Effects of silver incorporation on electrical and optical properties of $CuAl_xO_y$ thin films

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ABSTRACT

The transparent conductive property based on Ag-doped delafossite nanomaterials is attractive for optical sensing applications due to their good electrical conductivity, good optical transparency and high temperature coefficient of resistance. Several delafossite nanomaterials and Ag-doped nanomaterials have been reported, however, Ag-doped delafossite nanomaterials have not been explored, especially regarding the electrical property with high temperature coefficient of resistance. In this study, Ag-doped delafossite $CuAl_xO_y$ thin films were deposited by co-sputtering techniques. The electrical properties were carried out on a 4-point prober. The optical properties were characterized on an UV-VIS spectrometer. The results on $CuAl_xO_y$ doped Ag thin films showed that $CuAl_xO_y$ doped Ag could hardly be applied for transparent conductive layers. However, these films exhibited a relatively high temperature coefficient of resistance of about 3%/K, thus being suitable for applications in microbolometers.

Keywords: Transparent conductive; Delafossite; $CuAl_xO_y$ thin film; Co-sputtering techniques; Temperature coefficient of resistance; Microbolometers.

1. INTRODUCTION

Many delafossites ($A^+B^{3+}O_2$) were found in physical properties. They were reported for the first time as part of large research about structure, synthesis, and electrical transport properties by Rogers, Shannon, and Prewitt [1-3]. After a short time, the optical and electrical properties of certain copper delafossites in many papers were reported by Benko and Koffyberg [4, 5]. In 1997, the attention to delafossites decreased when a seminal paper by Kawazoe and co-workers found that a 500 nm thick film of $CuAlO_2$ transmitted about 70% in visible light and exhibited *p-type* conductivity of 0.95 $S.cm^{-1}$ [6]. Since then, delafossites, especially the Ag and Cu elements, have received considerable attention owing to their application as transparent conducting oxides [7, 8]. Particular emphasis was placed on optimizing their electrical and optical properties through proper selection of the parent delafossites, aliovalent dopant and control of the oxygen stoichiometry. Because of their compositional versatility, delafossites also were studied deeply as catalysts [2, 4, 5], luminescent materials [6, 7], and thermoelectrics [9-11].

Various methods for preparing delafossite oxides have been investigated, such as high-temperature solid-state reactions, cation exchange reactions, hydrothermal reactions for the powders synthesis, and sputtering, sol−gel, pulsed laser deposition (PLD) for the preparation of thin films. Since the target material for thin film deposition is composed of particles, the synthesis of phase-pure powder is the required initial step. Some general rules leading to the formation of ABO_2 oxides could be found in diverse synthesis methods. For example, copper-based delafossite oxides ($CuAlO_2$, $CuCrO_2$, $CuFeO_2$ and $CuScO_2$) could be synthesized readily via high temperature solid-state reactions under an inert atmosphere (N_2 or Ar) at ~ 800 - 1200 °C, since Cu^+ is even more stable than Cu^{2+} at high temperatures. However, for the low temperature hydrothermal synthesis of $CuAlO_2$, $CuCrO_2$, and $CuGaO_2$, the raised difficulty rests with how to reduce the soluble Cu^{2+} precursor to Cu^+ and maintain the valence of Cu^+ in the

monovalent state in a wet chemical environment. For the synthesis of silver-based delafossite oxides, solid-state reactions at high temperature generally encountered practical problems because of the easy decomposition of Ag_2O to elemental silver at a temperature of ∼300 °C. Therefore, most reported silver-based delafossite oxides, such as $AgInO_2$, $AgCrO_2$, $AgAlO_2$, and $AgGaO_2$, were synthesized via low-temperature hydrothermal methods in closed reaction systems. Moreover, among the various delafossite oxides, $CuAlO_2$ and $AgAlO_2$ are more difficult to synthesize because of the higher crystal formation energy barrier, which is associated with cleavage and reorganization of the high-energy Al−O bonds. Conversely, these two aluminum-based delafossite oxides are superior in chemical and thermal stability than other delafossite oxides; besides, high optical transparency and a low-cost aluminum source are two other important advantages of these two materials, which are highly desired in many practical applications. Although the synthesis of $CuAlO_2$ nanocrystals at 400 °C via supercritical hydrothermal methods has been reported since 2004, few reports have followed up on such a procedure, which might be hard to reproduce. Besides, until now, there have been few systematical studies focusing on the hydrothermal synthesis mechanism of aluminum-based delafossite oxides [12].

