

## Fabrication of $\text{TiO}_2$ and $\text{V}_2\text{O}_5$ Thin Films by Powder Coating Technique

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Received on: 26/3/2013 & Accepted on: 11 /6/2013

### ABSTRACT

Titanium dioxide ( $\text{TiO}_2$ ) and vanadium oxide ( $\text{V}_2\text{O}_5$ ) in different mixing percentage (100, 50, 0)% from them powders as thin film on substrate of glass. the coating thickness was ( $0.37 \pm 0.03 \mu\text{m}$ ).

Thin films were inspection by microphotographs with scanning electron microscopy (SEM) and x-ray diffraction (XRD).

The results showed that thin films was prepared crystalline and also the compound ( $\text{TiO}_2, \text{V}_2\text{O}_5$ ), and the structure was regular and smooth.

**Keyword:**  $\text{TiO}_2$ ,  $\text{V}_2\text{O}_5$ , THIN film, SEM, XRD.

تصنيع أغشية رقيقة من أوكسيد التيتانيوم و أوكسيد الفناديوم  
بتقنية مساحيق الطلاء

### الخلاصة

تم في هذا البحث ترسيب أغشية من أوكسيد التيتانيوم  $\text{TiO}_2$  و أوكسيد الفناديوم  $\text{V}_2\text{O}_5$  بنسب خلط مختلفة بلغت (0,50,100%) لهما على قواعد من الزجاج وبسمك طلاء بلغ ( $0.37 \pm 0.03 \mu\text{m}$ ).

للأغشية المحضرة تم فحص التركيب المجهرى بواسطة المسح باستعمال المجهر الإلكتروني و فحص حيود الأشعة السينية .

بينت النتائج بان جميع الأغشية المحضرة كانت متبلورة و إن الأغشية المترابكة (50%) من ( $\text{TiO}_2, \text{V}_2\text{O}_5$ ) كذلك , و إن البنية كانت على العموم منتظمة و ناعمة.

## INTRODUCTION

Recently titanium oxide ( $\text{TiO}_2$ ) and vanadium oxide  $\text{V}_2\text{O}_5$  ultra thin films have been investigated with regards to their remarkable optical, electrical and photo electrochemical properties[1].

A numbers of methods have been employed to fabricate thin films, including e-beam evaporation, sputtering, chemical vapor deposition [2,3] and sol-gel process .among these methods the sol-gel process is one of the most appropriate techniques to prepare thin film[4].

Application of  $\text{TiO}_2$  and  $\text{V}_2\text{O}_5$  like microfiltration media properties, catalytic reactors, cathodic protection, orthopaedics [5, 6].

The main purpose of the present paper is fabrication of  $\text{tio}_2$ ,  $\text{v}_2\text{o}_5$  and the mixing ( $\text{TiO}_2$ ,  $\text{V}_2\text{O}_5$ ) thin films deposited on the surfaces of glass from its powders and study its structural properties.

## EXPERIMENTAL WORK

Steps of the work are:

a- Raw materials for the preparation of thin films:-

The raw materials were used in this work to fabricate thin film are  $\text{tio}_2$  and  $\text{v}_2\text{o}_5$  powders. These powders are prepared in process as mention below:

- Preparation of materials powder are milling process, sieving process, particle size measurement, preparing of powder ratio.
- Sieving process was carried out in materials engineering department /university of technology by the equipment measurements (93, 75.53, 38, and 25)  $\mu\text{m}$ .
- Particle size test is carried out in advanced materials research center at the technology and science ministry. Specification of the device is (sald-2101) laser diffraction particle size analyzer shiatsu. The results of the powder that was used in the research are as follows:  $\text{v}_2\text{o}_5=0.421 \mu\text{m}$ ,  $\text{tio}_2 = 0.390\mu\text{m}$  as in Figures (1 and 2) below.
- The powders materials have been weighed according to the selected ratio to prepare batches for the spray process by device type Denver instrument balance, maximum weight (210) g.

B-System preparation:-

The system which we used for preparation of thin films by powder deposition process are constructed from (nozzle, compressor device, electric heater, flow meter, beaker, connection and temperature measuring device remotely) as shown in Figure (3).

c- Substrate preparation:-

Glass substrates have a standard dimension as (5\*2\*0.2) cm<sup>3</sup> and purity (99.99%), cleaning process for the glass substrates was done by ethanol alcohol for 10 min then rinse with distilled water and drying in air. at last the glass substrates was putted and fixed on the electric heater with control temperature.

