Effect of Sputtering pressure and partial pressure on Structural Properties of TiO_2thin Films

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Abstract:

Effect of sputtering pressure and partial pressure on structural properties of TiO_2 films prepared using DC-sputtering to glass substrate was investigated. sputtering pressures are changed (1.8,2.8,3.8,4.3 pa) at constant O_2 /Ar Ratio(5%). Measurements reveal that the TiO_2 films at the sputtering pressure 1.8 pa is amorphous , while at increasing pressure to 2.8 pa becomes crystalline with Rutile phase (110) and when we increase the pressure to 4.3 pa get crystalline structure with anatas phase (101) .Grain size is calculated per crystalline structure of the anatas and rutile (15.7) nm and(14.2) nm respectively. For constant sputtering pressure (2.8pa) and changedO_2/Ar Ratio(10% ,15% ,20% ,25% ,30% , 35%),TiO_2 thin films are a amorphous, except percentage (25%) is crystalline with anatas phase (101) and with grain size (15.7 nm).

Key words: Structural properties,TiO_2 thin films, DC-Sputtering .

تاثير ضغط الترذيذ والضغط الجزيئي على الخصائص التركيبية لأغشية ثنائي اوكسيد التيتانيوم المحضرة باستخدام الترذيذ بالتيار المستمر

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الخلاصة:

تم في هذا البحث دراسة تاثير كل من ضغط الترذيذ والضغط الجزيئي على الخصائص التركيبية لاغشية ثنائي اوكسيد االتيتانيوم المحضرة باستخدام منظومة الترذيذ بالتيار المستمر على ارضيات زجاجية . الخطوة الاولى في البحث تم تثبيت الضغط بتثبيت الضغط الجزيئي للاوكسجين عند النسبة (5%) وتغير ضغط غاز الترذيذ (1.8,2.8,3.8,4.3pa) وثانيا تم تثبيت ضغط الترذيذ عند الضغط (2.8 pa) وتغير الضغط الجزيئي للاوكسجين(10%,15%,20%,25%,30%,35%) .من قياسات حيود الاشعة السينية تبين ان الاشعة عند الضغط (1.8 pa) عشوائية التركيب ولكن عند الضغط (2.8 pa) تصبح بلورية التركيب وبطور الروتيل وبزيادة الضغط اكثر تصبح الاغشية وبالتحديد الضغط (4.3 pa) بلورية التركيب وبطور الاناتاس وبتوجيهية (101) , بينما عند تثبيت النسبة وزيادة الضغط سوف نحصل على تركيب بلوري وبطور الاناتاس وبتوجيهة (101) عند النسبة 25%.
قمنا بحساب الحجم الحبيبي للاغشية المحضرة كدالة لكل من ضغط الترذيذ مرة والضغط الجزيئي مرة اخرى.

الكلمات المفتاحية :الخصائص التركيبية ،الاغشية الرقيقة للثنائي اوكسيد التيتانيوم،الترذيذ بالتيار المستمر.

1- Introduction:

Titanium oxide (TiO_2) can exist as an amorphous layer and also in three crystalline phases: anatase (tetragonal), rutile (tetragonal) and brookite (orthorombic). Titanium oxide (TiO_2) is one of the most extensively studied materials, and is known to exist in an amorphous form and to crystallize in three distinct structures: two tetragonal phases, anatase (a = b=3.785 °A, c = 9.514 A^o) and rutile (a = b=4.593 A^o, c = 2.959 A^o), and a third orthorhombic phase, brookite (a = 5.456 A^o, b = 9.182 A^o, c = 5.143 A^o). Among them, rutile is not only the densest, but also thermodynamically the most stable phase, so it is interesting for optical coatings [1, 2] .The figures (1a),(1b)and(1c) are represented three crystalline phases: anatase (tetragonal), rutile (tetragonal) and brookite (orthoromic) for TiO_2 [3]. The refractive index at 500 nm for anatase and rutile bulk titania is about 2.5 and 2.7 respectively [4]. TiO2 anatase nanoparticles were synthesized from a titanate for application in dye-sensitized solar cells[5]. These remarkable properties make them suitable for wide applications, such as dye-sensitized solar cells[6,7,8] , gas sensors[9] , and dielectric applications[10] . It was found that the rutile phase crystallinity increased with decrease in total pressure[11] . Pure anatase phase is only attained when the total pressure is higher than 0.7 Pa [12]. XRD results show that the rutile phase is dominant at low pressure range (0.3-0.6 pa) whereas the anatase phase is predominant in the high pressure range (0.8-2 pa)[13].There are many deposition methods used to prepare TiO_2 thin films, such as electron-beam evaporation , ion-beam assisted deposition , DC reactive magnetron sputtering, RF reactive magnetron sputtering , Sol-gel methods , chemical vapor deposition and plasma enhanced chemical vapor deposition .