2. PROBLEM

2.1. Theoretical foundations

Electronic structure calculations and analysis of the Kubelka–Munk absorption data reveal that similar to copper delafossites, silver delafossites have a disparity in energy between the "forbidden" fundamental direct and indirect band gaps and optically measured band gap. While their optically measured band gaps widen with an increase in the radius of the B-site cation, the decreased fundamental band gaps for larger B-site cations result in some absorption of photons in the visible light range for $AgGaO_2$ and $AgInO_2$, which reduces their optical transparency and thereby colors these delafossites. When corresponding copper and silver delafossites with the same B-site cation are compared, however, silver delafossites have larger band gaps and lower visible light absorption, owing to a shift in the valence band states to lower energy upon replacement of copper 3d states with silver 4d states. Thus, the electronic structure prediction is general (i.e., any silver delafossite has a larger band gap than the corresponding copper delafossite). Moreover, while silver delafossites have conductivities lower than those of polycrystalline powders of copper delafossites, this study provides a starting point for the difficult process of improving the conductivity of delafossites through extrinsic doping without significantly compromising their optical properties [13].

Another important electrical property of these materials is temperature coefficient of resistance (TCR). The most important property of microbolometers is its infrared sensitive layer TCR. Vanadium oxide is the traditional materials for the sensing layer of microbolometers due to the high value of TCR which is in the range of 2%/K to 3%/K [14]. However, single crystal vanadium oxide, which has higher TCR value of around 4%/K, is rather difficult to achieve [15]. In this study, ternary oxide minerals ($M^IM^{III}O_2$, also known as delafossite oxide materials), or in particular, $CuAlO_2$ and $AgAlO_2$, are chosen to be studied as an infrared sensitive material. This type of materials has shown some interesting properties such as thermoelectric effect or being used as transparent conducting oxide. However, not much research has been done on the TCR of this type of material.

Copper aluminate ($CuAlO_2$), which is stable at high temperatures up to 1,400 K and possessing good thermoelectric power, is expected to be another promising material for thermoelectric devices. This type of material has also gained much attention in the field of optoelectronic applications due to the fact that $CuAlO_2$ has a direct band gap of 3.5 eV and is a transparent

conductor. $CuAlO_2$ crystallizes in the rhombohedra delafossite p-type structure (a = 2.85670 Å, c = 16.9430 Å) and shows p-type semiconductivity [16]. Park et al. have investigated the thermoelectric properties of $CuAl_{1-x}Ca_xO_2$ ($0 \leq x \leq 0.2$) and found that the substitution of Ca for Al up to x = 0.1 increases both the electrical conductivity and the Seebeck coefficient [17]. Lately, the effects of Mg or Fe substitution for Al in $CuAlO_2$ were also reported. Among these studied elements, the highest value of power factor (1.1 × 10-4 W/mK) was obtained for the $CuAl_{0.9}Fe_{0.1}O_2$ sample at 1,140 K [18, 19]. Moreover, the calculation of the electronic structure of Ni or Zn doped $CuAlO_2$ using a full potential linear augmented plane-wave method, reported by Lalic et al., showed that Ni and Zn substituted for Cu-sites act as acceptor and donor impurities, respectively [20]. As for delafossite p-type materials, the effect of Ag substitution for Cu-sites in $CuRhO_2$ has been investigated [21]. However, to our knowledge, the effect of element substitution for Cu-sites in $CuAl_xO_y$ has not been reported to date.

This study focuses on the substitution of Ag to Cu-sites in $CuAl_xO_y$ and systematically investigates their effects on the high temperature thermoelectric properties of these compounds. Ag-doped delafossite $CuAl_xO_y$ thin films were deposited by co-sputtering techniques. The electrical properties were carried out on a 4-point prober. The optical properties were characterized on a UV-VIS spectrometer. The results on $CuAl_xO_y$ doped Ag thin films showed that $CuAl_xO_y$ doped Ag could hardly be applied for transparent conductive layers, however, it is potential for applications in microbolometers.