D-spraying process:-

Low-velocity (gas- powder) mixture moves from the feeder into pre-chamber under pressure. Gas which we used in this work is air. After powder particles leaving the nozzle, interact with substrate and create a coating.

Spray parameters are listed in Table (1).

E- Annealing: was done at  $(350)^\circ\text{C}$  for (one hour) in furnace type (nabertherm) .

f- Testing:

XRD test was carried out in nanotechnology and advanced materials research center / the university of technology type (xrd-600), manufacturer company name (shimadzu) japan, while sem test was carried out in nanotechnology and advanced materials research center / the university of technology.

## RESULTS AND DISCUSSION

Figure (4) shows the result of x-ray diffraction to the titanium oxide  $\text{TiO}_2$  thin film, after compared the results with ASTM card no. 21.1276 it was identical. Where the film was poly crystalline with tetragonal crystal system, comparing the results that matching with higher intensity [7,8].

While for vanadium oxide ( $\text{V}_2\text{O}_5$ ) thin films:

Figure (5) shows the results of x-ray diffraction for vanadium oxide film shows the identical state with ASTM card no. 41.1426 with crystalline was the type of orthorhombic crystal system. no peaks corresponding to any vanadium oxide other than  $\text{V}_2\text{O}_5$  or secondary phases and the higher intensity peak for pure  $\text{V}_2\text{O}_5$  was at (110) compared to other (hkl) [9].

After making a comparison between the standard values and the results obtained, we find it is shown in tables. and thus can be calculated lattice constants for both articles.

thus can be calculated lattice constants for the both films, by using the following equations for  $\text{TiO}_2$ :

$$1/d^2 = \{(h^2+k^2)/a^2\} + \{l^2/c^2\} \quad \dots (1)$$

and the following equations for  $\text{V}_2\text{O}_5$ :

$$1/d^2 = (h^2/a^2) + (k^2/b^2) + (l^2/c^2) \quad \dots (2)$$

as shown in Table (2).

By comparing the results obtained with the of previous research special thin films for  $\text{TiO}_2$ ,  $\text{V}_2\text{O}_5$  it find a clear identical [9, 10, 11]

After making mixing for powders at ratio (50% $\text{TiO}_2$ , 50% $\text{V}_2\text{O}_5$ ) in order to determine the type of the crystalline system and structure, results shown in Figure (6).

The results of the mixing film is identical crystalline and did not show any new phases or solid solution, neither show compound or complex, nor other oxides. Vanadium /titanium oxide observed are coexist with each other [12, 13].

Figure (7) shows SEM for  $\text{TiO}_2$  film .it can be seen the presence of a smooth structure and there are arranging of area that appears in clustered groupings in the

pocket with a lots of nano grain and pores of  $\text{TiO}_2$  and the diameters of about  $0.2\mu\text{m}$  cover the surface [8].

Figure (8) shows the SEM for  $\text{V}_2\text{O}_5$  which consists of aggregates of thin sheet particles with irregular shape and also varying sizes, some of which have up to needle smooth and some of them have almost laminated irregular shape and groupings, this agree with other search [11].

Figure (9) shows the composed mix. of (50%  $\text{TiO}_2$ , 50%  $\text{V}_2\text{O}_5$ ) film and it can seen from the picture it found homogenous and construction structure [14].

## CONCLUSIONS

1-A crystalline titanium dioxide ( $\text{TiO}_2$ ) film with structure of tetragonal and also a crystalline vanadium oxide ( $\text{V}_2\text{O}_5$ ) film with structure of orthorhombic, were successfully deposited with the cold spray process and coating thickness was about ( $0.37\mu\text{m}$ ).

2-Mixing powder were used to deposited for (50%) ratio without any phases or compounds.

3- Scanning electron microscope photographs identified that the structure has been homogeneous and suitable for applications in general.

