

**Coating Silicon and Glass Substrate with Colloidal Silver Nano
Particles Using Thermal Spray Pyrolysis and Studying Electrical Properties.**

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Ministry of science and technology

Director of materials sciences

Center of Advance materials

Abstract

The aim of this research is to use thermal spray pyrolysis technique for coating silicon and glass surfaces with colloidal silver nano particles solutions . The importance of nanotechnology methods for nano thin films is due to their advanced properties in industry applications.is important in our point of view in industry applications for nano thin films. Spraying parameters such as spraying time, substrate temperatures, solution temperature, electrical current, voltage and the electrical frequency with colloidal silver nano particles solutions. Silver thin film coating was examined. Particle size distribution was determined using AFM technique and electrical conductivity were measured for nano silver thin films.

Keywords: Spray pyrolysis, Colloidal silver nano particles, Nano silver thin films, Electrical conductivity, Structural properties.

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طلاء قواعد من الزجاج والسليكون بعوالق جسيمات الفضة النانوية باستخدام تقنية الرش الحراري ودراسة مواصفاتها الكهربائية .

حسن اسماعيل دمبوس، وسام عباس مهدي، صبيحة جبار بدن، رياض ابراهيم عطية، حسين علي احمد،

اروي غازي ناجي ، فرح اسعد مبدر
وزارة العلوم والتكنولوجيا | ادارة بحوث المواد | مركز بحوث المواد المتقدمة

الخلاصة:

يهدف البحث الى استخدام تقنية الرش الحراري في طلاء سطوح من القواعد الزجاجية والسليكونية بمحاليل من جسيمات الفضة النانوية. تأتي اهمية البحث في بناء القاعدة العلمية لتقنيات النانوتكنولوجي في تحضير الاغشية النانوية ذات المواصفات العالية في التطبيقات الصناعية . تم دراسة ظروف عملية الرش ومنها زمن الرش ، درجة حرارة السطوح ، درجة حرارة المحلول ، التيار والفولطية الكهربائية اضافة الى التردد الكهربائي المناسب للموجات مع طبيعة الطلاء. تم فحص غشاء الفضة النانومتري باستخدام تقنية مجهر القوة الذرية وقياس التوصيلية الكهربائية للاغشية المحضرة.

1: Introduction

Thermal spray pyrolysis was first used commercially more than half a century ago in 1947 as in U. S. patents registered for (H. A. Mastier and W. O. lytle) to deposited conductive oxide films on heated glass substrate. ^[1, 2]. Spray pyrolysis involves spray of an aqueous solution of desired compounds on heated substrates, the liquid droplets vaporize before reaching the substrate in heat range of (200 -500)°C as recorded by several researches ^[6].

Control of the spray process by adjusting the different parameters , especially the spraying temperature and the spraying time ; is necessary to obtain consistent optical properties of the films ^[6] .

In spray pyrolysis method, the starting material is required to be in solution form which when sprayed on the preheated substrate[7,8]. Thin film will be formed on the substrate , when the droplets of spray solution reach the hot substrate ;decomposition character of the solution , they will be well adhered . The aim of this research, Nano-collidal Ag solutions thin films are prepared by thermal spray pyrolysis technique for different thickness , deposit on two substrates .

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The first type is a glass substrate at temperature (150-200) °C with spray rate (25-50ml/hr.) , while the second type is silicon solar cell at temperature (200-300) °C with same spraying rate . These two types of substrates are used to study the optical - electrical properties (nano – collided Ag) films and the results could be take parts for using silver thin films in solar cell studies as reflection or anti-reflection coating on glass and silicon to solar radiation.[6,7,8,9,10]

2. Experimental work

Nano colloidal silver solution (CSS) were used in this work were prepared by Arc-Discharge method in our lab. The Dc arc-discharge system consist of DC pulsar power supply provides a pulse voltage of (70 - 100) V for (2 ms) and (20 -40) V around (10 μ s). The electrical current could reach during that moment (4-10 A) where the etching current caused arc-discharge pulse etched Ag wires (anode and cathode). Ag wire evaporated and condensed in water. Servo control system keep the gap between the Ag electrodes in few microns⁽¹⁾ . Other parameters should be control during this technique such as the speed of stirring - time of process, water volume, distance of the gap, arc-discharge current and other parameters. The transparent solution converts to characteristic pale yellow color(400nm) with particle size distribution (50-560)nm by using AFM technique ^[12,13] Before deposition process; Ag solutions were reactivated using ultrasonic nebulizer for 15 min.