2.2. Experiment preparation

2.2.1. Instrumentation

In this study, Ag-doped $CuAl_xO_y$ thin films were deposited on corning glass substrates by using co-sputtering techniques. The corning glass substrates were cleaned by acetone and isopropanol solution in ultrasonic vibration then were soaked in piranha solution and discharged with deionized water and nitrogen gun. All chemicals used are of high quality and purity from Xilong Chemical Co., Ltd., China. Commercial 2-inch of diameter Cu, Ag and Al_2O_3 targets which have a purity of about 99.95% from Changsha Xinkang Advanced Materials Co., Ltd., China, were used. Two series of $CuAl_xO_y$ thin films were fabricated by co-sputtering with: (i) the Cu and the Al_2O_3 targets, (ii) the Cu, the Ag and the Al_2O_3 targets. All of the samples were deposited on a Syskey SP-01 2-DC and 2-radio frequency (RF) magnetron gun system. The Al_2O_3 target was loaded on the RF_1 gun, the Cu target was loaded on the DC_2 gun and the Ag target was loaded on the RF_2 gun. The distance between guns and substrates was fixed at 9cm. The oil pump and the turbo pump were used to reduce the pressure in the chamber to below $5x10^{-6}$ Torr. Argon (Ar) air and Oxygen (O_2) were used together for sputtering: Ar was set = 16 sccm, O_2 was set = 4 sccm. Pressure in the chamber was set = $7.5x10^{-3}$ Torr. The thicknesses of the films were characterized on an alpha-step model NanoMap-500LS. The optical properties of all the thin films were characterized by an ultraviolet-visible (UV-VIS) spectrophotometer, Shimadzu UV-2450. The sheet resistance of thin films was measured on a four-point prober, Jandel RM3000. The measurement and the control temperature system, which are Self-designed by us, are combined with the 4-point probe Jandel RM3000 and the emitter current source Keithley 2400.

2.2.2. Experimental materials

From the list of $CuAl_xO_y$ samples shown in table 1 below, these samples were co-sputtered by aluminum oxide target, copper target and silver target. However, silver sputtered in some seconds, while copper and aluminum oxide sputtered in 1 hour. We kept the sputtering

temperature at 200 °C, sputtering power of Al_2O_3 at 40 W, sputtering power of Cu varying from 20 W to 80 W and the sputtering power of Ag at 20 W.

Table 1. *The list of $CuAl_xO_y$ samples.*

Number of samples	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Sputtering power of Cu (W)	20	40	60	80	60					80				
Ag-20W sputtering time (s)	0				5	10	15	30	60	5	10	15	30	60

3. RESULTS AND DISCUSSION

3.1. Thickness of thin films

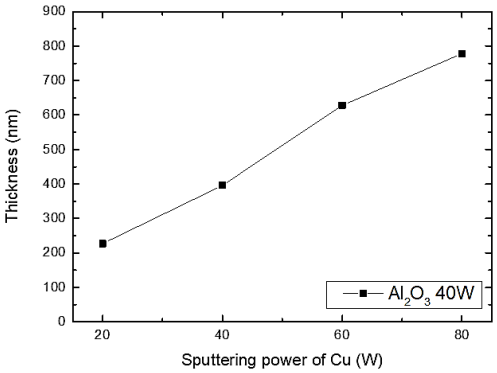

Figure 1a. *The thickness of $CuAl_xO_y$ thin films depended on the sputtering power of Cu.*

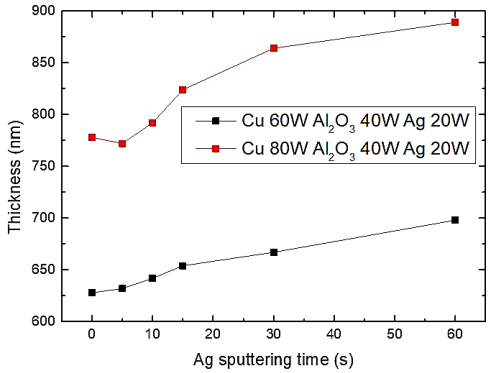

Figure 1b. *The thickness of $CuAl_xO_y$ thin films depended on Ag sputtering time.*

Fig. 1a shows clearly that the thickness of $CuAl_xO_y$ sample increased as the sputtering power of copper increased. The thickness rose quite linearly from 227 nm to 778 nm, corresponding to the sputtering power of copper increased from 20 W to 80 W.

Fig. 1b illustrates that the thickness of $CuAl_xO_y$ samples depended on the silver sputtering time. It had significant errors, however, the trend of the thickness of samples increased as silver sputtering time increased.

The thickness of all samples was suitable for studying the optical and electrical properties of nano-thin films but is not the main criterion for selecting the sample fabrication conditions.

3.2. Optical properties

From Fig. 2a, we can easily see that the transmission of $CuAl_xO_y$ thin films decreased as the sputtering power of copper increased. Especially the transmission between Cu-20W sample and Cu-40W sample was very different; however, the transmission between Cu-40W and Cu-80W sample was not much different. Besides, the transmission of Cu-20W sample at 550 nm wavelength was higher than 10%, however, the transmissions of others were nearly down to 0%.