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**Table (1) spray parameters.**

gas flow	gas pressure	gas temperature	distance	substrate temperature
2.5 ± 0.01 l/min	7 bar	25 c°	20 cm	100 c°

**Table (2) the results of lattice constant.**

Powder	$a_{ASTM}$	$a_m$	$b_{ASTM}$	$b_m$	$c_{ASTM}$	$c_m$
$TiO_2$	4.5933	4.60	—	—	2.9592	2.960
$V_2O_5$	11.516	11.645	3.565	3.560	4.372	4.377

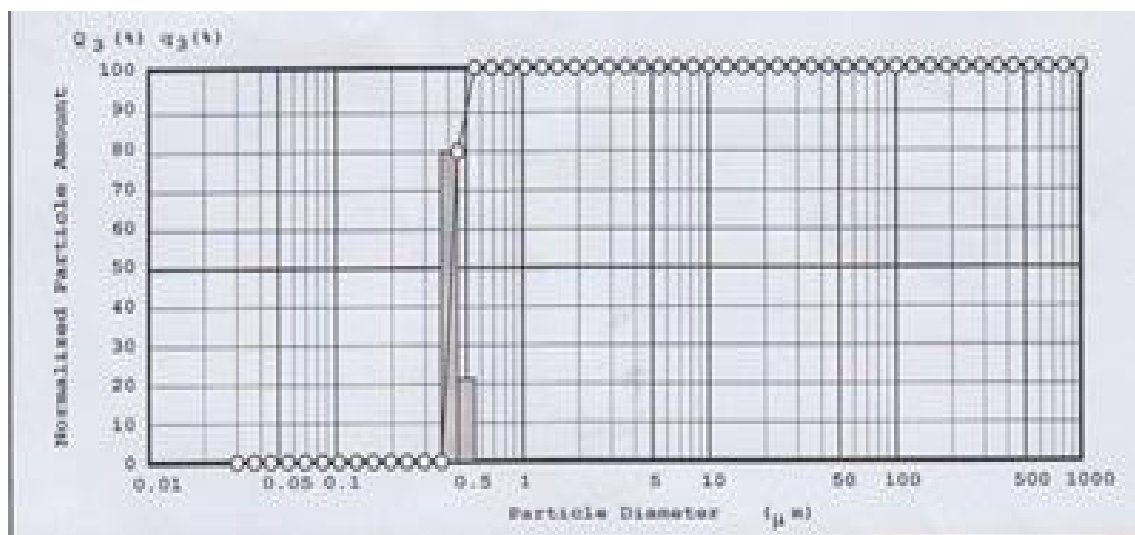


Figure (1) Shows particle size for  $\text{V}_2\text{O}_5$ .

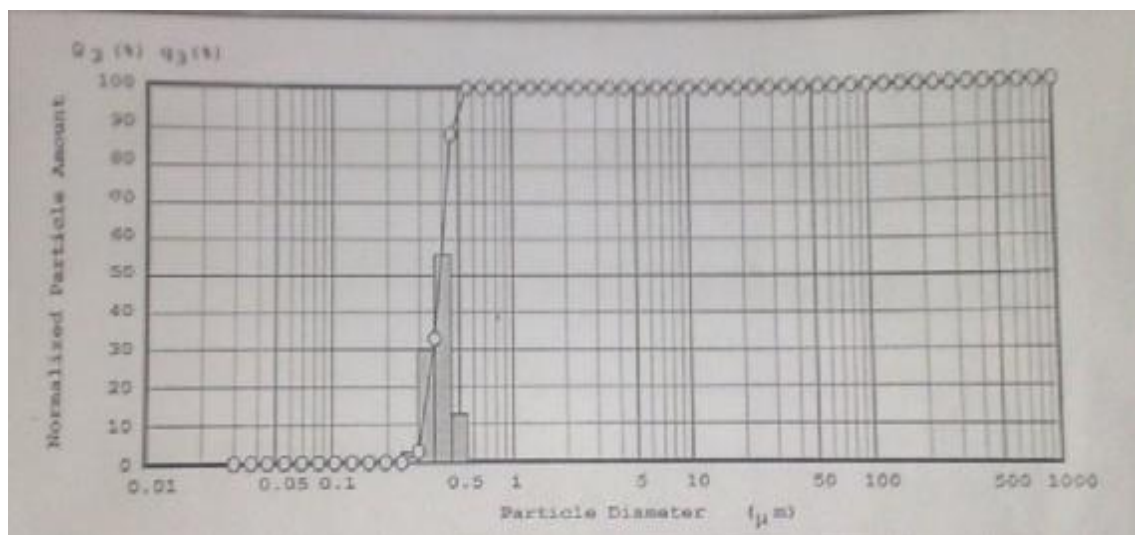


Figure (2) shows particle size for  $\text{TiO}_2$ .