Nano-colloidal silver thin films deposited on glass substrates, made in Germany from "objekhrager factory", have dimensions relate to examination holder test .The cleaning of substrates is very important process where dust or oil affected on the properties of thin films. Washing the glass substrate with water and cleaning solution to remove any oil or dust that might be on the substrate of the surface and then washing with tap water and Place the glass slides in a clean beaker containing distilled water and alcohol, and then rinse in ultrasonic bath kind for 60 min .

The system consists of many simple interments to be used in preparation of thin films. 40 ml glass container with less than 1 mm diameter nozzle spray conducted with plastic tube for gas carrier and Heater (hot plate), for heating substrate. Temperature and pressure controller. Thin films are deposited on heated glass and silicon substrate at temperature rang (150 -300) °C within time

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of deposition (3s) each in order that the substrate does not lose its thermal stability and to start spray a gain many times as desired thickness of films obtained .

This part involves description of the equipment used in thermal pyrolysis technique and the method of preparation of the colloidal silver nano particles thin films and their optical and electrical measurements. Ag thin films coating on glass and silicon substrates were examined using XRD technique (SHAMADZU-JAPAN,220V/50Hz) for crystal structures of Ag thin films ,UV-vis spectrophotometry for optical examination (SP-3000,JAPAN), atomic force microscopy AFM(ANGSTROM adv.AA3000,USA) and optical microscope (MG↓ 600,TYPE 120,JAPAN) for surface morphology, thickness measurements using(SLI-FITER SIN:100 8/911,STELLER NET ,INC) and electrical conductivity measurement by four probe technique using (HMS-3000,ECOPIE) and roughness measurement for thin films examination using (TR-100,CANDA PRODUCTS) were used for each sample.

Results and Discussion

Film homogenous is examined by photo microscope and crystalline structure by optical microscope and X-ray diffraction (type shamadzo) CuK α target , 1.5406 Å X- ray wavelength, current (30 mA) and voltage (40 kV) .Film thickness is the most important parameters since it is used to allocate the properties of the films ^[12].Laser method was used for thickness measurements and weight method also .The substrate are weighted before and after deposition using electrical balance of type (AE 166 met) . The variation in weight of substrate represent the thin film thickness, (t) is calculated according to the following equations : ^[11]

$$t = \frac{\Delta m}{f \cdot a \cdot b} \dots\dots\dots(1)$$

Where Δm is the variation mass of substrate which is the mass of thin films in (gm) .a,b : are density substrate ,f : the density of material(Ag).This method given a thickness approximately because not all the material is deposited on the substrate but some of the material lost or fleeing on sides of the heater.

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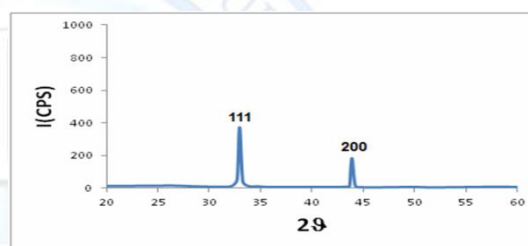
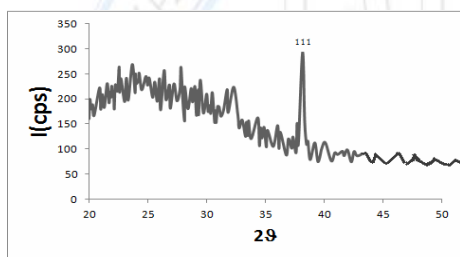
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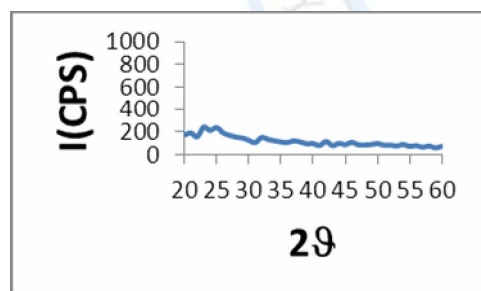
Figure (1) shows the silver powder samples on glass substrate were subjected to x-ray diffraction to study their structure and it shows the crystalline patterned of silver powder with characterized peak position(33.0337 degree)with high relative intensity (100%) to plane (111) and second peak position(43.9817 degree) with relative intensity (52%) to plane (200)and this results was agreed with other study^[12-16] ; while for silicon substrate as shown in figure (2) ;its show the effect of sintering process of Ag thin films for 1hr.at 300°C where X-ray diffraction was so characteristic to high intensity(100)peak at angle position (29.6472)degree for plane indexes(100) with other low intensity peaks were related to silicon substrate.^[14,15]

(a)
(b)
(b)

