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Electrodepositing of Multi-Layer Ni-Ag Coated by Copper Nanoparticles for Solar Absorber

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Abstract

In this work, the effect of the addition of bright nickel plating and silver carried out by the electroplating method has been studied, on the coating of copper nanoparticles on the copper base metal via the process of thermal evaporation. The improvement of the solar absorber using CuNP in combination with the bright nickel and silver was obtained to be better than copper nanoparticles individually. A bright nickel enhanced the absorbed thermal stability. Also, other optical properties, absorptions, and emissivity slightly decreased from (93% to 87%), while the existence of silver had a slight impact on absorption of about (86.50%). On the other hand, thermal conductivity was evaluated using hot disk analyzer. The results showed a good improvement obtained by copper nanoparticles of about (89%) deposited on the copper substrate, while it decreased with 18.8% in the presence of bright nickel combined with copper nanoparticles. The other characteristics, like structure and phases of coating layers, were achieved using X-ray diffraction. Topographic maps were obtained using an Atomic Force Microscope and Scanning Electron Microscope.

Keywords: *Bright Nickel, Copper nanoparticles CuNP, PVD, Silver, Solar absorber.*

1. Introduction

Due to the increasing demand for energy, the need for alternative clean energy sources with the least emission of dioxide has increase; sun is one of the most important sources of energy at all, in order to convert this thermal energy into useful energy, selective coatings are emerging as an important industrial application [1, 2]. The proper coating of the selective solar absorber must possess two criteria; a high absorption across the wavelength of the solar spectrum in the ultraviolet area visible (UV-Vis), and a low thermal emissivity close to the infrared (NIR). In general, the optical properties are obtained by reflectance spectrophotometry, and the reflectance must be less than (10%) in the (UV-Visible) range and more than (90%) in the (NIR) [3]. Because metals possess almost low thermal emissivity, coatings of the selective solar absorber are, in general, produced on the metallic substrates with high thermal conductivity and a good corrosion resistance [4, 5]. There exist several processes and methods to produce the selective coatings, including paint, chemical vapor deposition (CVD), Sol-Gel, Spray coating, and physical vapor deposition (PVD), Electroplating is simple and an allured choice owing to its reproducibility, adequate governing over the thickness and morphology of the coating, short deposition times and less cost [3,4,6]. Bright nickel is an allured, less-cost solar absorber metal, the coatings produced with the electrolyte and same formulations for the chemical converting baths exhibited good optical characteristics [7-9]. Nickel is usually used as an intermediate layer to protect the thin film and prevent the diffusional problem of the nanoparticle to the material substrate also giving thermal stability [10, 11]. Silver electroplating was used due to a good appearance, the highest electrical and thermal conductivity was the main remarkable as well as the possibility of depositing over the hollow, and table parts, in addition to corrosion resistance. Industrial and engineering use of silver plating due to high thermal and optical properties resulting that popped up. It used, also in the mirrors, electronic and printed circuits [12, 13]. The field of solar selective coating attracted many researcher's interests. Copper is an allured substrate metal for the selective coatings due to its more reflectance (less emissivity) close to NIR, stability and good thermal conductivity. Furthermore, copper is too convenient as a substrate for the electrodeposition. From the other side, copper nanoparticles are completely different from the bulk, where they characterized by their unique and distinctive properties in the field of thermal and electrical conductivity as well as their use in selective coating [14]. Recently, researches have focused on using nanotechnology in the field of solar energy coatings in order to improve the efficiency of the absorber surface, J. El Nady et al. (2016) [15], increased the efficiency of the solar absorber using black paint coated on copper once and comparing that with using black paint combined with bright nickel once again. Nanoparticles layer consist of bright nickel base was deposited on copper substrates using electrodeposition technique, before spraying the paint. M.A. Estrella - Gutiérrez et al. (2016) [16], studied the effect of nickel interlayer between the copper base metal and black nickel as absorber surface as compared with black nickel directly coated on copper. The aim of this study is to implement an efficient solar selective coating based on nano-thin film combined with multilayers (Ni and Ag), to be employed in thermal energy application, whereas the electroplating of bright nickel is working as an interlayer between the copper substrate and copper nanoparticles (CuNP) selective absorber. In addition, the effect of this layer on the optical and thermal properties has been reported; also, silver electroplating has been carried out as a second layer on the nickel layer. This nickel layer coating has slightly lower thermal absorption and emissivity than copper nanoparticles coated onto copper directly. Coatings based on bright nickel and silver deposited on copper substrates using a watts bath performed has evolved the optical and physical properties as compared with the nanocoatings directly deposited onto the substrates of copper, or on the copper covered with an electroplating bright nickel.

2. Experimental Part

2.1 Substrate Preparation

Copper substrates cut like rectangular with dimensional (20 mm × 200 mm × 3 mm) using band saw machine type Knuth German manufacturing, and in order to hang the samples in an electroplating bath, it should have a drill about (5mm). Fig. (1) shows the geometry of the samples.

