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Effect of Ion Beam Bombarding on Stress in TiO_2 Thin Films

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Abstract

TiO_2 films were fabricated on Si substrate by using electron-beam gun evaporation. Influence of deposition rate, deposition temperature and ion beam bombarding on stress in TiO_2 films was studied by AFM. The results show that deposition temperature of 423K and deposition rate of 0.2nm/s, the average stress in TiO_2 thin films is less than 48.2MPa. The average stress decreases to compressive stress of 16.7MPa from tensile stress of 72.9MPa by the ion beam energy of 113eV and bombarding time of 300s.

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Keywords: Thin film; TiO_2; Ion beam bombarding; Tensile stress; Compress stress

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1. Introduction

TiO2 films have not only low absorption in the visible and near-infrared spectral regions but also high refractive index and excellent chemical stability [1]. It is an importance optical film material. TiO_2 films have been produced by several methods such as sol-gel method [3], magnetron sputtering [4, 5], Ion-beam assisted deposit technique [6], and so on. There are different optics and stress by different deposit techniques. There are big stresses in TiO_2 films by electron-beam evaporation. The spectrum often changes because the stress produces deformation, so optical and mechanical performance of filters reduced.

In this study, Influence of deposition rate, deposition temperature and ion beam bombarding on stress in TiO_2 films was studied in order to solve crack and break of TiO_2 films. It can provided importance data for optimum design, produce and use of optical thin film filters.

2. Experimental Procedure

2.1. Sample preparation

TiO_2 films were prepared by Denton Ingrety-39 optical evaporation system in Fig.1. The equipment was equipped Denton Vacuum CC-105 cold cathode ion source, Leybold-inficon IC/5 quartz crystal rate controller and electron-beam evaporation source.

TiO_2 films were deposited on 350μm thick, 50.8mm diameter Si circular substrate (<100>) using black grains Ti_2O_3 of 99.99% purity with chamber pressures of approximately 8.0×10^{-3} Pa. Time of ion beam bombarding substrate before deposition was 120s. Deposition rate was 0.2-1.5nm/s, deposition temperature was 323-475K, and Thickness of the films was 555-600nm.

TiO_2 films were bombarded by Denton Vacuum CC-105 cold cathode ion source. In order to obtain bombardment uniformity, workpiece is rotation, at the same time sample frame is rotation. The bombardment was carried at temperature of 312K and Ar flow rate of 22sccm, bombarding time was 300s, work vacuum was superior to 7.9×10^{-2}Pa, background vacuum was superior to 4.0×10^{-2}Pa, and the rotate speed of sample was 15RPM.

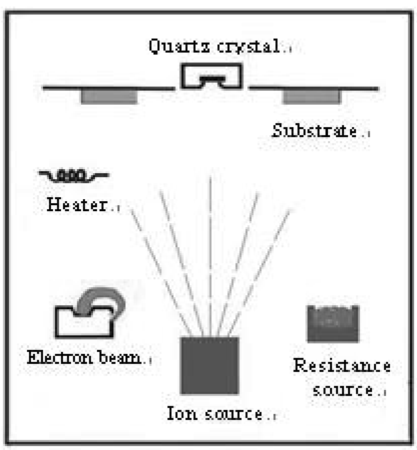


Fig.1 Sketch of Denton Ingrety-39 optical evaporation system

2.2. Sample characterization

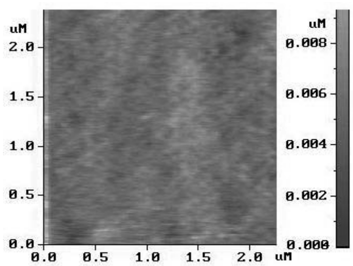
Morphologies of the TiO_2 films were observed by atomic force microscope (AFM-SPM9500J3, Japan). The apparatus is contact pattern. curvature radius of needlepoint is 20nm, detect size 30μm, plane maximum scan range 125μm×125μm. It can observe the disfigurements, rearranges, Morphologies of sorbent, and so on.

Stresses in TiO_2 films was analyzed by Film Stress Measurement (FSM-BGS6431, China)[7]. BGS643 Film Stress Measurement determines stress by measuring the curvature change of pre- and post-deposition of the film. This difference in curvature is used to calculate stress by way of Stoney's equation, which relates the biaxial modulus of the substrate, thickness of the film and substrate, and the radius of curvatures of pre-and post-process. Curvature is measured by directing a laser at a surface with a known spatial angle. The reflected beam strikes a position sensitive photodiode. The geometry of the film is recorded by scanning the surface.