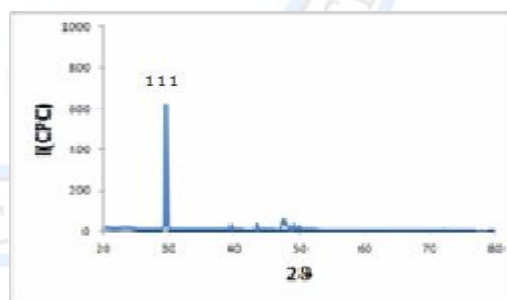
**Figure
(1): XRD
glass**



**pattern of nano Ag thin film (50 nm)on
substrate (a) before (b) after sintering.**



(e)



(f)

**Figure (2): XRD pattern of nano Ag thin film (46)nm on silicon substrate (e) before
(f)after sintering.**

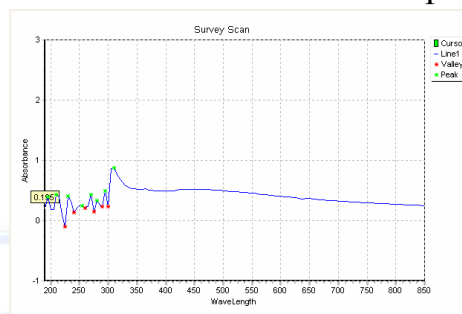
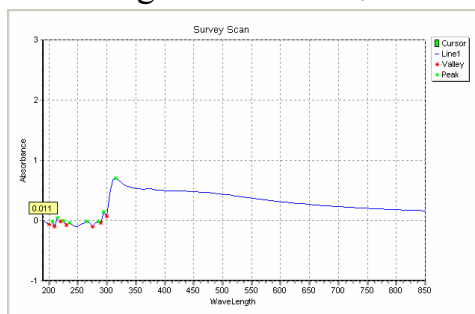
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Figure (3g,3h) shows typical UV-vis spectrometer analysis for the silver thin films on glass substrate , and this method show maximum absorption ratio to



UV.
energy
band
about
320 nm

wavelength. The band at higher energy is reported to be due to the formation of various f silver particle sizes. Such presence of absorption peak in the present study confirms of formation of nano particles. ^[13-16]

(h)

(g)

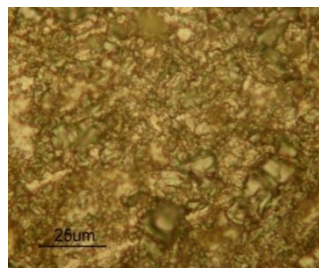
Figure(3): The UV-vis. spectrum of nano Ag thin film (g)(60)nm, (h)(65)nm on glass substrate.

The optical microscopic images figure (4)show Ag thin films on Si substrate show good alignment with Si direction plans and the vacancies could be covered by additional sparying of Ag solutions;while the AFM 3D.(figure 5) image within smaller area show the growth of silver particles in certain direction with vallays between them ; that is because of the direction of spraing is the same during the formation of the films. The depostion layer should have the same thickness and covered the the substrate spot with higher concentration and with rotating hot plate.

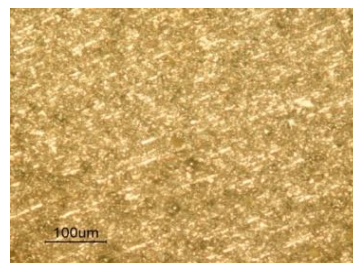
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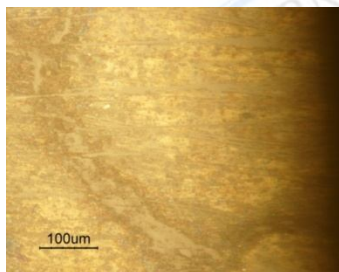
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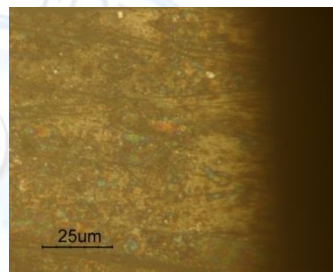
(j)Si50x



(i)Si1000x

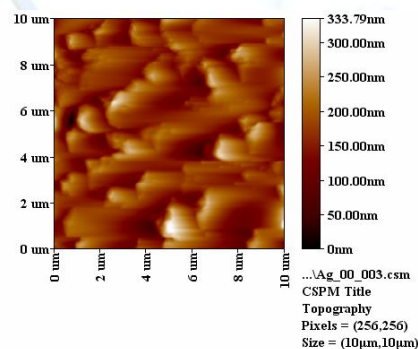


(k)g-50x

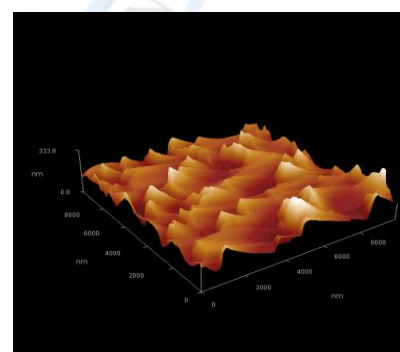


(f) g-1000x

Figuer(4): (i,j,k,f) show the surface images of nano Ag thin films on silicon(Si) and glass (g) substrat with 50x and 1000x magnification.



