



Preparation, formation mechanism and mechanical properties of multilayered TiO₂–organic nanocomposite film

Wen-ning MU¹, Yu-chun ZHAI¹, Shuang-zhi SHI²

1. School of Resources and Materials, Northeastern University at Qinhuangdao, Qinhuangdao 066004, China;

2. Qinhuangdao Shougang Plate Co., Ltd., Qinhuangdao 066003, China

Received 14 May 2014; accepted 5 August 2014

Abstract: TiO₂–organic multilayered nanocomposite films were deposited on a self-assembled monolayer-coated silicon substrate based on layer-by-layer technique and chemical bath deposition method by a hydrolysis of TiCl₄ in an acid aqueous solution. The chemical compositions, surface morphologies and mechanical properties of the films were investigated by X-ray photoelectron spectrometer (XPS), scanning electron microscopy (SEM) and nanoindentation depth-sensing technique, respectively. The results indicate that the major chemical compositions of the films are Ti and O. The principal mechanism for the nucleation and growth of the films is homogeneous nucleation, and the layer number of films has great influence on the surface morphology and roughness of the films. In addition, mechanical nanoindentation testing presents a significant increase in hardness and fracture toughness of titanium dioxide multilayered films compared with single-layer titanium dioxide thin film.

Key words: titanium dioxide; multilayered film; self-assembly monolayer; formation mechanism; mechanical properties

1 Introduction

As we all know, nacre is a typical natural nanostructured material that has extraordinary mechanical strength and toughness. These mechanical performances are generally believed to originate from a highly regular “brick-and-mortar” structure composed of alternating layers of a soft organic biopolymer and a hard inorganic material [1–7].

Numerous researchers have focused on the fabrication of organic–inorganic nanocomposites with structural design principles found in nacre [8], wherein the inorganic layers are formed via solution-based approaches, such as electrophoresis [9], dip-coating [10], sol–gel [11], deposition of mineral or clay platelets [12–15], while the organic component is usually deposited by electrostatic adsorption of oppositely charged polyelectrolytes based on layer-by-layer self-assembly technique [16]. However, there are still some problems on the preparation or properties of synthetic materials that are not easy to be solved. On one hand, the artificial materials containing laminated

structure of nacre are hard to be synthesized, because it is absolutely impossible to achieve complete replication of nacre-like structure. On the other hand, although the mechanical properties of artificial materials have been found to be enhanced prominently compared with their substrates such as silica, glass and metal, there is still a significant gap from the excellent performance of nacre.

At present, chemical bath deposition methods have shown an advantage in bioinspired synthesis of inorganic films. This method offers a low operating temperature in the range of 30–100 °C in aqueous solutions, which is similar to the biomineralization process of natural organic–inorganic nanocomposites [17]. Currently, chemical bath deposition has been used to deposit various metal oxides on the self-assembled monolayer in aqueous solutions by simulating the bioinspired processing [18]. This application is mainly attributed to some advantages of this method including less investment of equipment, low temperature and more suitability to the substrates. In recent years, large scientific interest has been paid to using titanium dioxide as inorganic filler to prepare polymer matrix materials [19,20]. This originates from the wide use of

titanium dioxide materials as microelectronics [21], photocatalysis [22,23], solar cell [24,25], anti-fogging [26] and self-cleaning coatings [27,28], etc.

In our group, a single-layer titanium dioxide thin film was obtained via titanium dioxide deposition from aqueous titanate solution on a self-assembled monolayer-coated silicon substrate [29]. Although the deposition solution chemistry and physical properties of single-layer film have been investigated, the preparation, deposition mechanism and mechanical properties of multilayered TiO₂-organic films are not mentioned. Thus, in this work, an alternate deposition method was used to fabricate TiO₂-organic multilayered nanocomposite films with nanolaminar nacre-like structures. The chemical compositions, surface morphologies and mechanical properties of the thin films were detected by X-ray photoelectron spectrometer (XPS), scanning electron microscopy (SEM) and nanoindentation depth-sensing technique, respectively. Through this research, the formation mechanism of multilayer films was revealed, and the thin films with excellent mechanical properties were prepared. It would provide a theoretical basis and technical support for the preparation of a new type of biomimetic materials.

2 Experimental

2.1 Fabrication of self-assembled monolayers (SAMS)

P-type (100) single-crystal silicon wafer that was cut into 2 cm × 2 cm was used as the substrate for SAMS preparation via layer-by-layer self-assembly technique. Firstly, the silicon substrate was cleaned with organic solvents and heated up to 80 °C for 40 min in piranha solution (3:1, volume ratio of mixed solution of concentrated H₂SO₄ to 30% H₂O₂). A negatively charged hydrophilic Si—OH layer was formed on the substrate surface, which was necessary for SAMS fabrication [30]. Then, the silicon substrate was washed with deionized water and dried spontaneously.