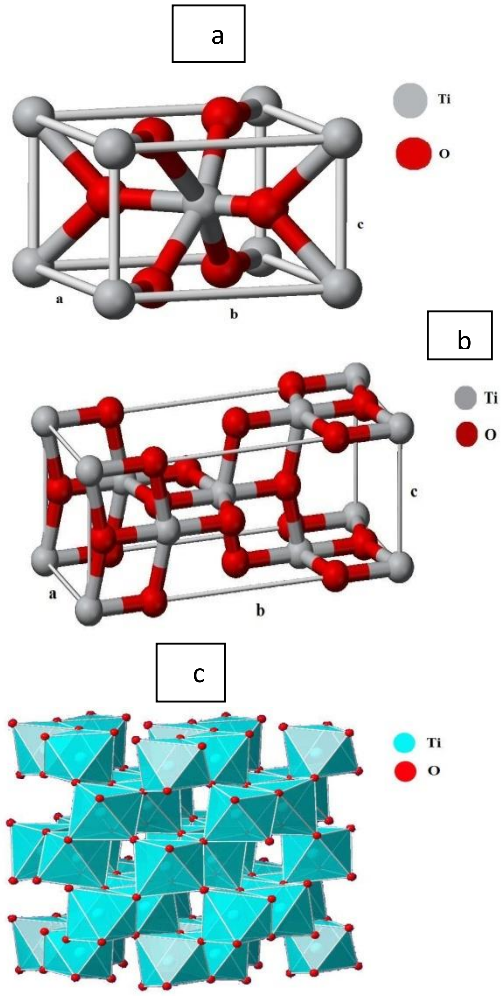

Fig. (1) a. Rutile structure for crystallineTiO_2 . b. Anatase metastable phase for crystalline TiO_2 . c. Brookite structure for crystalline TiO_2[3].

2-conclusion:

Effect of sputtering pressure and partial pressure on structural properties of TiO_2 films prepared using DC-sputtering to glass substrate was investigated. sputtering pressures are changed (1.8,2.8,3.8,4.3 pa) at constant O_2 /Ar Ratio(5%). Measurements reveal that the TiO_2 films at the sputtering pressure 1.8 pa is amorphous , while at increasing pressure to 2.8 pa becomes crystalline with Rutile phase (110) and when we increase the pressure to 4.3 pa get crystalline structure with anatas phase (101) .Grain size is calculated per crystalline structure of the anatas and rutile (15.7) nm and(14.2) nm respectively.$d = \frac{0.9\ \lambda}{\beta \cos\theta}$

3-Experimented setup :

The TiO_2 films were grown on unheated substrates from glasses by DC- sputtering with sputtering voltage 1kv . The purity of the titanium target fixed on the holder represent the cathode was 99.9 % with diameter (Φ60×2mm). The base pressure was changed (1.8,2.8,3.8 and 4.8 pa), at constant O_2 /Ar Ratio(5%). the Deposition time was constant at 1:15 h. The desired total pressure for deposition was maintained by flow matter vacuum valve with vacuum pumpe . High purity argon (99.999 %) and oxygen (99.98 %) were used as the sputtering and the reactive gases. Sputtering chamber is made up of sintered hard glass, which can observe the whole sputtering process directly. DC-sputtering system provided with circulation of cooling water The circulation of cooling water is carried out by water tank and pump in the system, thus guarantees the requirement of water temperature for normal operation. There is no special requirement for the water resource in laboratory.

X-ray diffraction (XRD) is a very important experimental technique in revealing the crystal structure of bulk solid microstructure of thin films . X-ray diffraction device has been used with properties.
Source: Cu tube .
Wave length :1.5406A° .
Voltage : 40.0 (kV).
Current : 30.0 (mA).

Full-width at half –maxima(FWHM) data was analyzed by scherer s formula to determine average particle size .Scherers equation is given by[ ]

…………………..(1-2)

Where λ is the X-ray wavelength ,β is the peak width and θ is the bragg s angle.