Fig. 1. Copper substrate alloys Pre-sample cutting.

Later Copper substrates post cut carried out with dimensional (20 mm × 20 mm × 3 mm), as present in Fig. (2). In order to use the samples in a vacuum chamber of the evaporation system, wire cutter machine type Knuth was used to make the samples more suitable.

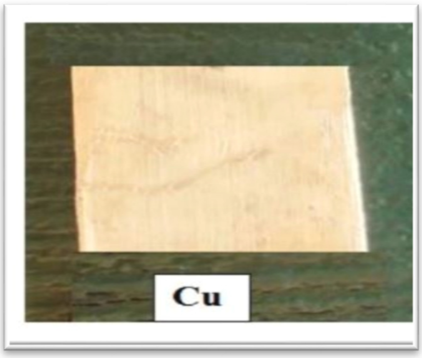


Fig. 2. Copper substrate alloys post-sample cutting.

2.2 Chemical analysis of the Substrate Alloys

The chemical composition of the copper substrate alloy has been analyzed with a spectrophotometer at the condition of temperature with 20˚C, and the humidity of 62% the chemical analysis was carried out using by using optical emission spectrometer (OES) type (Foundry-Master x pert) S.N 52Q0089 German manufacturing. The results of the analysis have been illustrated in the tables (1), which include the actual measured and standers values, while Fig. (3) shows the microstructure of copper under an optical microscope.

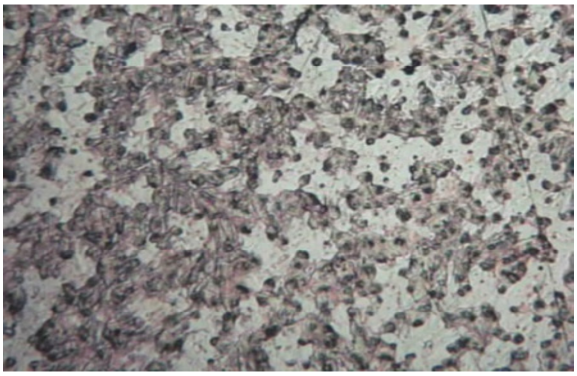


Fig. 3. Microscopic structure of copper substrate.

Table 1,
The actual chemical compositions of copper.

Chemical Composition%	Standers Values	Actual Values
Cu	Min.99.3	99.330
Fe	0.10	0.142
Si	0.015 - 0.040	0.496
P	-	0.000
Pb	0.001 - 0.005	0.001
Ag	Min. 0.027	0.019
Mn	0.005 - 0.012	0.007

On the other hand, copper Nanoparticles used as raw nanomaterial absorber was present with a mean particles size of 24 nm. Table 2 gives the general specifications of CuNP and the physical properties of copper powder; it was fabricated by Nanjing Nanotechnology Co. Ltd.

Table 2,
Specifications of copper nanoparticle.

Property	The value	Units
Average particle diameter	24	nm
Purity	99.98	%
Bulk Density	0.46	g/cm3
True Density	4.23	kg/m3
Color	Black	-

2.3 Preparation for Electroplating Process

Before employing the copper substrates pieces into the chemical bath of electroplating system, the Substrates were prepared for coating. The impurities, contaminants, dust, grease and other stranger particle remain from manufacturing operations must have been removed. It may sometimes require removed layer in order to achieve free surface like remove the oxides this will lead surface ready for the electroplating process. In this work, different techniques carried out to prepare the copper substrate for nickel electroplating to have flowed with silver electroplating electropolishing, ultrasonic cleaning, alkaline cleaning, acid clearing, cleaned and polishing as illustrative in Fig. (4).

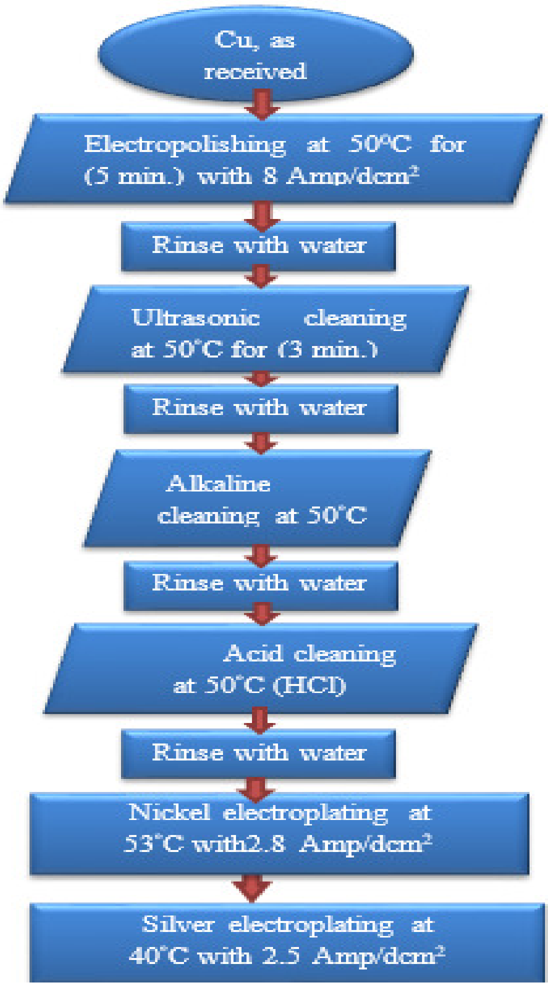


Fig. 4. Scheme represented the surface methods achieved in this study.