Poly (sodium 4-styrenesulfonate) (PSS, $M_w=70000$), poly (allylamine hydrochloride) (PAH, $M_w=70000$) and poly (ethyleneimine) (PEI, $M_w=70000$) were purchased from Sigma-Aldrich. Three polymers were dissolved in deionized water to prepare PSS solution of 4 g/L, PAH solution of 2 g/L and PEI solution of 1.25 g/L, and the solution pH was adjusted to 3.5, 3.5 and 9.0 by adding hydrochloric acid, respectively. The fabrication of self-assembled monolayers was carried out as described in Ref. [29]. Namely, negatively charged silicon wafer was alternately immersed into the polycation (PEI or PAH) and polyanion (PSS) solutions for 20 min. After deposition of each layer, the substrate was cleaned with deionized water in order to remove unbound or weakly bound polyelectrolytes and to prevent the cross-

contamination of the solutions. After the second layer, only the solutions of PAH and PSS were used, and this sequence was repeated four times. Finally, multilayered film composed of (PEI/PSS) (PAH/PSS)₄, corresponding to a total thickness of 10 nm, was assembled.

2.2 Deposition of multilayered TiO₂ thin films

Reagent grade titanium tetrachloride (TiCl₄), hydrogen peroxide (H₂O₂), and hydrochloric acid (HCl), were used. The titanium tetrachloride was dissolved in 30% hydrogen peroxide to prepare fresh precursor solutions. The titanium concentration and pH of precursor solutions were adjusted by adding deionized water and concentrated hydrochloric acid to yield a suitable condition for titanium dioxide thin film deposition. The samples with SAMS were placed in the running solution and kept for different time at the constant temperature of 60 °C. During the fabrication process of thin film, a peristaltic pump was used to drive the reaction solution cycling at the flow rate of 20 mL/min. After reaching the desired time, the samples were ultrasonically cleaned completely with deionized water and then dried in air. Multilayered structure was obtained via sequential deposition of self-assembled monolayer and titanium precursor solution. In order to obtain a relatively thick titanium dioxide layer, a multi-step solution deposition was used on the same sample. The procedures for the fabrication of TiO₂-organic multilayered thin films are shown in Fig. 1.

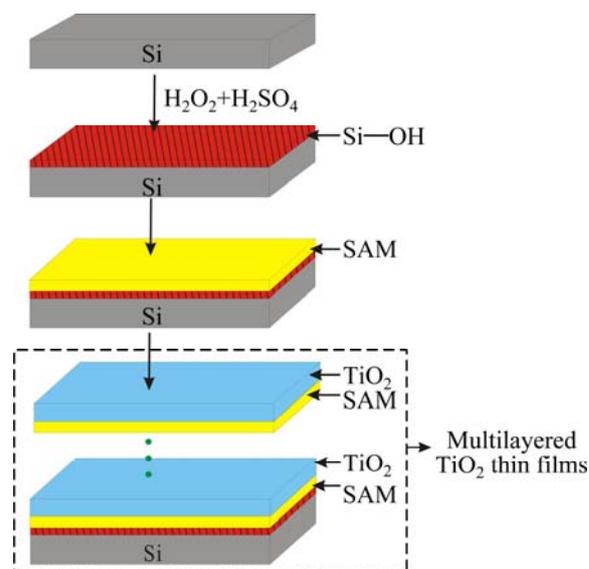


Fig. 1 Procedures for deposition of titanium dioxide multilayered thin films

2.3 Characterization of multilayered thin films

The surface morphologies of the TiO₂-organic multilayered films were observed using a scanning electron microscope (SEM, Leo Supra 35, Germany).

The elemental composition of multilayered films was determined by X-ray photoelectron spectrometer (XPS, ESCALAB 250, USA). XPS measurements were carried out with Al K_{α} irradiation (photon energy is 1486.6 eV) at a pass energy of 50 eV. Nanoindentation tests were performed to evaluate the hardness and fracture toughness of the thin films. The indentations were made by applying a load of 10 g for the films, in order to reach the maximum penetration depth. For each specimen, three indentations were made at different locations.

3 Results and discussion

3.1 Formation of multilayered structure

Many instances of thin films deposited on self-assembled monolayer have been reported, but the exact role of the self-assembled monolayer is still unclear. In our experiments, the self-assembled monolayer was indispensable for the formation of titanium dioxide thin film. In other words, no film growth was observed on a bare silicon substrate under the same conditions where thin film could form on the sulfonated self-assembled monolayer-coated substrate. The $-\text{SO}_3\text{H}$ group in the self-assembled monolayer provides a high degree of surface acidity and net negative charge, so the polycations of titanium salt hydrolysis could be adsorbed on the self-assembled monolayer surface through electrostatic force. After the self-assembled monolayer has been completely covered, attractive van der Waals force will continue to exist between the deposited particles and those in the solutions.