4-Results and discussion:

The XRD pattern in fig-2-reveal that the TiO_2 films deposited at sputtering pressure(1.8 pa) are amorphous. This is attributed to decrease deposition rate , hence decrease crystalline size. while Fig.(3) shows the XRD pattern at increasing sputtering pressure to 2.8 pa the structure of films become crystalline with rutile phase .This is attributed to increasing rate of deposition . Fig.(4) sputtering pressure(3.8pa) of TiO_2 films are amorphous, that due to collisions between ions and gas atoms the next from the target .and when we increase sputtering pressure(4.3 pa) lead to get the anatase phase as showen in fig. (5).this agrees with researchers H. Toku *et al*.[12] and Ab. Benyoucef *et al*.[13]. The fig. (6) shows formation anatase phase orientation (101) to percentage (25%) and agrees with researchers Yuh-Fan Su el al[14]. The fig. (7) shows that the grain size increase with increasing sputtering pressure which agree with Wei Zhou [15]. The fig. (8) shows the grain size as a function of partial pressure. The maximum value get at percentage (25%) this due to the increasing percentage which lead to increase energy gap ,hence formation anatase phase and this agree with M. Stamate and I. Vascan[16].

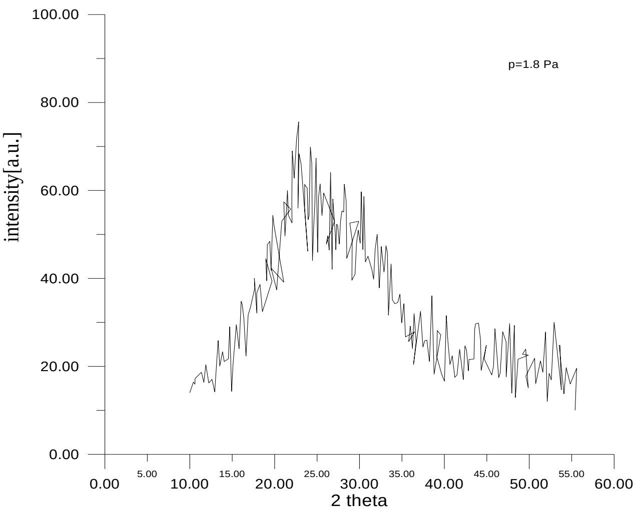

Fig. (2) XRD spectra of TiO_2 deposited at sputtering pressure(1.8 pa) andO_2/Ar percentage (5%).

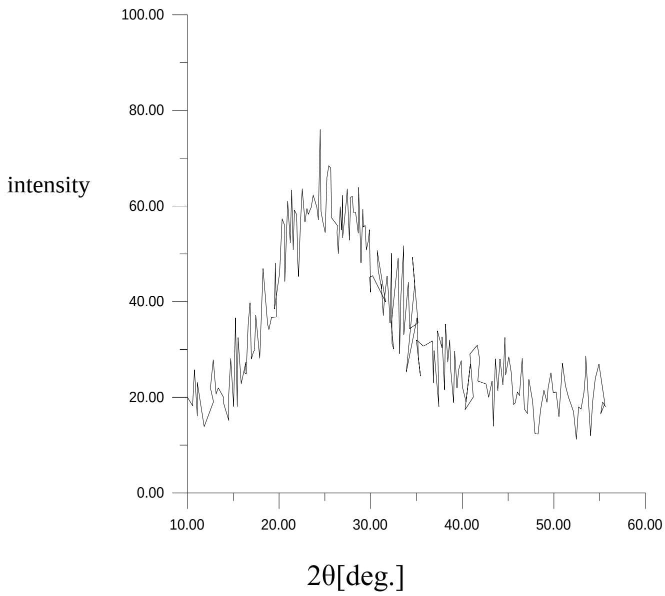

Fig. (4) XRD spectra of TiO_2 deposited at sputtering pressure(3.8 pa) andO_2/Ar percentage (5%).

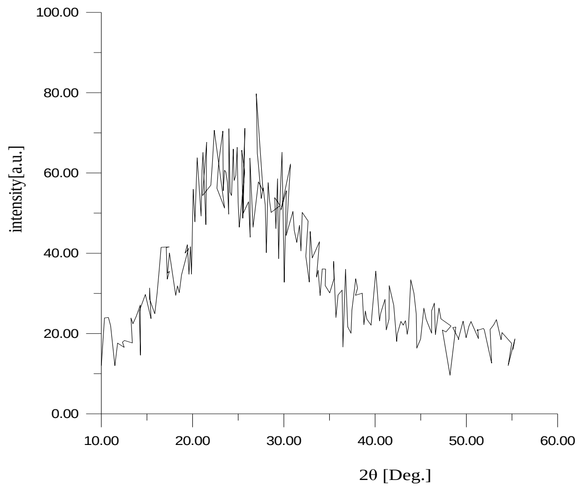

Fig. (3) XRD spectra of TiO_2 deposited at sputtering pressure(2.8 pa) andO_2/Ar percentage (5%).