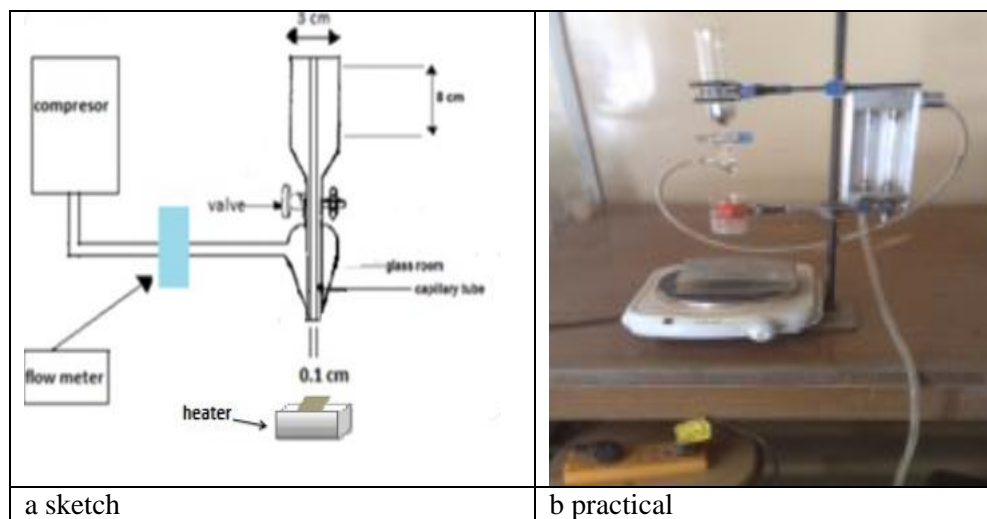


Figure (3) System for process.

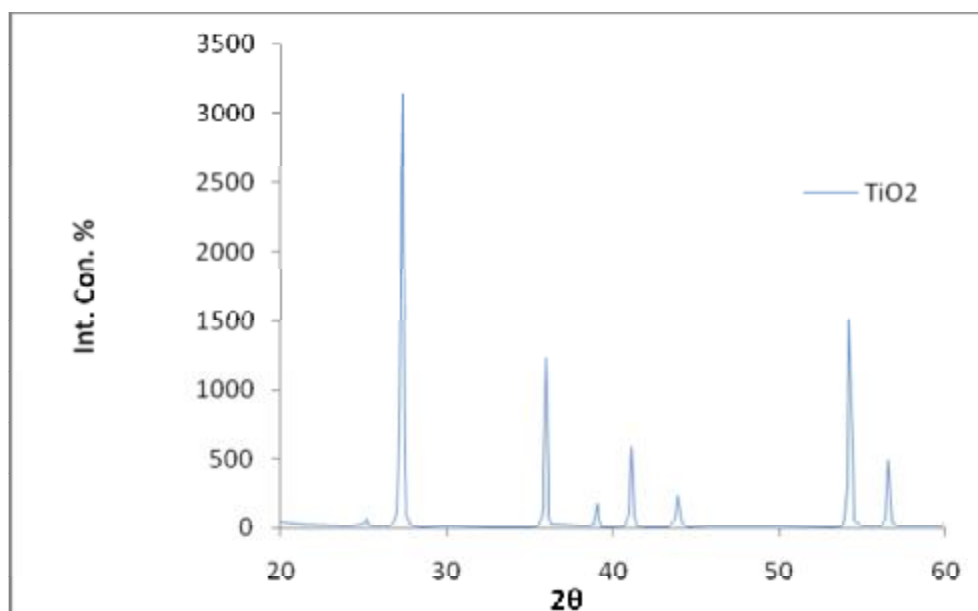


Figure (4) Show x-ray diffraction for  $\text{TiO}_2$ .