After several cycles of organic layer deposition and solution deposition of titanium dioxide, a multilayered structure could be obtained on a self-assembled monolayer-coated silicon substrate. A secondary electron image of double-layered TiO_2 -organic thin film obtained by Leo Supra 35 SEM is presented in Fig. 2. It can be seen that organic self-assembled monolayer appears as bright layer with a thickness of 15–20 nm, while

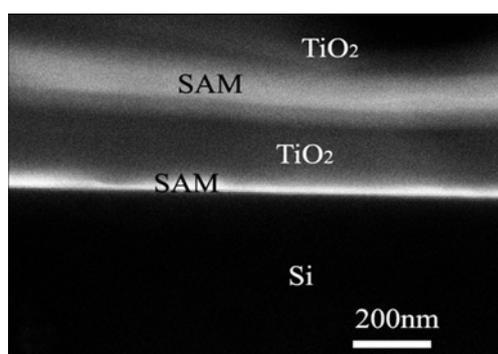


Fig. 2 SEM image of double-layered TiO_2 -organic thin film structure on self-assembled monolayer-coated silicon substrate

titanium oxide layer appears as a dark region with a thickness of about 200 nm. It is fully demonstrated that the layers of both organic material and titanium oxide were obtained successfully, and the interface region is much clearer than the film prepared in Ref. [31].

3.2 Deposition mechanism of TiO_2 thin film

In an aqueous solution, the thin film formation can be achieved through two nucleation mechanisms: heterogeneous nucleation at the interface between the substrate and solution, and homogeneous nucleation by forming stable nuclei in a supersaturated solution. These mechanisms have been considered for the deposition of titanium dioxide film by immersing a self-assembled monolayer-coated substrate in aqueous solution of inorganic titanium salts. In the former mechanism, dissolved ionic species may attach to the self-assembled monolayer surface through an ion-by-ion growth mechanism. In the latter mechanism, titanium dioxide particles formed by homogeneous nucleation can form colloidal particles in solution and be attracted to self-assembled monolayer surface due to electrostatic interactions.

The temporal growth of titanium dioxide film on the self-assembled monolayer-coated substrate in this work is presented in Fig. 3. Figure 3(a) shows titanium dioxide nanoparticles of 10–20 nm in size formed on the surface of the self-assembled monolayer. Once these nanoparticles are grown or formed on the surface, they would adsorb more nanoparticles, thereby leading to the formation of isolated islands (Fig. 3(b)). These islands will further grow up and connect to their neighboring islands, forming interconnected titanium dioxide islands (Fig. 3(c)). As deposition goes on, the island structures continue to grow and spread to the entire surface of self-assembled monolayer, and several hours later, a continuous titanium dioxide film was produced on the surface, as shown in Fig. 3(d). Based on the analysis above, it is believed that the mechanism of homogeneous nucleation is responsible for the titanium dioxide film formation with our current processing settings.

3.3 Surface characterization of TiO_2 multilayered films

XPS measurements were performed to identify elemental composition of titanium dioxide multilayered films deposited on self-assembled monolayer-coated silicon substrate, and the results are presented in Figs. 4(a)–(c). From Fig. 4(a), the signals of Ti, O, C, S, N and Si can be seen clearly. The peaks detected at 168.83, 287.86 and 399.9 eV are ascribed to the S atoms in $-\text{SO}_3\text{H}$ groups, the C atoms in $-\text{CH}_2$ groups and the N atoms in $-\text{NH}_2$ groups, respectively. The absence of Si signals at 101.55 (Si p) and 150.44 eV (Si 2s)