3. Bright Nickel Electroplating

Bright nickel deposited on copper substrates as interlayer carried out by electroplating technique. The bright nickel layer was achieve using the plating cell rectangular shape with size (50 liters) containing ($NiSO_4.6H_2O$ 240 g/L, $NiCl_2.6H_2O$ 20 g/L, H_3BO_3 20 g/L and with different concentrations of KNO_3) as electrolyte solution sometimes called Watts bath. While the plating bath was made from Polyphenols Chloride (PVC), Nickel sheets with dimensions (25× 25×3) cm were used as the anode, while copper pieces with dimensions (20 × 2× 0.3) cm used as the cathode. Before each run, the nickel plating sample preparation carried out. A D.C power supply unit supplied direct current. However, the conditions of coating applied current density (2.4-2.8) Amp/dcm^2 and coating time (0.5-1) minute, the electroplating process conditions are listed in the table (3).

Table 3,
Nickel electroplating process conditions.

Electroplating Condition	Standard Parameter	Actual parameter
Temperature of electroplating	50 - 60°C	53°C
P.H of electrolyte	3.5 - 5	4.8
Size of bath	-	50 liters
Sample size	-	20cm x 2cm
Period of electroplating	Max. 30 min.	0.5-1 min.
Number of anodes	At least 2	2
Area of anodes	Double cathodes area at least	2 (625 cm^2)
Area of the cathode (sample)	Less than anodes area	40 cm^2
The cathodic current efficiencies CCE	High	89%
Nickel bath type	Watts bath	Commercial watts bath
Range of anode and cathode	Minimum 2.5cm	25 cm

While the second stage represented by silver layer has been obtained via electroplating with the conditions displayed in table (4). They have been firstly immersion in glass bath containing 2 liters consist of silver cyanide 1 g/L, bicarbonate sodium 20 g/L and Silver Nitrate 1 g/L at warm temperature 40°C, and 2.5 Adcm^2 for (30 sec) to obtaining the desired thicknesses.

Table 4,
Silver electroplating conditions.

Electroplating Conditions	**Standard Parameters**	**Actual Parameters**
Temperature of electroplating	38 - 50°C	40°C
Current density	0.5-10 Amp/dcm^2	2.5 Amp/dcm^2
pH.	3.5 - 5	4.8
baths size	-	2 liters
samples size	-	(20 x 2) cm
Electroplating time	Max. 37 min.	2 min.
Area of anodes to the cathode	2 -1	2(4 x20 x0.9 cm)
The cathodic current efficiencies CCE%	Mid-high	70%
Anode type	Ag regular grade	Commercial, S.S 304
Type of bath Sliver	Cyanide bath	Commercial Cyanide bath
Anode and cathode distance	Minimum. 2.0 cm	3 cm

4. Thermal Evaporation Preparation Procedure

Thermal evaporation, which is a kind of physical vapor deposition system that has been presented in Fig. (5), is used to obtain a thin film. Prior to placing the substrates inside the vacuum chamber, the substrates were prepared for coating material into the boat made from molybdenum, the substrate pieces were cleaned and polished in order to remove the dust, grease, and other strange particles, and the vacuum chamber was evacuated to a 2.5×10^{-4} Pa base pressure, as presented in table (5).

Table 5,
Evaporation and melting temperatures of CuNP

Metal	**Density Bulk g/cm^3**	**Melting Temperature, ° C**	**Vapor pressure, mbar.**	
			10^{-4}	**10^{-5}**
Copper (Cu)	0.46	1083	1.1-2.7	3-3.7

The process started with weighed of powder nanomaterials to be filled it into Molybdenum boat, and placed at the center of the champers. The evaporating of copper powder nanoparticles begin by heating the boat through the control of the current and voltages smoothly and gradually to get a uniform deposition rate resulting in a thin film of copper. The deposition process used in this work carried out by the PVD system consists of units, as shown in Fig. (5).