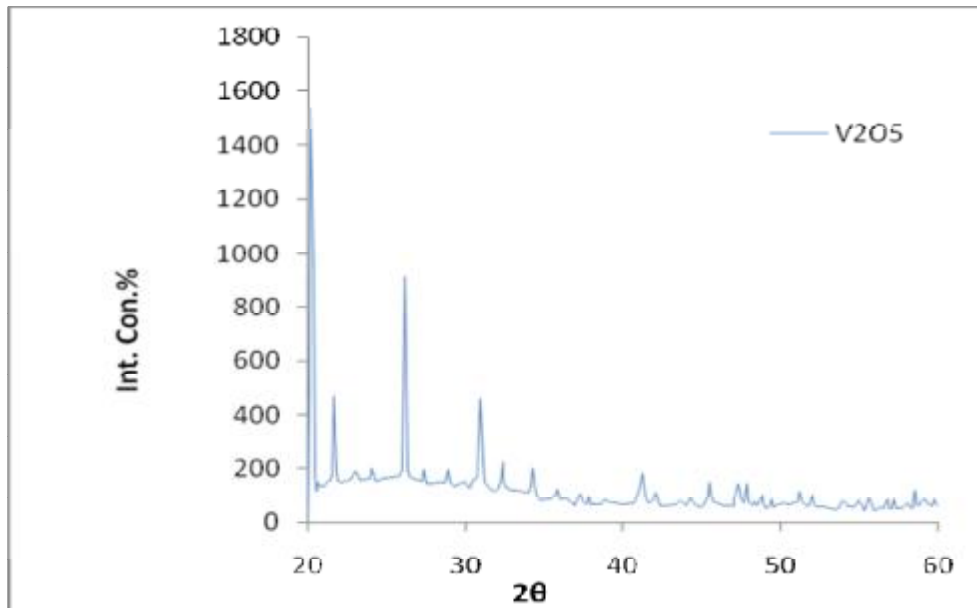


Figure (5) Shows x-ray diffract for  $\text{V}_2\text{O}_5$ .

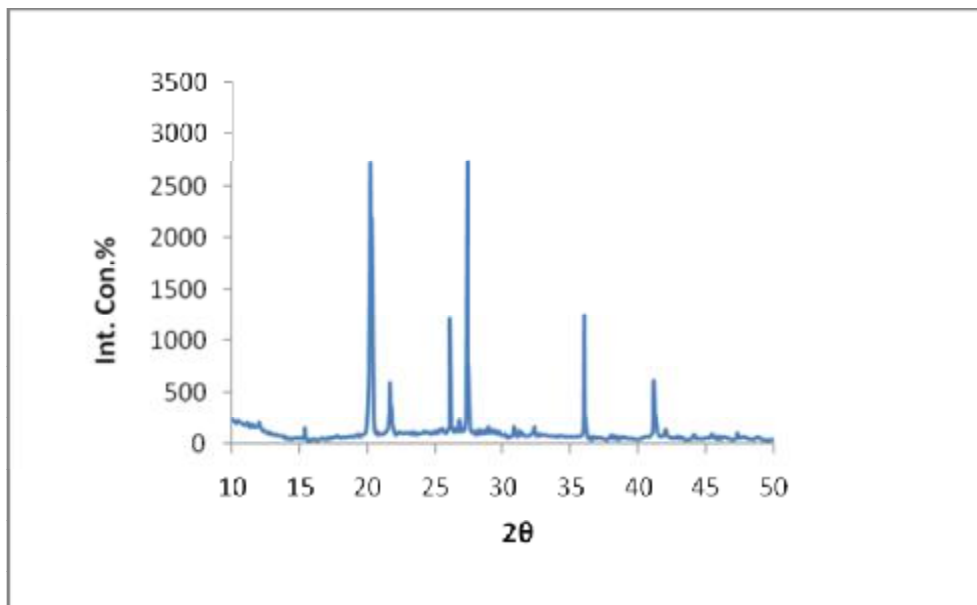


Figure (6) Shows x-ray diffraction for 50%  $\text{TiO}_2$ , 50%  $\text{V}_2\text{O}_5$ .