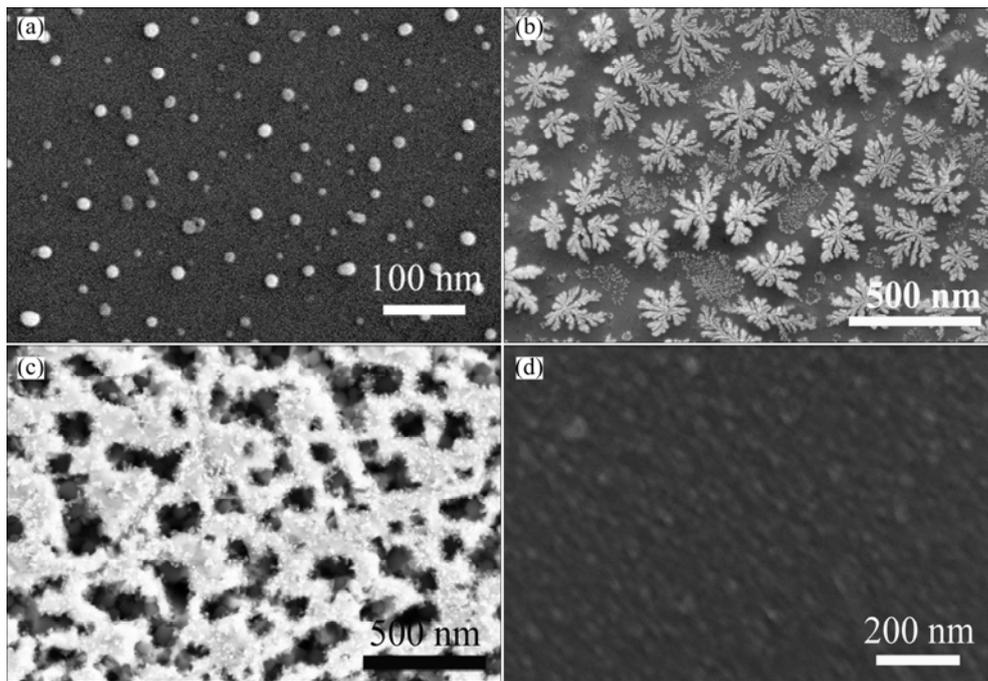


Fig. 3 Plan-view SEM images showing temporal growth of titanium dioxide films on self-assembled monolayer-coated silicon: (a) Nanoparticles (10–20 nm in size) at very early stage; (b) Island structures made of nanoparticles at intermediate stage; (c) Islands gradually connected together indicating growth of titanium dioxide islands; (d) Continuous thin film formed at final stage

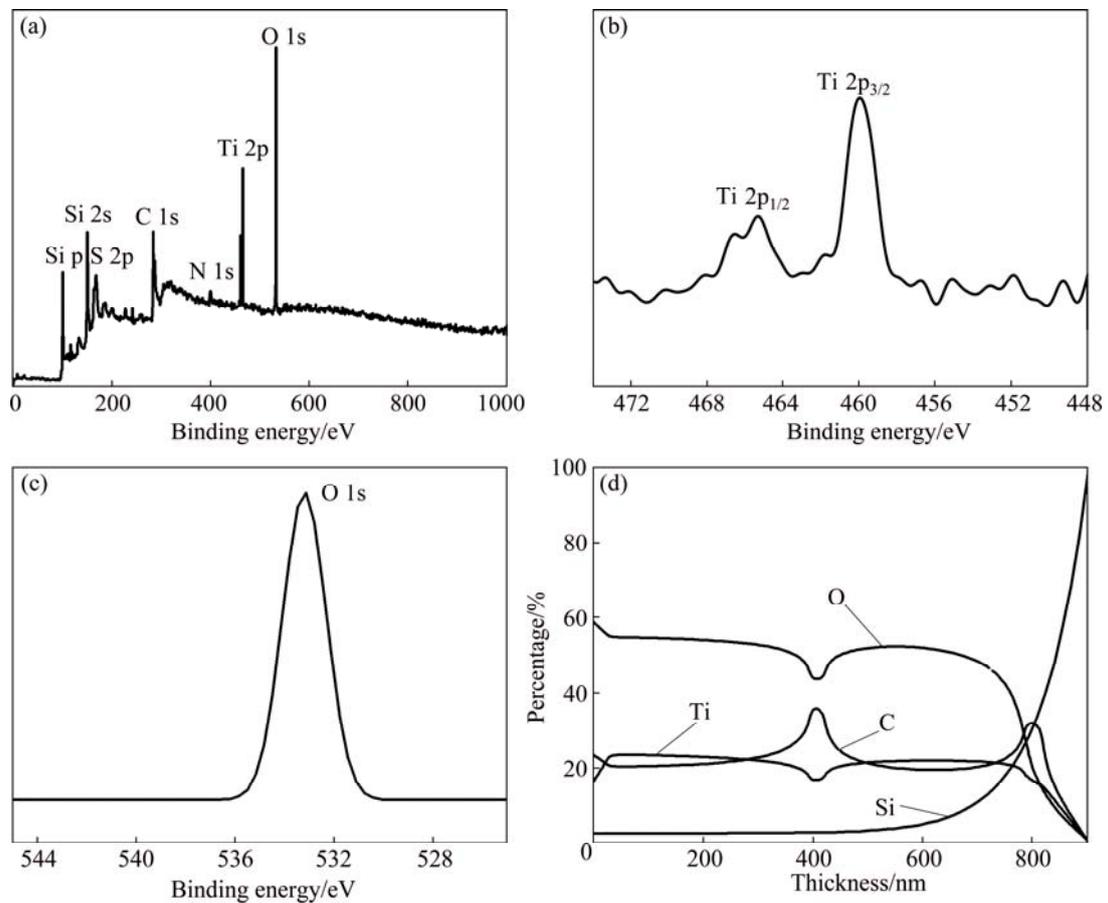


Fig. 4 XPS spectra of titanium dioxide multilayered films deposited on self-assembled monolayer-coated silicon substrate (a), Ti 2p of titanium dioxide multilayered films (b), O 1s of titanium dioxide multilayered films (c) and film element distribution in depth (d)