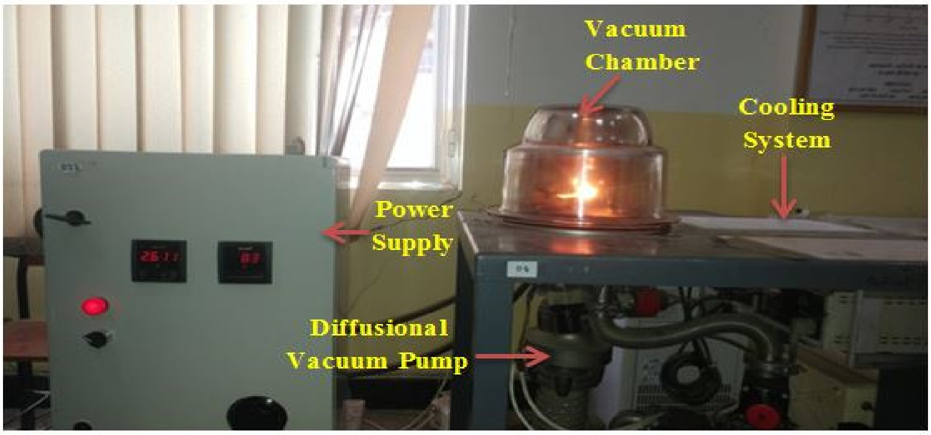


Fig. 5. Thermal evaporation unit using in study.

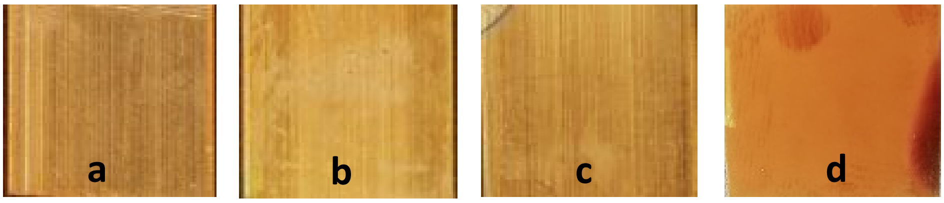


Fig. 6. Photo images of coated samples; (a) Cu-CuNP (b) Cu-Ni-Ag-CuNP (c) Cu-Ni-CuNP and (d) Si-CuNP.

5. Results and Discussion

5.1 X-Ray Diffraction Result

X-ray diffraction (XRD) spectrum of the Cu with Cu Ka radiation (E=1.0454A°) at a scanning rate of (10 deg. per sec) and angle (Θ) ranging from 20 to 80, Fig. (7) shows the X-ray patterns of crystalline structure of the copper substrate, Cu-Ni, Cu-Ni-Ag and copper nanoparticles (CuNP) combined with Ni-Ag from bottom to top, respectively. Black lines diagram indicted the strong peaks at angles 43.4029^{o} and 50.4952^{o}, and 74.1857^{o} corresponding to the copper substrate without coatings [7], while the green color shows peaks and presence of nickel that plated on copper substrate demonstrated at peaks 49.7181^{o} with structure (200) regarding to XRD card number 451027, on the other side sliver revealed in diagram blue color at peak 44.3887^{o} with structure (200) (XRD card number 04-0783). Fig. (6) displays with a red color showing nano-copper thin films strong peaks at angles, 43.2435°, 50.3690°, 74.0647° with structure (1 1 1) (2 0 0) and (2 2 0), respectively according to XRD card number 04-0836, moreover, the indicated nanostructure of CuNP is in agreement with T. Thirugnanasambandan [17]. These results are stated according to XRD standard numbered cards issued by the Joint Committee on powder diffraction standers (JCPDS), as the cards of diffraction depicted these result.

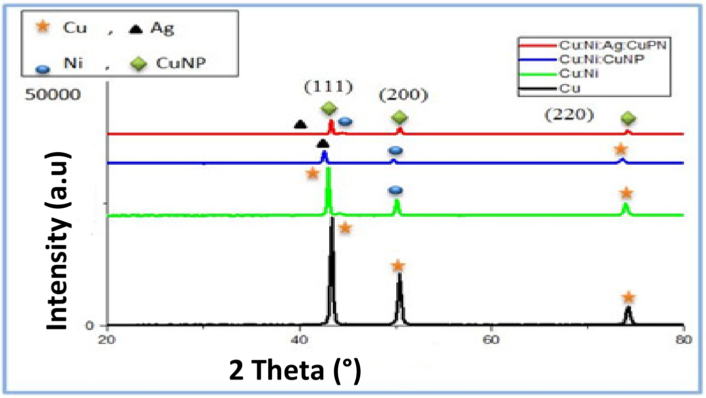


Fig. 7. X-ray diffraction pattern Ni, Ag, and CuNP deposited on copper.

5.2 Energy dispersion X-ray fluorescence (ED-XRF) results

ED - XRF result calculating the composition of thin films with micro multilayers, were investigated quantitatively and qualitatively. They exhibited the percentage of elements that are composed thin film combined with Ni-Ag, strong peak belongs to the copper, nickel and silver as illustrated in Fig. (8), while this test was strong evidence of the presence of nanoparticles belong to copper deposited directly on a pure silicon substrate with orientation (111) as shown in Fig. (9).