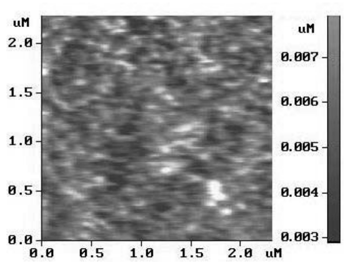
3. Results and discussion

3.1. AFM analysis

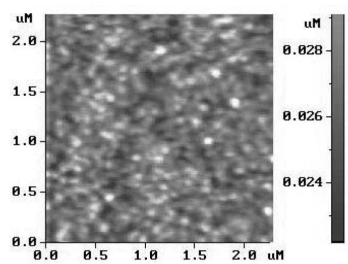
Fig.2a–c show AFM micrographs of TiO2 films at different deposition rates. It should be noted that the TiO_2 films grew with island model besides sample (a). Different deposition rates showed the different rough surface. It can be attributed to the scattering of incident light at the rough surface. TiO_2 films surface were flat and compact at little deposition rates, the average stress in TiO_2 films was less than 48.2MPa. Deposition rate of 0.5nm/s, the film surface had some small grooves, the surface roughness increased obviously, and the average stress in TiO_2 films is 80.2MPa. Deposition rate of 0.7nm/s, small grooves disappeared, the small island increased, TiO_2 films surface become more compact, and the average stress in TiO_2 films was only 14.6MPa.



(a) 0.2nm/s, 423K



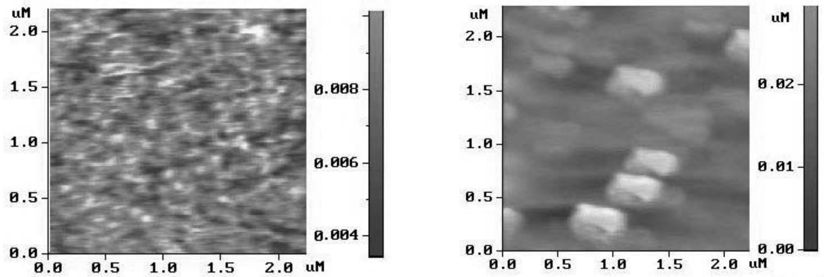
(b) 0.5nm/s, 423K



(c) 0.7nm/s, 423K

Fig.2 AFM images of TiO_2 films at different deposition rates

Fig. 2a and Fig. 3a–b show AFM micrographs of TiO_2 films at different deposition temperatures. Films deposited at low temperature had low stress value, 45.8MPa, the film surface were relatively flat and had some small grooves. Films deposited at 423K had big stress value relatively, 80.2MPa, the film surface was accidented, and the small island density decreased. Films deposited at 473K had bigger stress value, 95.4MPa, there were some cavities and interspaces between the small island, and the small island become bigger and cylinder.

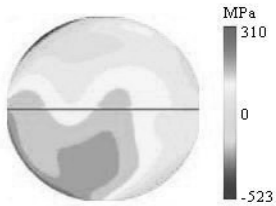


(a) 0.5nm/s, 323K (b) 0.5nm/s, 473K

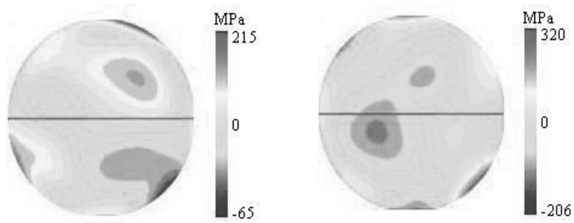
Fig.3 AFM images of TiO_2 films at different deposition temperatures

3.2. Stress analysis

Fig. 4 shows the stress distributions in TiO_2 films at different deposition rates. From the figure it was observed that the stress distribution uniformity in TiO_2 films is not good, such as Fig. 6a, the maximum tensile stress is 310.3MPa, the minimum compressive stress is 522.7MPa.



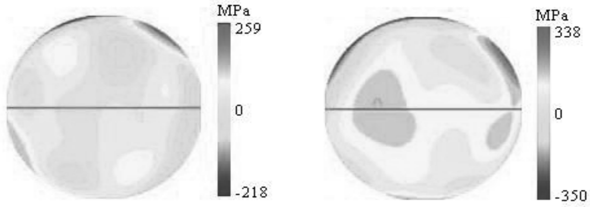
(a) 0.2nm/s, 423K



(b) 0.5nm/s, 423K (c) 0.7nm/s, 423K

Fig.4 Stress distribution in TiO_2 thin films at different deposition rates

Fig. 4b and Fig. 5a–b show the stress distributions in TiO_2 films at different deposition temperatures. From the figure it was observed that the stress distribution uniformity in TiO_2 films is good at deposition temperature of 323K, the average stress value is 45.8MPa. The average stress value in TiO_2 films is 80.2MPa When deposition temperature up to 403K. The average stress value in TiO_2 films is 95.4MPa When deposition temperature up to 473K. It shows that deposition temperature is obvious effect on thin film stress.