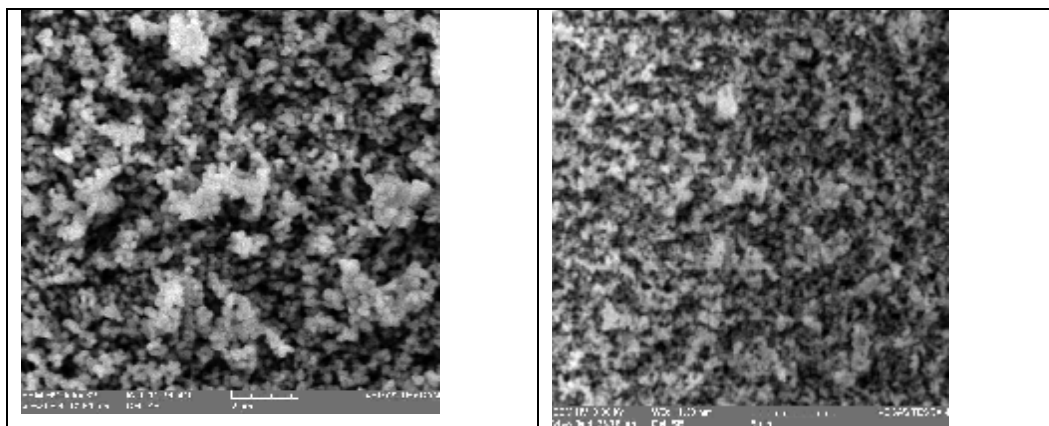


Figure (7) Shows SEM for  $\text{TiO}_2$  100 %.

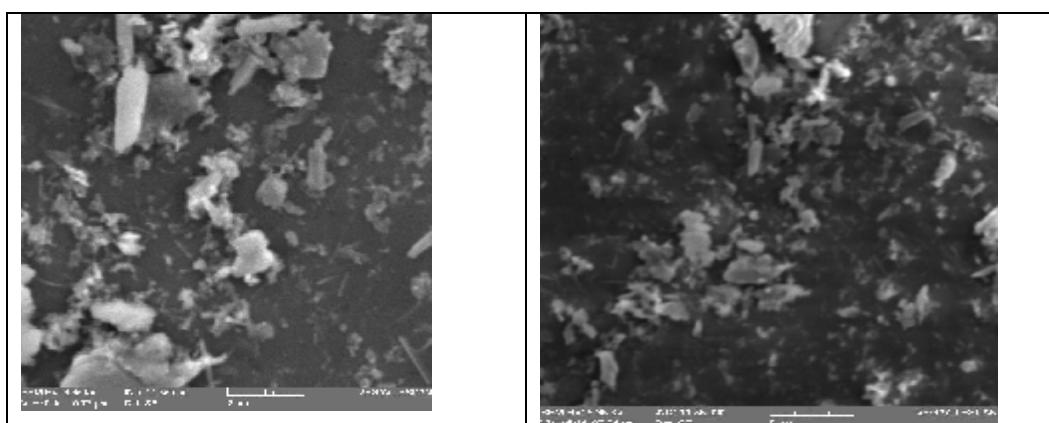


Figure (8) Shows SEM for  $\text{V}_2\text{O}_5$  100 %.

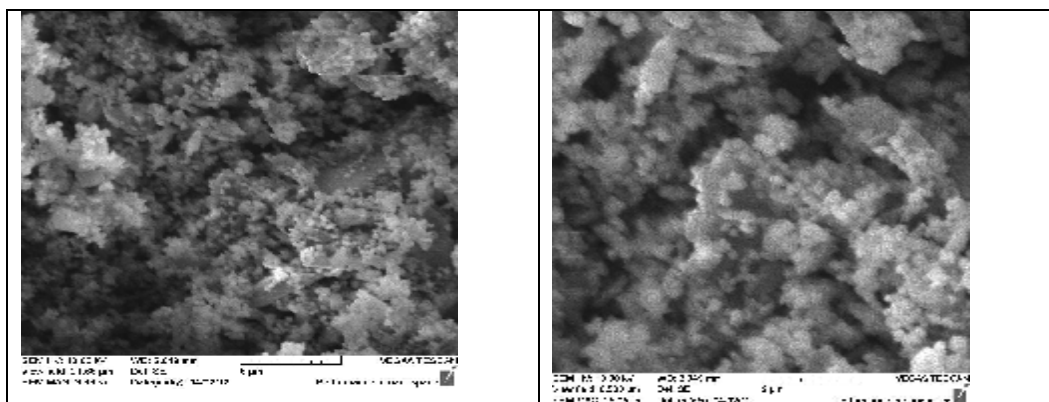


Figure (9) Shows SEM for  $\text{TiO}_2$  50% ,  $\text{V}_2\text{O}_5$  50% thin film.