Group : P					
Sample: 25					
Analyze	**Result**	**[3-sigma]**	**Proc.Calc.**	**Line**	**Intensity**
Ni	10.544%	[0.028]	Quant-FP	Ni Ka	1798.9316
Cu	89.337%	[0.087]	Quant-FP	Cu Ka	13692.9209
Ag	0.120%	[0.010]	Quant-FP	Ag Ka	4.0013

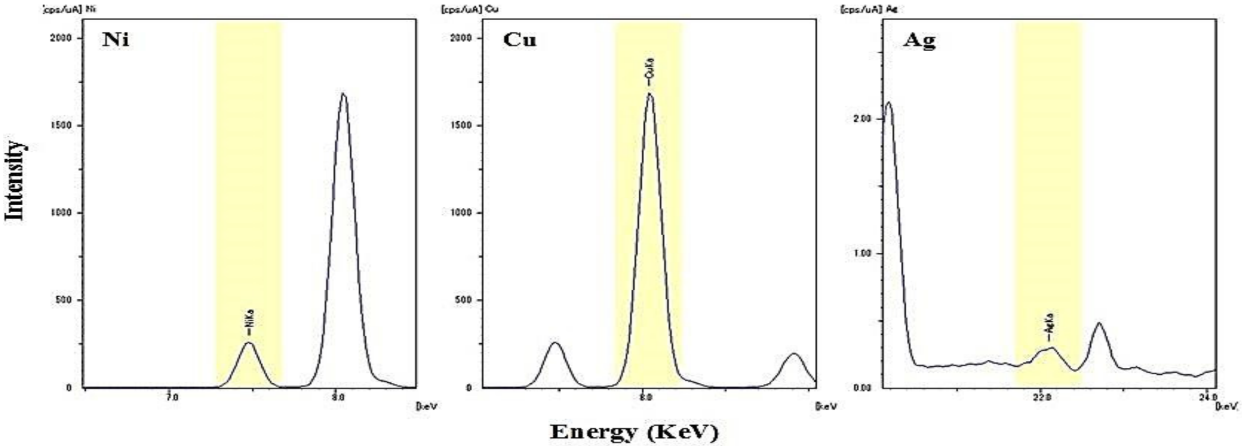
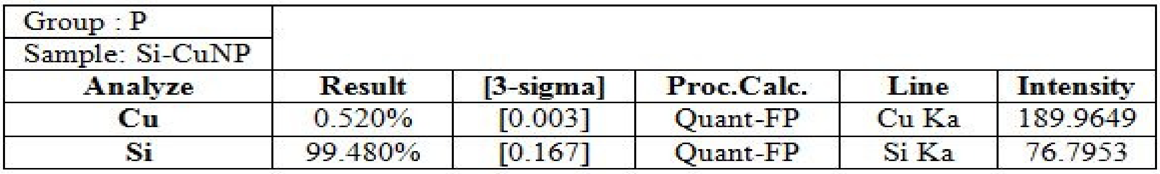


Fig. 8. Thin film ED-XRF analysis of elements (Ni, Cu and Ag).



Group : P					
Sample: Si-CuNP					
Analyze	**Result**	**[3-sigma]**	**Proc.Calc.**	**Line**	**Intensity**
Cu	0.520%	[0.003]	Quant-FP	Cu Ka	189.9649
Si	99.480%	[0.167]	Quant-FP	Si Ka	76.7953

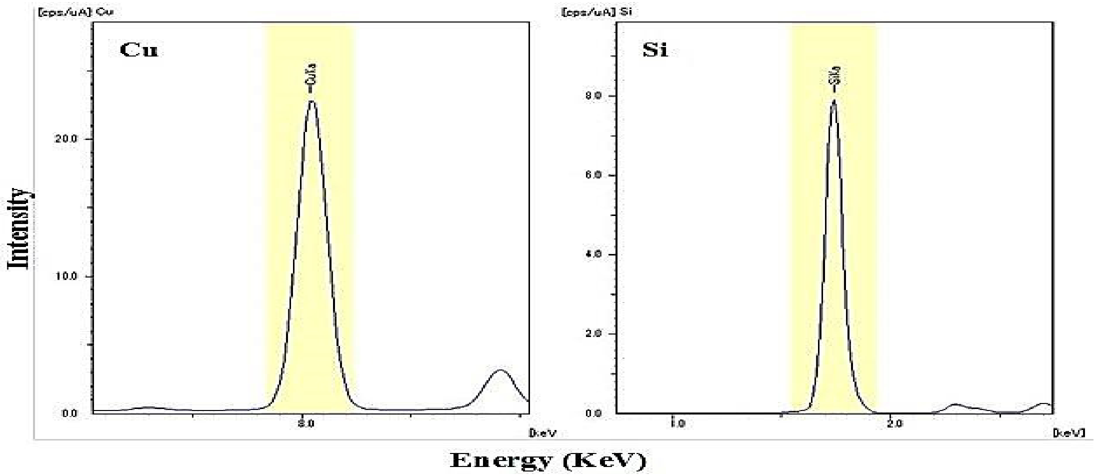


Fig. 9. Thin film ED-XRF analysis of CuNP coated on Si.