(n)



(m)

Figuer(5): show partical size determination(50-330)nm (m),and (n)3D.image of Ag nano thin film on glass substrat.

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Table 1 and 2 show the properties of silver and silicon substates.

Table (1): Electrical properties of nano Ag thin films on silicon substrate.

Sample	Thickness nm	Electricity nA	Resistivity Ω Cm	Roughness	R_H -effect m^2/C	Mobility $Cm^2/V.S$	Conductivity $1/\Omega$ Cm
Si-25(Ag1)	41	1	2.57	62	2.5-	2.245	3.89
Si-50(Ag2)	46	1	8.919	63	2.509	1.679	1.121

Table (2): Electrical properties of nano Ag thin films on glass substrate.

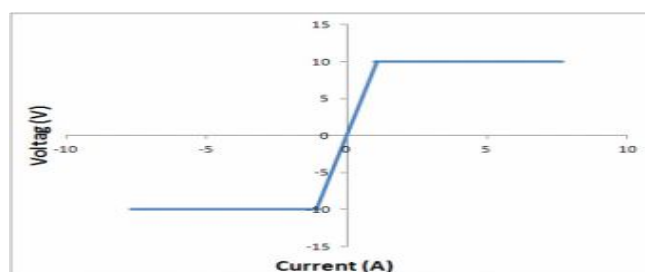
Sample	Thickness nm	Electricity nA	Resistivity Ω Cm	Roughness	H. effect m^2/C	Mobility Cm^2/VS	Conductivity $1/\Omega$ Cm
g-50-g(Ag3)	60	1	3.171	0.02	- 2.932	5.724	3.154
g-50-b(Ag4)	65	1	2.04	0.05	2.336	2.758	4.899

According to electrical properties examinations for two Ag thin films on silicon and glass substrat ; the electrical conductivity increases proportionally with thin films thickness for (1nA) A.C. current (figure 6and 7).That mean we can get conductive transperant glass coated with (60,65) nm thin film of silver nano-colloidal solution and conductive silicon coated with (40,41) nm thin film of Ag nano-colloidal solution using thermal spray pyrolises succssfully.

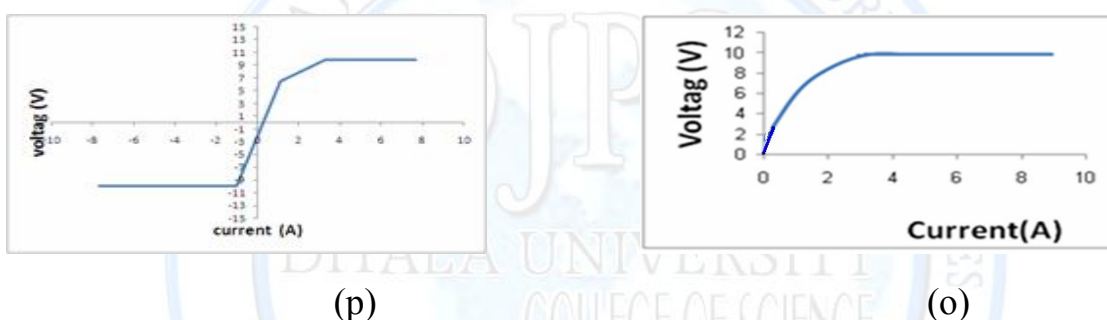
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Figuer(6):The I-V characterization of nano Ag thin film(60,65)nm on glass substrat.



Figuer(7): The I-V characterization of nano Ag thinfilm(40-41,p-46)nm on Si substrat.

Conclusions

- 1-The chemical –thermal spray pyrolysis is good tool to prepare thin layer of nano –colloidal silver (40-65)nm thick with various preparation parameters.
- 2-Colloidal silver thin films examinations show electrical properties depend on thin film thickness and surface morphology.
- 3- Crystallinity structure of Ag thin films on glass and silicon substrate should be achieved during sintering process with (300°C) for 1hr.

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