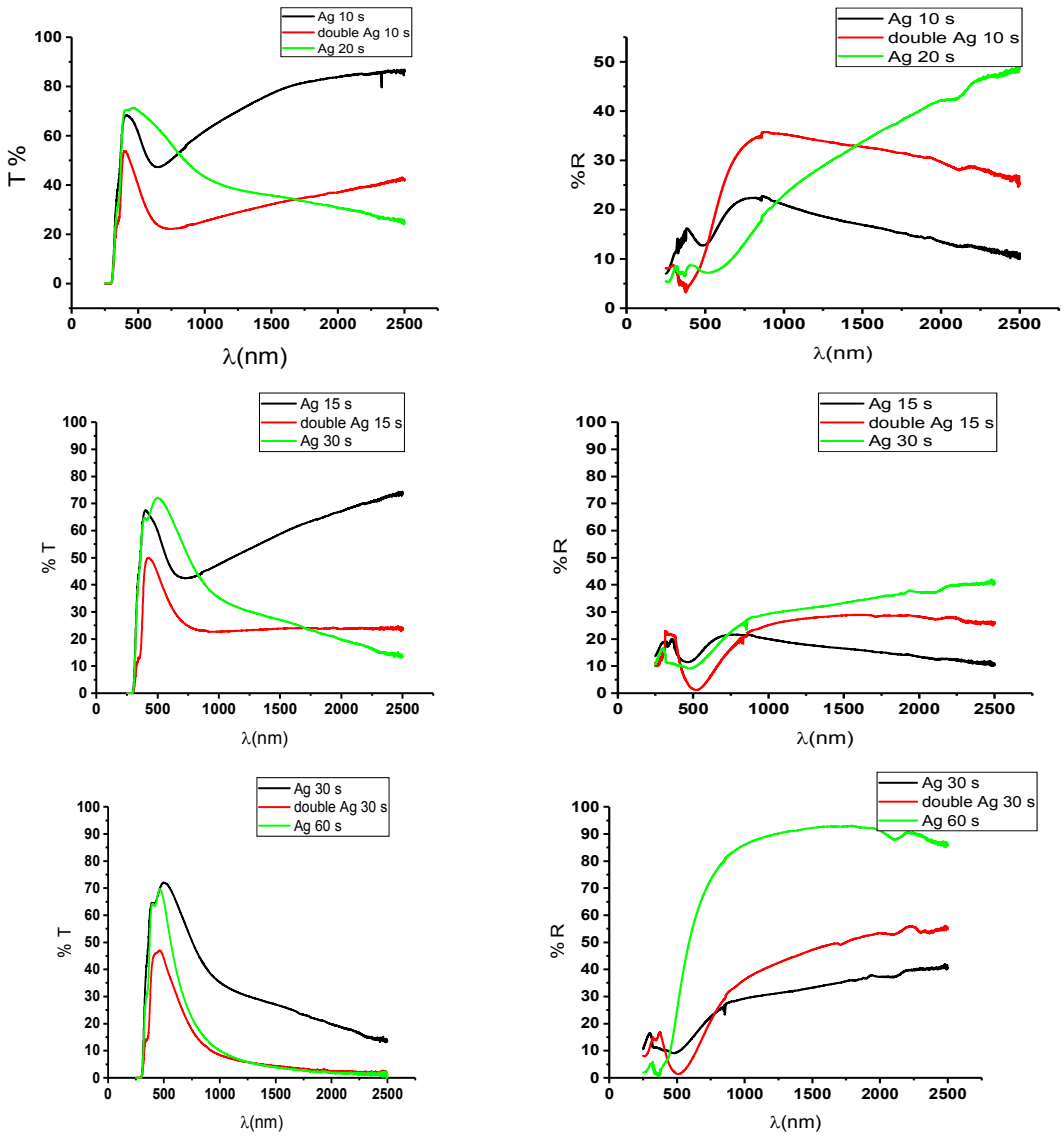

Figure 5: Transmittance and reflectance spectra of single and double Low-E coatings for 10, 15 and 30 s Ag deposition time.