Fig. 2b illustrates the transmission of series Cu-60W-Al_2O_3-40W-Ag-20W with the silver sputtering time increased steadily. When the silver was doped in 5 s the transmission of the sample increased significantly. Up to 10 s, the transmission was higher than 5 s sample at longer than 800 nm wavelength but lower than 5 s sample in the visible range. Up to 15 s, 30 s and 60 s, the transmissions of these samples were continuous reduction in all wavelengths from 300 nm to 900 nm.

From Fig. 2c, the transmission on a series of Cu-80W-Al_2O_3-40W-Ag-20W with the change of silver sputtering time was examined. There were 2 parts of wavelength to be evaluated here. In visible range, the transmission of 0s-Ag sample was the lowest and 5s-Ag sample was the highest then decreased steadily by silver sputtering time. In the contrary, at the longer wavelength, especially higher than 850 nm, the transmissions of 5s-Ag and 10s-Ag samples were lowest and the transmission of 60s-Ag sample was highest.

The average transmittance of all $CuAl_xO_y$ samples in visible range and easily found that 2 important features. The transmission decreased strongly as the sputtering power of copper increased. Besides, the decrease in transparency also depended on the increase in silver sputtering time.

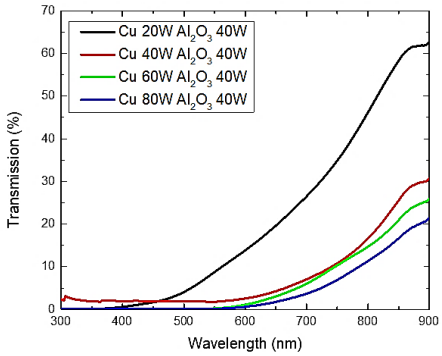

Figure 2a. *Effect of sputtering power of copper to the transparency of $CuAl_xO_y$ thin films.*

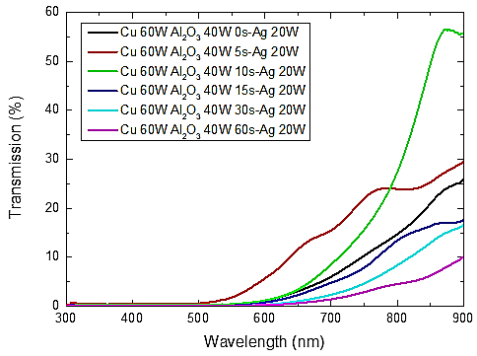

Figure 2b. *Effect of silver sputtering time to the transparency of $CuAl_xO_y$-Cu 60W thin films.*

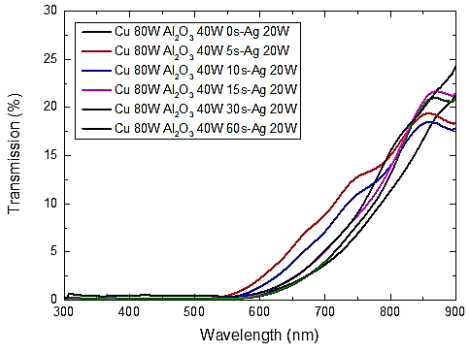

Figure 2c. *Effect of silver sputtering time to the transparency of $CuAl_xO_y$-Cu 80W thin films.*

3.3. Electrical properties

Fig. 3a shows the resistance depended on the temperature of 4 series of $CuAl_xO_y$ thin films. On this figure, we could confirm that these samples had negative thermal coefficient of resistance as a semiconductor. Besides, the resistance of Cu-60W sample was highest and the resistance of Cu-20W sample was lowest.

Fig. 3b gives us the results of resistance depending on temperature by increasing the silver sputtering time of a series of Cu-60W samples. From this figure, we could find the highest resistance on the 15s Ag sample and the lowest resistance on the 30s Ag sample.