5.3 Scanning Electron Microscope (SEM)

The results pointed to the topography of the films prepared by this method more uniform as compared to the topography that prepared by other methods. It's clear from two Fig. (10) and Fig. (11) obtained by the scanning electron microscope and Field Emission Scanning Electron Microscope (FESEM) with a magnification force of (1000 x and 2٠ Kx), for Figs. (10 and 11), respectively, that the analysis of surface belongs to copper thin film prepaid by thermal evaporation appear as a dense layer of small, semi-spherical nanoparticles and distributed uniformly over the sample surface area. The presence of bright nickel layer on the surface metal substrate, which acts as an intermediate layer in addition to a good surface finish has been obtained, that will affect the characteristics of the surface morphology. This, in turn, will give uniformity and homogeneity for thin film nanoparticles as compared with the coating of copper nanoparticles directly deposited on the base metal as in Fig. (11).

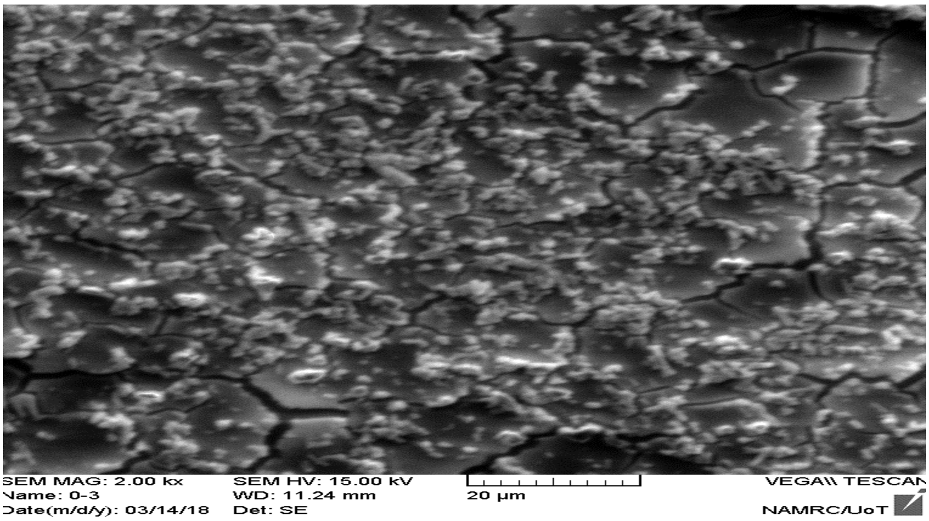


Fig. 10. SEM image of surface CuNP deposited on Cu.

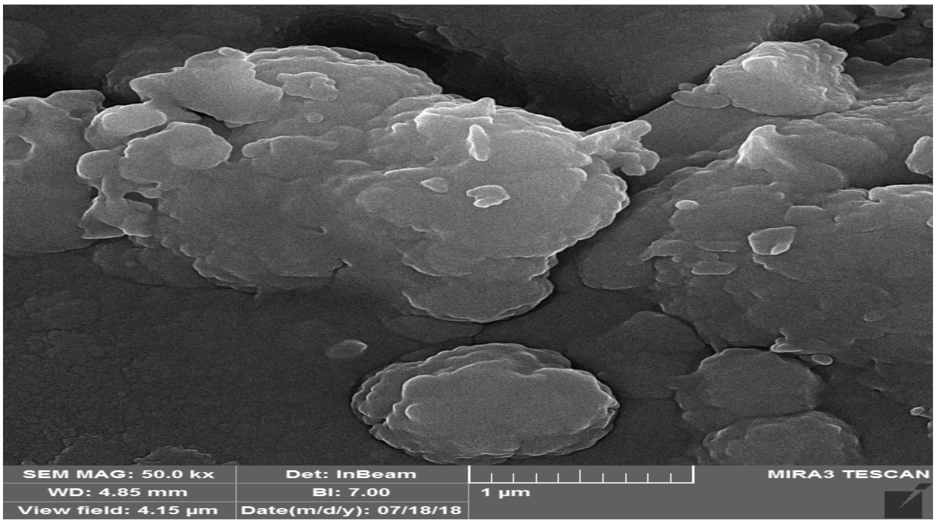


Fig. 11. FESEM image of surface Copper nanoparticles deposited on bright Nickel-copper substrate.

5.4 Atomic Force Microscope (AFM) Analysis

The surface roughness and topographies of the prepared thin films were examined by AFM. Fig. (12) Shows the AFM images of the 3D surface belong to CuNP thin films that were prepared by thermal evaporation as a function to the coating time. From the topographic results, it can be noted that the substrate preparation and time coating plays an important role affected by both grain size and average surface roughness as present in table (6) that shows the values of grain size, average surface roughness, and root mean square values for copper thin film prepared with different thickness. It was found that the average surface roughness of 1.19 nm and average grain size of 102 nm are optimum surface data due to the improvement of the solar thermal absorber output performance. It is concluded that increasing thickness as a result of increased coating time, lead to an increment in roughness values. The Fig. (13) Demonstrated the distribution chart of copper grain thin film surface with average 102 nm.