The sheet resistance of the coatings is very high in low Ag deposition time, 10 and 15 s, for both single and double Low-E specimens, as shown in Table 2. Based on these data, it is impossible to measure other electrical parameters. It can be attributed to the amorphous states for the ultra-thin thickness that the increase of the grains boundary acts as a carrier trap and the electrical resistance increases. Also, the mobility reduces due to the poor crystallinity of these layers [19]. The reduction of mobility is related to many of defects and the increase of scattering.

However, crystallinity of the coatings improves with increasing the Ag thickness so the scattering reduces. It leads to advancing the free carrier concentration and thereby decreasing sheet resistance of the coatings. The sheet resistance of the single Low-E coatings is lower than double Low-E coatings in the same thickness of Ag for single Low-E (ZnO/Ag/ZnO) and total Ag thickness of double Low-E (ZnO/Ag/ZnO/Ag/ZnO) coatings. It can be attributed to the continuous thickness and adequate crystallinity for thicker Ag layer [20-22]. Besides, the decrement of sheet resistance makes lower thermal emissivity of the coatings according to the equation 2. In agreement with the optical performance, lower thermal emissivity can strongly increase NIR reflectance as shown in Figure 6.

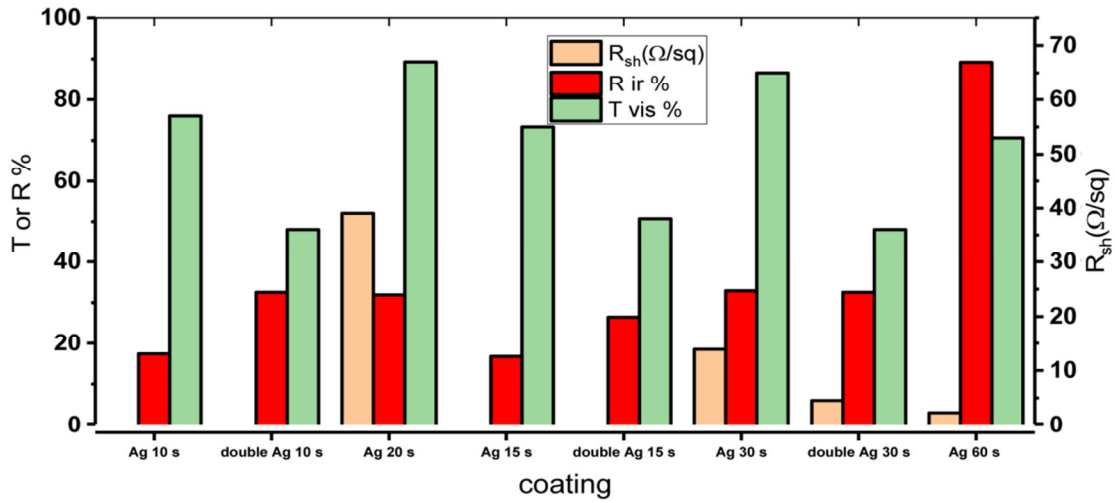

Figure 6: Comparison of optical and electrical properties of single and double Ag coatings.

4. Conclusion

In this paper, optical and electrical properties of single or double Low-E coatings were investigated as function of Ag deposition time. When the Ag deposition time is increased to 60 s, for single Low-E coatings, thermal emissivity and R_{NIR} reached 0.04 and 89%, respectively. Whereas thermal emissivity and NIR reflectance obtained 0.08 and 32%, for a double Low-E (ZnO/Ag/ZnO/Ag/ZnO) coating with 30 s Ag deposition time in the second and the fourth layers. This result can be attributed to the high absorption of surface plasmons in double Low-E coatings. Hence, it should be noted that the choice of the suitable thickness of each layer must be carefully performed to avoid any unwanted optical interference, and to lose optical characteristics for specific applications. On the other hand, intermediate layers can be used to improve the requested properties of the coatings. It seems that more desirable properties can be acquired by changing ZnO thickness in double Low-E coatings.

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