(a) 0.5nm/s, 323K (b) 0.5nm/s, 473K

Fig.5 Stress distribution in TiO_2 thin films at different deposition temperatures

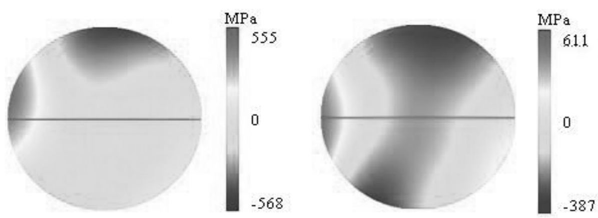
Bombardment time is same, 300s.The stress in TiO_2 thin films increased from 35.8MPa to 67.5MPa by the ion beam energy of 82eV. The stress in TiO_2 thin films changed from 72.9MPa to -16.7MPa by the ion beam energy of 113eV. The stress in TiO_2 thin films changed from 2.1MPa to -19.9MPa by the ion beam energy of 131eV. The results have been collected in Tab.1.

Table 1 Stress change in titanium TiO_2 films with different the ion beam energy

Ion beam energy (eV)	Average stress（MPa）	
	Without bombarding	bombarding
82	35.8	67.5
113	72.9	-16.7
131	2.1	-19.9

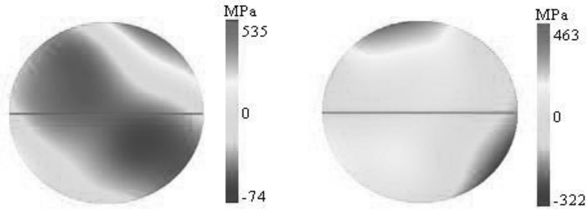
“-”is the compressive stress

When Bombardment time is same, from the analysis above it was known that the stress value in TiO_2 thin films changed by less ion beam energy, but the stress in TiO_2 thin films changed from tensile stress to compressive stress by higher ion beam energy. It indicates that the stress in TiO_2 thin films can be changed by ion beam bombarding.



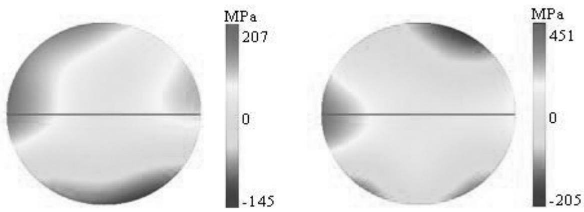
Without bombarding Bombarding

（a）Ion beam energy: 82eV, bombarding time: 300s



Without bombarding Bombarding

（b）Ion beam energy: 113eV, bombarding time: 300s



Without bombarding Bombarding

（c）Ion beam energy: 131eV, bombarding time: 300s

Fig.6 Stress distribution in TiO2 thin films at different ion beam energy

Fig.6 shows stress distribution of TiO_2 thin films at different ion beam energy. The stress change field is in the middle of substrate by the ion beam energy of 82eV and bombarding time of 300s, the average stress increase, stress uniformity become badly. The stress change field extends the whole substrate by the ion beam energy of 113eV and bombarding time of 300s, the stress in TiO_2 thin films changed from tensile stress to compressive stress, stress uniformity become well. The change field is in the edge of substrate by the ion beam energy of 131eV and bombarding time of 300s, the stress in TiO_2 thin films changed from tensile stress to compressive stress, stress uniformity become badly.

4. Conclusions

TiO_2 films have big tensile stress by using electron-beam gun evaporation. Influence of deposition rate, deposition temperature and ion beam bombarding on stress in TiO_2 films was studied by AFM and XRD. The results show that deposition temperature of 323K and deposition rate of 0.2nm/s, the average stress in TiO_2 thin films is less than 48.2MPa. The average stress decreases to compressive stress of 16.7MPa from tensile stress of 72.9MPa by the ion beam energy of 113eV and bombarding time of 300s. The microstructure change of TiO_2 films is main factors of stress development.

Acknowledgements

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