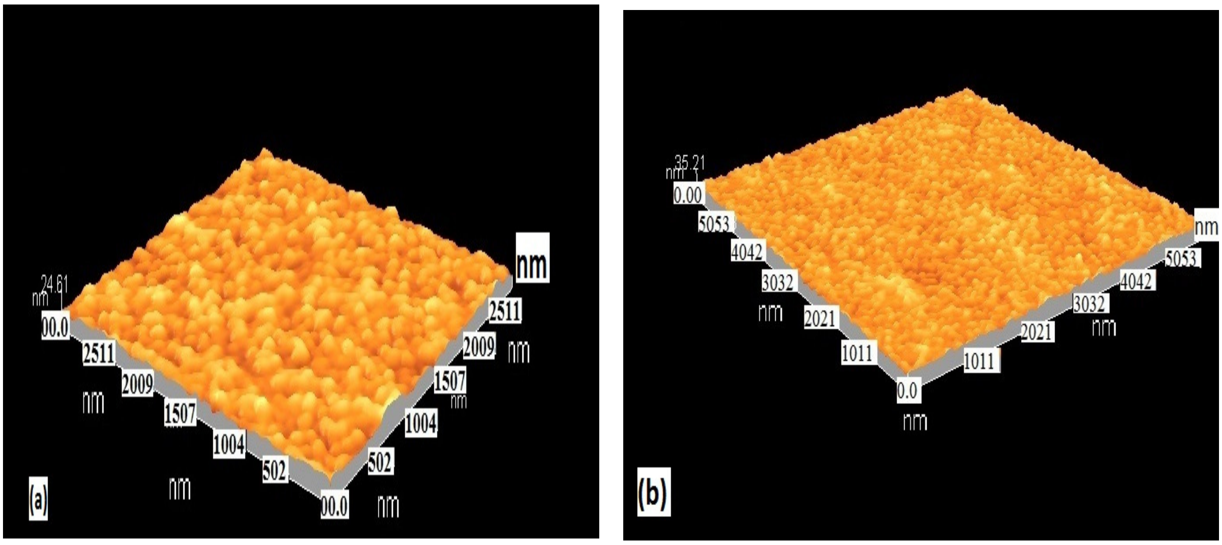


Fig. 12. The 3D surface topography of copper nanoparticles thin film with different coating times (a) 4 min, (b) 2 min.

Table 6,
Grain size distribution and roughness data values of copper thin film.

Grain Size (nm)	Average Roughness (nm)	Root Mean Square (nm)	Coating Time (min)	Thin Film Thickness(nm)
24.40	0.822	1.23	2	50
39.63	0.959	1.73	2	50
102	1.19	1.94	4	100

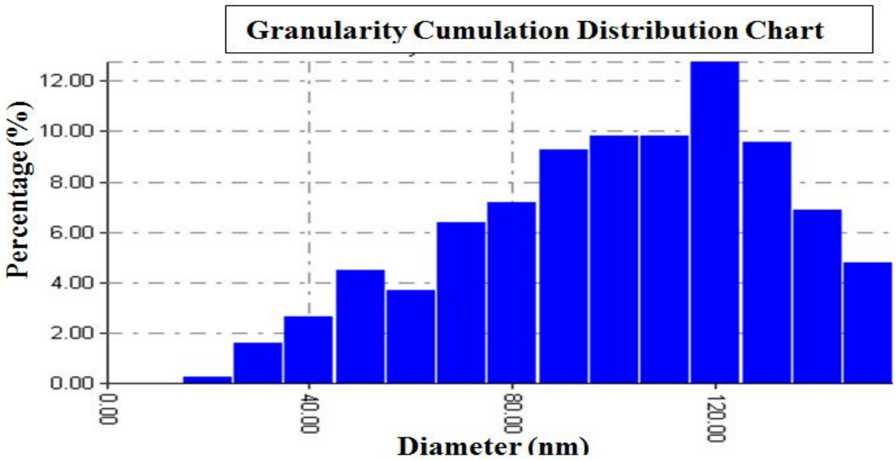


Fig. 13. Grains size distribution chart along thin film surface area examined.

5.5 Thermal Conductivity

Using hot disk equipment (Thermal Constant Analyzer) type Keithley, TPS-500 two side sensor with conditions parameters temperature 23.0 °C, out power 2 Watt, and measurement time (5sec). This was achieved to study and evaluate the thermal conductivity behavior of copper nanoparticles and influencing of bright nickel and silver on thermal properties. It was found that a slight decrease in thermal conductivity occurred, while thin film of nanoparticles exhibited a good enhancement in thermal conductivity, which led to a good improvement in absorption by the data shown in table (7), and also a good enhancement with the presence of copper nanoparticles as compared with copper nanoparticles combined with bright nickel and sliver [18, 19].