indicates the silicon substrate under the as-deposited film. The Ti 2p peaks are located at 461.88 eV (Ti 2p_{2/3}) and 465.63 eV (Ti 2p_{1/2}), while the O 1s peak is located at 534.46 eV, as seen in Figs. 4(b) and (c), which demonstrates the yield of titanium dioxide in the multilayered composite films and the results are identical to Ref. [32]. The composition of thin films can be analyzed further through XPS spectrum, and the relationship curve between elemental concentration and film depth is shown in Fig. 4(d). The results reveal that the structure of multilayer films is alternating deposition of TiO₂ and PE. The mole ratio of oxygen to titanium in thin films is somewhat higher than the corresponding ratio of 2:1 of stoichiometric TiO₂ because the grown films contain some amount of water [33].

The surface morphologies of titanium dioxide multilayered films deposited on a self-assembled monolayer-coated silicon substrate were characterized by SEM, and the results are presented in Fig. 5. The smoothness and roughness of titanium dioxide film surface vary constantly with the increase of the layer number. The single-layer titanium dioxide film surface is flat, dense and continuous, as seen in Fig. 5(a). With the increase of the layer number, the grain sizes on the film surface vary in a wide range, and the thin film presents obvious embossment, also the smoothness of thin films is reduced, which can be observed from Figs. 5(b) and (c). Figure 5(d) shows that the new platforms begin to grow on the thin film surface, when the layer number reaches 10. In addition, the film layer number has a great influence on the surface roughness of titanium dioxide

thin films. From Fig. 6, it can be seen that the surface roughness of titanium dioxide thin films increases from 2.76 nm to 6.99 nm when the layer number increases from 2 to 8. The increase of film layers would lead to many small fluctuations on the film surface overlapped constantly during the deposition of titanium dioxide thin films, and form large bulges, which causes the uneven thickness and large undulating of film surface, so eventually resulting in the increase of film roughness.

3.4 Mechanical properties

In order to evaluate the mechanical properties of the titanium dioxide single layer and multilayered samples, nanoindentation experiments were performed on deposited titanium dioxide thin films that have different layer numbers at an indentation load of 10 g. According to the information provided by the indentation crack, the hardness and fracture toughness values of the thin films were calculated. Figures 7(a) and (b) respectively show the hardness and fracture toughness of the titanium dioxide thin films containing different layers prepared at a 10 mmol/L titanium precursor solution on self-assembled monolayer-coated silicon at 60 °C for 2 h. It can be seen that the hardness and fracture toughness significantly increase with the increase of layer number. When layer number of thin film is 8, the hardness can be up to 11.77 GPa, exhibiting an increase of 31.5% and 13.6% respectively by comparing with silicon substrate and single-layer titanium dioxide film, as shown in Fig. 7(a). While the fracture toughness increases from