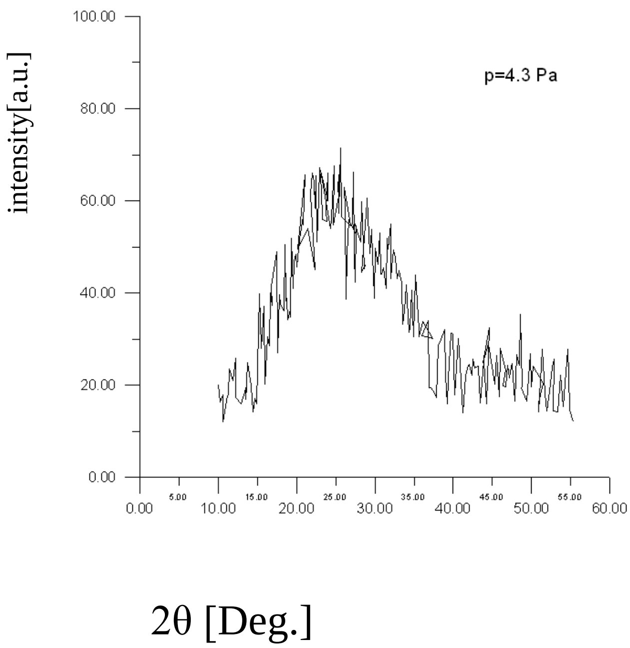

Fig. (5) XRD spectra of TiO_2 deposited at sputtering pressure(4.3 pa) andO_2/Ar percentage (5%).

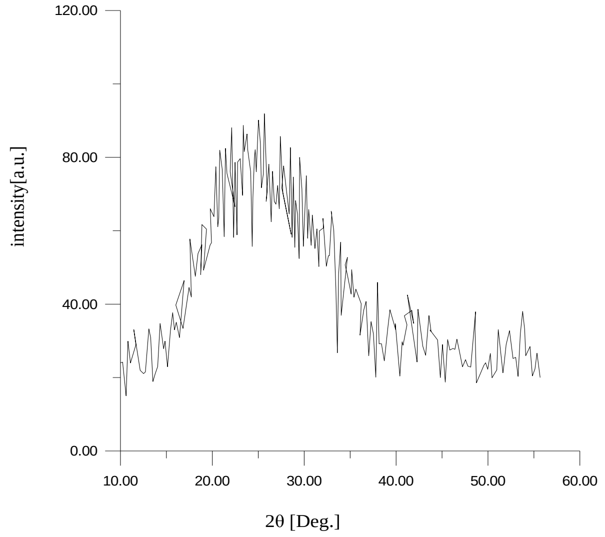

Figure (6) XRD spectra of TiO_2 deposited at sputtering pressure(2.8 pa) andO_2/Ar percentage (25%).

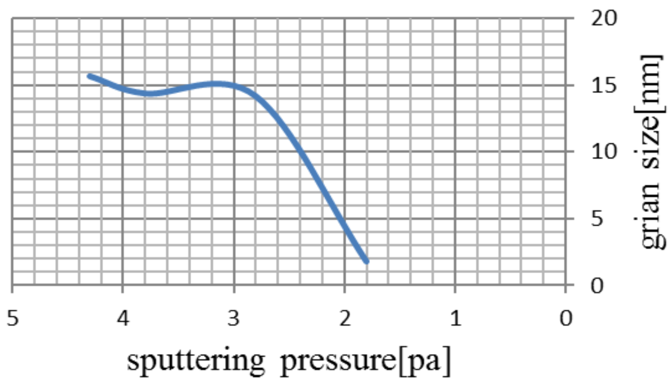

Figure (7) grain size as a function of sputtering pressure at O_2/Ar percentage (5%).

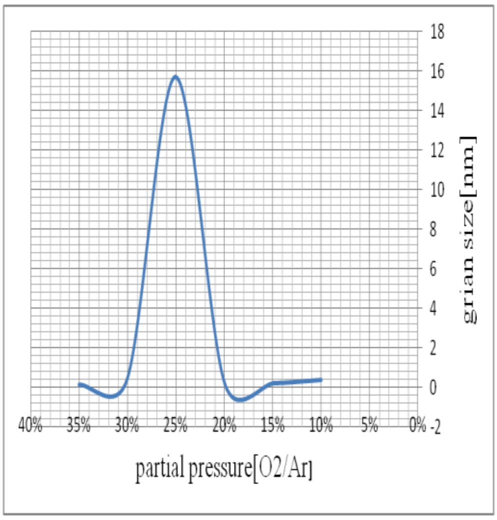

Figure (8) size as a function of partial pressure [O_2/Ar]at sputtering pressure (2.8 pa).

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