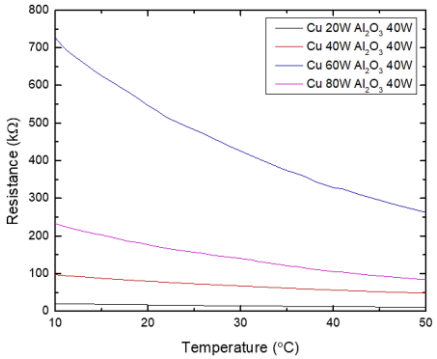

Figure 3a. *The resistance depended on temperature of $CuAl_xO_y$ thin films.*

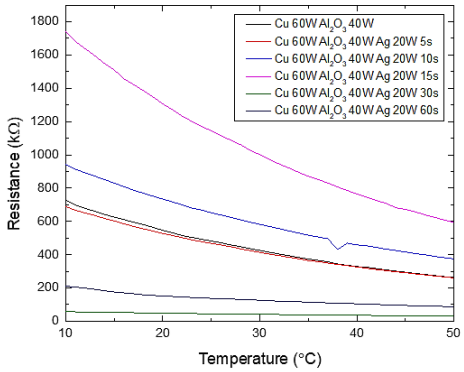

Figure 3b. *The resistance depended on temperature of Ag doped $CuAl_xO_y$-Cu 60W thin films.*

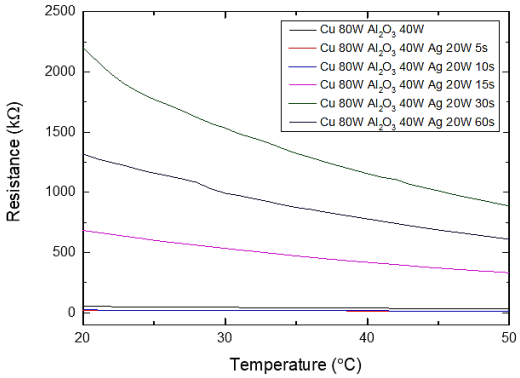

Figure 3c. *The resistance depended on temperature of Ag doped $CuAl_xO_y$-Cu 80W thin films.*

Fig. 3c shows the resistance depends on temperature of series of silver doped $CuAl_xO_y$-Cu 80 W thin films. There is quite clear that the resistor increased strongly when the silver sputtering time increased more than 15 s. The highest resistance was observed on 30s Ag sample.

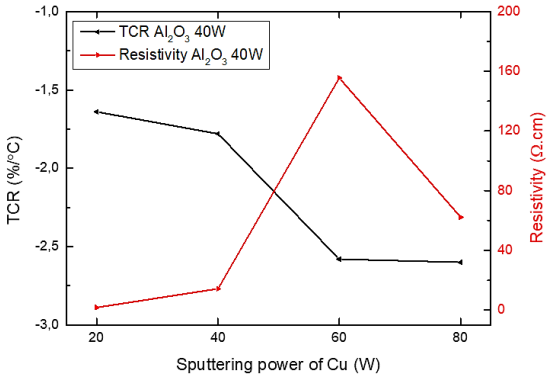

Figure 4a. *The TCR and the conductivity of $CuAl_xO_y$ thin films depended on sputtering power of copper.*

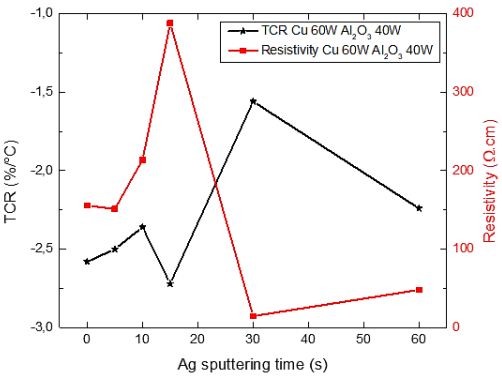

Figure 4b. *The TCR and the conductivity of $CuAl_xO_y$-Cu 60W thin films depended on silver sputtering time.*

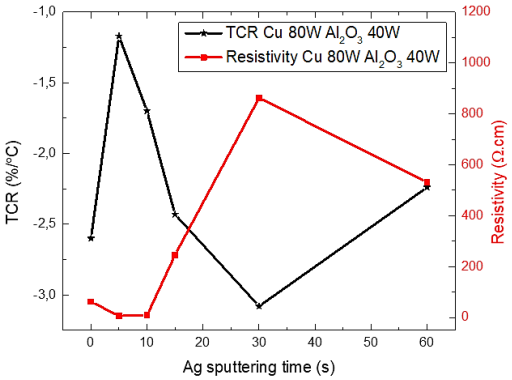

Figure 4c. *The TCR and the conductivity of $CuAl_xO_y$-Cu 80W thin films depended on silver sputtering time.*

The most important of our concerns on $CuAl_xO_y$ thin films were TCR and resistivity. We would like to get the sample which had the highest TCR but its resistivity was not too high as

good conductivity. From the Fig. 4a, we can easily to find out that the TCR of Cu-60W sample and the TCR of Cu-80W sample were highest at more than 2.5%. These results were as good as the best TCR of Vanadium pentoxide material, which was the most popular material, be used for microbolometers, which was 2.57% [22]. Therefore, in the next step, we would like to study the effect of silver on these two samples.