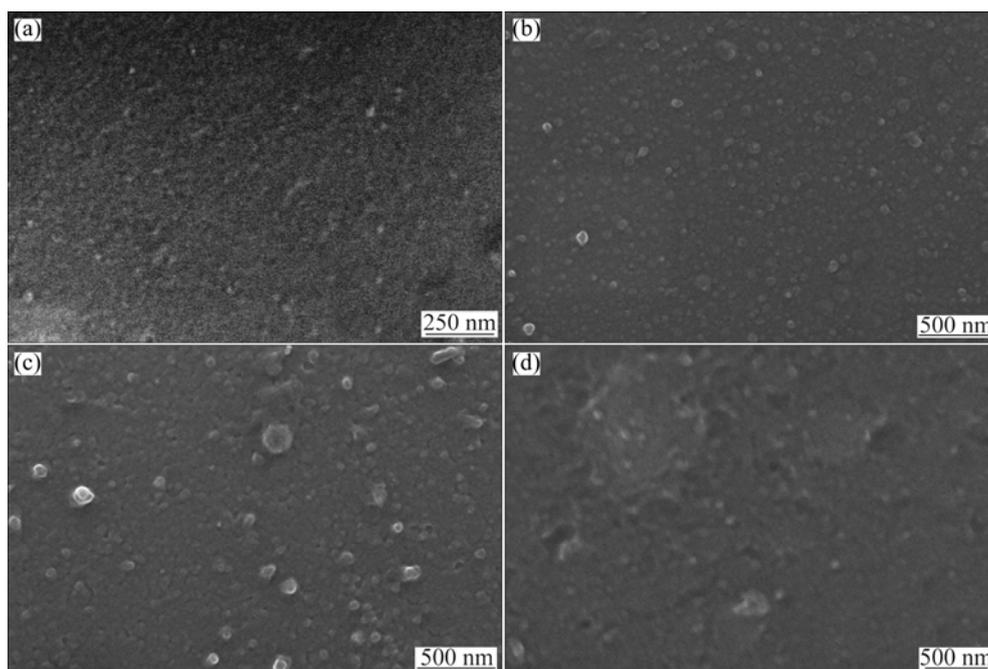


Fig. 5 Surface morphologies of titanium dioxide multilayered films deposited on self-assembled monolayer-coated silicon substrate: (a) 1 layer; (b) 5 layers; (c) 8 layers; (d) 10 layers

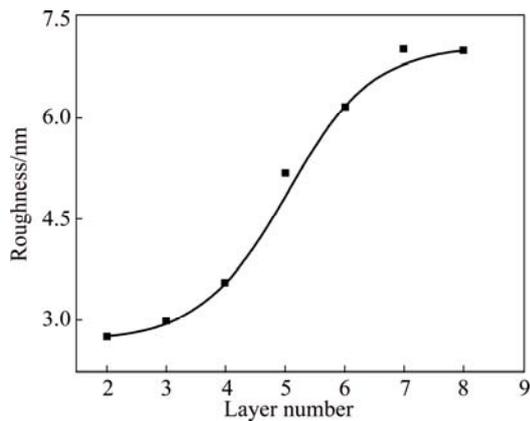


Fig. 6 Relationship between thin film roughness and layer number

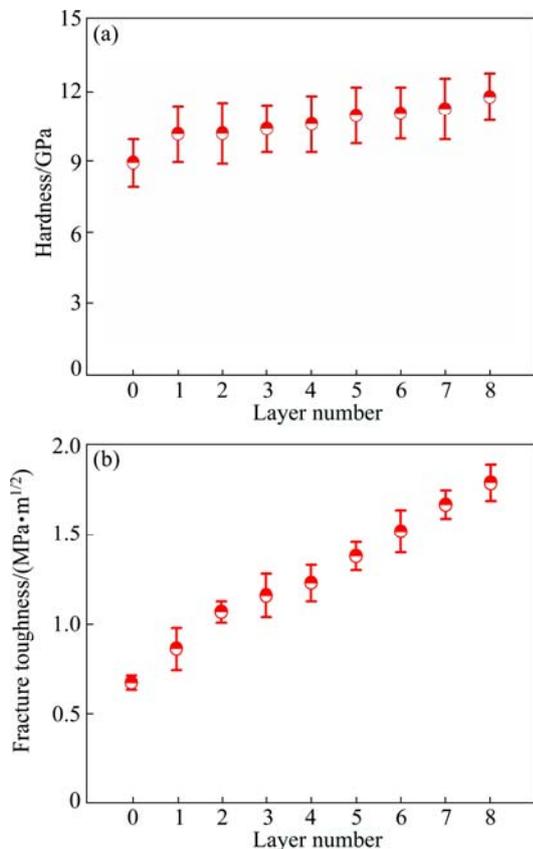


Fig. 7 Mechanical properties of titanium dioxide thin films containing different layer numbers: (a) Hardness; (b) Fracture toughness

0.86 MPa·m^{1/2} in titanium dioxide single layer film to 1.79 MPa·m^{1/2} in titanium dioxide of films with 8 layers, as seen from Fig. 7(b).

Monolayer structure is composed of amorphous nanoparticles through weak van der Waals force. Multilayer films consist of two kinds of hard and soft materials, wherein the inorganic phase provides excellent plasticity for the film, and the organic phase provides outstanding toughness for the film. Thus, the plastic

behavior of the film can be improved effectively. Overall, TiO₂–organic multilayered nanocomposite films exhibit an enhancement in mechanical properties relative to titanium dioxide single layer film.

4 Conclusions

1) TiO₂–organic multilayered nanocomposite film was grown on self-assembled monolayer-coated silicon substrates based on layer-by-layer technique and chemical bath deposition method by sequential solution deposition of titanium dioxide and organic self-assembled monolayer. The major chemical compositions of thin films determined by XPS are Ti and O, suggesting the yield of titanium dioxide in the multilayered composite films.

2) The principal mechanism for the nucleation and growth of titanium dioxide thin film is homogeneous nucleation, and the surface of single-layer film is flat, dense and continuous, the surface roughness of films increases considerably from 2.76 to 6.99 nm when the layer number increases from 2 to 8.