Table 7,
Thermal properties of different samples used in this work.

Samples	**Thermal Conductivity, W/m.K**	**Thermal Diffusivity, mm^2/s**	**Specific Heat, MJ/m^3·K**
Cu	3.392	0.0083	4.840
Cu- Ni -Cu NP	5.213	6.427	0.8111
Cu- Ni-Ag-Cu NP	4.268	0.6642	6.426
Cu-Cu NP	6.426	0.9874	6.026

5.6 Optical Properties

Solar selectivite coating depends on the optical properties, hence the increase in general absorption is a result of increasing the thickness of thin film. This is due to the increase in the degree of crystallization caused by increasing the thickness. This will lead to increase in the particle size hence, the incident photon on the thin film surface, which will suffer from sequential absorption done by the crystals in the same particles and, therefore the possibility of reflection or transmission will decrease, primarily by increasing the particles size. On the other hand, this may obtain higher roughness causing efficient absorption, and this agrees with researchers J. El Nady et al. [15] and M. A. Estrella [16]. The higher thickness of the thin films, resulting in greater absorption. Fig. (14) showed a comparison between the behavior of the absorption spectrum of copper nanoparticles as a function of thickness, it was found a high absorption in the wavelength range from 300-1100 nm to reach the maximum value of 93.191%, while the bright nickel and silver layers combined with CuNP deposited on copper, caused a slight decreases in thermal absorption. The presence of bright nickel and sliver reduced these values to 87.73%, 86.50%, respectively, which were concerned with the researcher Stefano Pratesi [20].

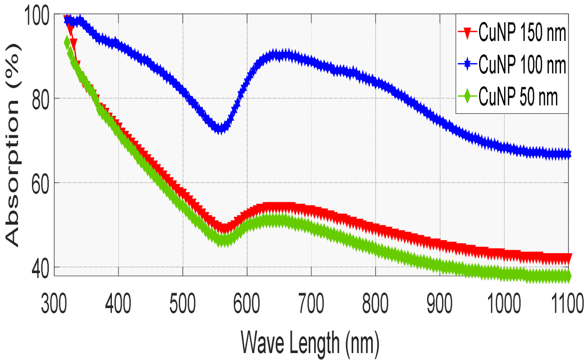


Fig. 14. The spectral absorption in the UV-Vis region for copper nanoparticles prepared with different thickness.

6. Conclusion

According to the results achieved CuNP, it revealed a good optical and thermal properties, the obtained thermal absorption was approximately equal to (93.191%), While Ni and Ag layers combined with CuNP are considered an excellent choice to avoid the diffusion of the copper thin film nanoparticles toward the copper substrate, good thermal stability and reducing the thermal emissivity. Slight decrease was observed, in the optical absorption of the coatings of about (87.73% and 86.50%) with bright nickel and silver layers gathered with CuNP, respectively. Thermal conductivity has been improved, when compared with copper substrate alone, whereas the copper nanoparticles exhibited excellent improvement of about 89.44%. On the other hand, slight decrease in thermal conductivity of about

18.87% was observed in the presence of Ni layer. These characteristics that have been obtained are desirable and can be regarded as an improvement of the performance of the efficient solar absorber.

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ألطلاء الكهربائي المتعدد الطبقات من النيكل والفضة على معدن النحاس ،والمرسب على حبيبات النحاس النانوي والمستخدم في السطوح الماصة

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الخلاصة

في هذا العمل، تمت دراسة تأثير إضافة طلاء النيكل اللامع والفضة بطريقة الطلاء الكهربائي، على غشاء رقيق من حبيبات النحاس النانوية CuNP والذي رسب عبر طريقة التبخير الحراري. أظهرت النتائج تحسنا" في قيم الامتصاصية بوجود الغشاء النحاس النانوي ، بينما أظهر وجود طبقات النيكل اللماع والفضة تعزيزا" لثباتية الحرارية. كما لوحظ أيضا الانخفاض في الخواص البصرية (الامتصاصية، والانبعاثية الحرارية) بشكل طفيف من (٩٣٪ إلى ٨٧٪) ، بينما كان للفضة تأثير طفيف اخر على الامتصاص حوالي (٨٦,٥٠٪). من ناحية أخرى، تم تقييم التوصيلية الحرارية باستخدام تقنية التحليل بالقرص الساخن(TPS)، وقد بينت النتائج تحسن جيد حصل بوجود غشاء CuNP المرسب على النحاس مباشرة بحوالي (٨٩ ٪)، في حين انخفض بنسبة ١٨,٨ ٪ في وجود طبقة النيكل اللماع CuNP كما تم دراسة التراكيب والبنية والاطوار للغشاء النانوي مع طبقات الطلاء الاخرى باستخدام تقنية حيود الاشعة السينية (XRD)، علاوة على دراسة طبوغرافية وخشونة السطح باستخدام المجهر الالكتروني الماسح (SEM) ومجهر القوة الذرية (AFM).