Fig. 4b indicates the TCR and the resistivity of series of Cu-60W samples. There was a little contradiction here. The samples, which had high TCR, also had high resistivity. In the contrary, the samples, which was good conductivity as had low resistivity, also had lower TCR. However, we could confirm that doping silver into $CuAl_xO_y$ thin films was suitable for increasing TCR or reducing resistivity of them.

Fig. 4c also illustrates the contrast between TCR and the resistivity of $CuAl_xO_y$-Cu 80W thin films. However, the biggest success, which we found here, was the highest TCR of 30s Ag sample as more than 3%. There was clearly better than $CuAl_xO_y$ sample without silver. On the other hand, doping Ag by sputtering in 10s gave a little lower TCR, however, increasing conductivity significantly as reducing resistivity of sample. The highest TCR, which was obtained, is not the highest TCR has been found in the world. There were higher TCR materials has been found in Metal-oxide manganite (4.4%/°K), $Ge_xSi_{1-x}O_y$ (5.1%/°K) [23], or silicon thin films (6%/°K) [24, 25], etc. However, these materials have some huge disadvantages that very high large 1/f noise or manufacturing conditions are complex and expensive. Therefore, $CuAl_xO_y$ and $CuAl_xO_y$ doping Ag are really valuable materials that can be applied well in microbolometers.

In order to explain why we can get higher TCR materials by doping silver, we assume that silver with a suitable amount of doping in materials can help to narrow the energy band gap to increase conductivity. Materials, which have high TCR, need to have a high band gap but not to have too high band gap. Ideal materials, which have high TCR, need both abilities to conduct and non-conduct and temperature can help to change that property. Therefore, because $CuAl_xO_y$ has high band gap (~3.48 eV), we need a small amount of silver to dope in order to improve the conductivity at high temperatures. Besides, at low temperatures, with a small amount of silver, the non-conductive property still remains high and is not affected. However, if the amount of silver doping was too high, the high conductive could also be seen at low temperatures and the TCR value could not be high.

4. CONCLUSIONS

In this study, a co-sputtered process for fabricating transparent conductive oxides $CuAl_xO_y$ thin films was developed. When doping 30s Ag in to the $CuAl_xO_y$ sample corresponding to the sputtering power of Cu target of 80 W, power of Al_2O_3 target of 40 W, power of Ag of 20 W, the thermal coefficient of resistance of thin films was higher than 3%. The higher the thermal coefficient of resistance, the better the sensitivity of the infrared sensor layer. Therefore, we could confirm that silver incorporation on $CuAl_xO_y$ thin films may be used for applications as an infrared sensing layer in microbolometers.

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TÓM TẮT

Ảnh hưởng của bạc pha tạp tới các tính chất quang và điện của các màng mỏng $CuAl_xO_y$

Tính chất dẫn điện truyền qua dựa trên vật liệu nano delafossite pha tạp Ag rất quan trọng đối với các ứng dụng cảm biến quang học do tính chất dẫn điện tốt, độ truyền qua quang học tốt và hệ số nhiệt điện trở cao. Một số vật liệu nano delafossite và vật liệu nano pha tạp Ag đã được báo cáo, tuy nhiên, vật liệu nano delafossite pha tạp Ag vẫn chưa được khám phá, đặc biệt là về tính chất điện với hệ số nhiệt điện trở cao. Trong nghiên cứu này, màng mỏng delafossite $CuAl_xO_y$ pha tạp Ag được lắng đọng bằng phương pháp đồng phún xạ. Các tính chất điện được đo bằng máy đo điện trở 4 điểm. Các tính chất quang học được đo trên máy quang phổ UV-VIS. Kết quả nghiên cứu cho thấy $CuAl_xO_y$ pha tạp Ag khó có thể ứng dụng cho các lớp dẫn điện trong suốt. Tuy nhiên, các màng mỏng này cho thấy hệ số nhiệt điện trở tốt với giá trị tương đối cao, khoảng 3%/ºK, do đó vật liệu thích hợp cho các ứng dụng cho microbolometers.

Từ khoá: Dẫn điện truyền qua; Delafossite; Màng mỏng $CuAl_xO_y$; Phương pháp đồng phún xạ; Hệ số nhiệt điện trở; Microbolometers.