3) Mechanical nanoindentation testing shows a significant increase in hardness and fracture toughness of the multilayered structures compared with single-layer titanium dioxide thin film. When layer number of thin film is 8, the hardness and fracture toughness can be up to 11.77 GPa and 1.79 MPa·m^{1/2}, respectively.

Acknowledgment

The authors wish to thank Institute of Metal Research, Chinese Academy of Sciences for the access to the XPS equipments.

References

- [1] JACKSON A P, VINCENT J F V, TURNER R M. The mechanical design of nacre [J]. *Proceedings of the Royal Society of London Series B*, 1988, 234: 415–440.
- [2] GAO H, JI B, JÄGER I L, ARZT E, FRATZL P. Materials become insensitive to flaws at nanoscale: Lessons from nature [J]. *Proceedings of the National Academy of Sciences of the United States of America*, 2003, 100(10): 5597–5600.
- [3] FRATZL P, GUPTA H S, PASCHALIS E P, ROSCHGER P. Structure and mechanical quality of the collagen-mineral nano-composite in bone [J]. *Journal of Materials Chemistry*, 2004, 14: 2115–2123.
- [4] FU G, QIU S R, ORME C A, MORSE D E, de YOREO J J. Acceleration of calcite kinetics by abalone nacre proteins [J]. *Advanced Materials*, 2005, 17(22): 2678–2683.
- [5] JI B, GAO H. Mechanical properties of nanostructure of biological materials [J]. *Journal of the Mechanics and Physics of Solids*, 2004, 52(9): 1963–1990.
- [6] OKUMURA K, de GENNES P G. Why is nacre strong? Elastic theory and fracture mechanics for biocomposites with stratified structures [J]. *The European Physical Journal E*, 2001, 4: 121–127.
- [7] SMITH B L, SCHÄFFER T E, VIANI M, THOMPSON J B, FREDERICK N A, KINDT J, BELCHER A, STUCKY G D, MORSE D E, HANSMA P K. Molecular mechanistic origin of the toughness of natural adhesives, fibres and composites [J]. *Nature*,

- 1999, 399: 761–763.
- [8] SANCHEZ C, ARRIBART H, GUILLE M M G. Biomimetic and bioinspiration as tools for the design of innovative materials and systems [J]. *Nature Material*, 2005, 4: 277–288.
- [9] LONG B, WANG C A, LIN W, HUANG Y, SUN J. Polyacrylamide-clay nacre-like nanocomposites prepared by electrophoretic deposition [J]. *Composites Science and Technology*, 2007, 67(13): 2770–2774.
- [10] SELINGER A, WEISS P M, NGUYEN A, LU Y, ASSINK R A, GONG W, BRINKER C J. Continuous self-assembly of organic–inorganic nanocomposite coatings that mimic nacre [J]. *Nature*, 1998, 394: 256–260.
- [11] MAMMERI F, BOURHIS E L, ROZES L, SANCHEZ C. Mechanical properties of hybrid organic–inorganic materials [J]. *Journal of Materials Chemistry*, 2005, 15(35–36): 3787–3811.
- [12] AKSAY I A, TRAU M, MANNE S, HONMA I, YAO N, ZHOU L, FENTER P, EISENBERGER P M, GRUNER S M. Biomimetic pathways for assembling inorganic thin films [J]. *Science*, 1996, 273: 892–898.
- [13] TANG Z, KOTOV N A, MAGONOV S, OZTURK B. Nanostructured artificial nacre [J]. *Nature Material*, 2003, 2: 413–418.
- [14] KOTOV N A, HARASZTI T, TURI L, ZAVALA G, GEER R E, DÉKÁNY I, FENDLER J H. Mechanism of and defect formation in the self-assembly of polymeric polycation-montmorillonite ultrathin films [J]. *Journal of the American Chemical Society*, 1997, 119(29): 6821–6832.
- [15] FAN X, LOCKLIN J, YOUK J H, BLANTON W, XIA C, ADVINCULA R. Nanostructured sexithiophene/clay hybrid multilayers: A comparative structural and morphological characterization [J]. *Chemistry of Materials*, 2002, 14(5): 2184–2191.
- [16] DECHER G. Fuzzy nanoassemblies: Toward layered polymeric multicomposites [J]. *Science*, 1997, 277: 1232–1237.
- [17] de GUIRE M R, NIESEN T P, WOLFF J, SUPOTHINA S, BILL J, RÜHLE M. Synthesis of oxide and non-oxide inorganic materials at organic surfaces [J]. *Z Metallkd*, 1998, 89: 758–766.
- [18] GAO Y F, KOUMOTO K. Bio-inspired ceramic thin film processing: Present status and future perspectives [J]. *Crystal Growth & Design*, 2005, 5(5): 1983–2017.
- [19] RAHIMPOUR A, MADAENI S S, TAHERI A H, MANSOURPANAH Y. Coupling TiO₂ nanoparticles with UV irradiation for modification of polyethersulfone ultrafiltration membranes [J]. *Journal of Membrane Science*, 2008, 313: 158–169.
- [20] WU G, GAN S, CUI L, XU Y. Preparation and characterization of PES/TiO₂ composite membranes [J]. *Applied Surface Science*, 2008, 254(21): 7080–7086.
- [21] BURNS G P. Titanium dioxide dielectric films formed by rapid thermal oxidation [J]. *Journal of Applied Physics*, 1989, 65(5): 2095–2097.
- [22] LIN X, RONG F, JI X, FU D. Carbon-doped mesoporous TiO₂ film and its photocatalytic activity [J]. *Microporous and Mesoporous Materials*, 2011, 142(1): 276–281.
- [23] PAN J H, LEI Z, LEE W I, XIONG Z, WANG Q, ZHAO X S. Mesoporous TiO₂ photocatalytic films on stainless steel for water decontamination [J]. *Catalysis Science and Technology*, 2012, 2: 147–155.
- [24] KHAN M A, AKHTAR M S, YANG O B. Synthesis, characterization and application of sol–gel derived mesoporous TiO₂ nanoparticles for dye-sensitized solar cells [J]. *Solar Energy*, 2010, 84(12): 2195–2201.
- [25] NIKRAZ S, PHARES D J, WANG H. Mesoporous titania films prepared by flame stabilized on a rotating surface: Application in dye sensitized solar cells [J]. *Journal of Physical Chemistry C*, 2012, 116(9): 5342–5351.
- [26] MASUDA Y, KATO K. Anatase TiO₂ films crystallized on SnO₂: F substrates in an aqueous solution [J]. *Thin Solid Films*, 2008, 516(9): 2547–2552.
- [27] FATEH R, ISMAIL A A, DILLERT R, BAHNEMANN D W. Highly active crystalline mesoporous TiO₂ films coated onto polycarbonate substrates for self-cleaning applications [J]. *Journal of Physical Chemistry C*, 2011, 115(21): 10405–10411.
- [28] WANG J, LI H, ZUO C, WANG H. Thermal stability and optimal photoinduced hydrophilicity of mesoporous TiO₂ thin films [J]. *Journal of Physical Chemistry C*, 2012, 116(17): 9517–9525.
- [29] MU Wen-ning, ZHAI Yu-chun, SHI Shuang-zhi. Fabrication of titanium oxide films at low temperature from aqueous solution [J]. *Transactions Nonferrous Metals Society of China*, 2013, 23(5): 1390–1394.
- [30] SEU K J, PANDEY A P, HAQUE F, PROCTOR E A, RIBBE A E, HOVIS J S. Effect of surface treatment on diffusion and domain formation in supported lipid bilayers [J]. *Biophysical Journal*, 2007, 92(7): 2445–2550.
- [31] BURGHARD Z, TUCIC A, JEURGENS L P H, HOFFMANN R C, BILL J, ALDINGER F. Nanomechanical properties of bioinspired organic–inorganic composite films [J]. *Advanced Materials*, 2007, 19(7): 970–974.
- [32] HOFFMANN R C, BILL J. Relation between particle growth kinetic in solution and surface morphology of thin film: implications on the deposition of titania on polyethylene terephthalate [J]. *Thin Solid Film*, 2005, 478(1–2): 164–169.
- [33] FUCHS T M, HOFFMANN R C, NIESEN T P, BILL J, ALDINGER F. Deposition of titania thin films from aqueous solution by a continuous flow technique [J]. *Journal of Materials Chemistry*, 2002, 12: 1597–1601.

二氧化钛有机多层纳米复合薄膜的 制备、形成机理和力学性能

牟文宁¹, 翟玉春¹, 石双志²

1. 东北大学(秦皇岛分校) 资源与材料学院, 秦皇岛 066004; 2. 秦皇岛首钢板材有限公司, 秦皇岛 066003

摘要: 基于层层自组装技术和化学与沉积方法, 由四氯化钛在酸性水溶液中水解在含有自组装涂层的硅基底上沉积二氧化钛有机多层纳米复合薄膜。分别采用 X 射线光电子能谱(XPS)、扫描电子显微镜(SEM)和纳米压痕深度传感技术研究薄膜的化学组成、表面形貌和力学性能。结果表明, 薄膜的主要化学成分为 Ti 和 O, 成核和生长的主要机理是均相成核, 薄膜层数对薄膜的表面形貌和粗糙度影响很大。此外, 纳米压痕实验表明, 与单层的二氧化钛薄膜相比, 多层纳米二氧化钛薄膜的硬度和断裂韧性得到显著提高。

关键词: 二氧化钛; 多层薄膜; 自组装薄膜; 形成机理; 力学性能

(Edited by Wei-ping CHEN)