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Improvement of mechanical properties of glass substrates

Ismail Hakki Cengizhan Karbay[a], Refika Budakoglu[b], Esra Ozkan Zayim[a,c,*]

[a] Nano Science & Nano Technology M. Sc. Program, Istanbul Technical University, Maslak, Istanbul, Turkey
[b] SISECAM Glass Research Center, Is Kuleleri Kule No: 3, 4. Levent, Istanbul 34330, Turkey
[c] Istanbul Technical University, Faculty of Science and Letters, Physics Department, Istanbul 34469, Turkey

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ABSTRACT

This paper aims to enhance the mechanical and optical properties of glass substrates with thin films by the sol–gel method. TiO_2–SiO_2 binary system and Ta_2O_5 were deposited on glass substrates with high transparency. Ring-on-ring flexure and scratch tests were the main mechanical characterization tests. Herein, we report that the thin films can be used to enhance the mechanical properties of the glass substrates efficiently and effectively. TiO_2–SiO_2 binary system shows more than two times and Ta_2O_5 thin films show nearly three times better ultimate strength in the ring-on-ring flexure test. Besides, Ta_2O_5 thin film samples show superior scratch resistance. Additionally, the finite element method was also used to check the conformity in the application of mechanical properties of composite materials. It is also worth noting that, the finite element method can be used to accurately analyze the mechanical stability of composite materials. The use of the finite element method can reduce the total number of experimental trials without losing reliability.

1. Introduction

Improving the mechanical stability of glass has been worked for decades. Thermal and chemical treatments and lamination are well-known procedures that enhance mechanical properties of glass substrates [1]. Since, glass is one of the most commonly used substrates in various applications, the optical properties e.g. transparency of the films also become crucial as well.

TiO_2, Ta_2O_5, SiO_2, their binary and ternary systems are extensively used oxide films for optical interference coatings. They are stiff and chemically resistant, transparent in the visible range and have a stabile refractive index, as well as having excellent environmental stability and non-toxicity. Thin film structures have very wide applications in microelectronics, optics, semiconductor multi-layers, superconducting systems, data storage systems, projection displays and solar cells [2–6].

A thin film is a layer of material ranging from fractions of a nanometer (monolayer) to several micrometers in thickness [2]. Thin film technology is an inexpensive method to synthesize rare materials efficiently. Recently, thin films have been studied extensively via the sol–gel method, which offers flexibility to the deposited film composition, high throughput and simplicity in material selection, when compared to conventional techniques. Whilst, optical and structural properties of transition metal oxide thin films have been studied considerably, their mechanical properties have not drawn as much attention as their other physical properties.

Shi et al. have used titania nanofilms to improve mechanical stability of glass plates via the sol–gel method [7]. Wenxiu et al. have worked on optical and mechanical properties of SiO_2/TiO_2 organically modified silane composite thin films [8]. Bursikova et al. have investigated mechanical properties of thin silicon films deposited on glass and plastic substrates [9]. Oscar Borrero-López et al. have used titanium oxide thin films on glass in order to enhance mechanical stability and scratch resistance [10]. Strauss has worked on gas pressures influence on the optical and mechanical properties of Ta_2O_5 films [11]. On the other hand, Zhang et al. have used theoretical analyses and numerical simulations on the mechanical strength of multilayers subjected to ring-on-ring tests [12]. Chen et al. have investigated residual stress for oxide thin films by the finite element method [13]. Venkateswara Rao has worked on application of the finite element method to evaluate the mechanical properties of thin films [14].

The novelty of this study comes from combining experimental and FEM analysis and matching their results. Our primary aim was to improve the mechanical stability of glass substrates with thin film technology by the sol–gel method while preserving their

* Corresponding author at: Istanbul Technical University, Physics Engineering Department, Istanbul 34469, Turkey.
E-mail address: ozesra@itu.edu.tr (E.O. Zayim).

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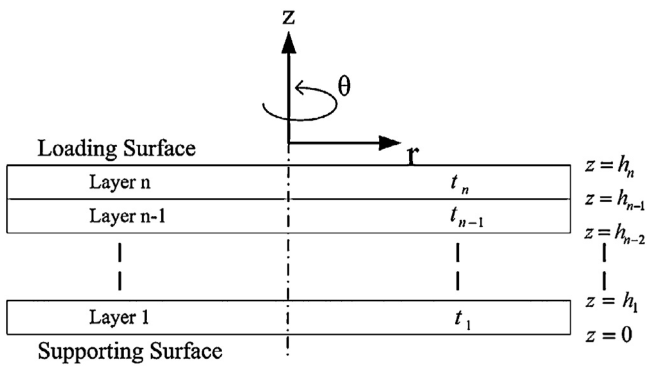

Fig. 1. Schematic representation of an axial symmetry of a thin elastic multilayered disk.

optical properties. And, by using FEM analysis, we aimed to lessen the total number of mechanical test without losing reliability.

2. Finite element analysis

The finite element method (FEM) is basically defined as dividing a continuum system to small elements. FEM analysis describes element properties as matrices and assembles them to reach a system of equations whose solutions give the behavior of the total system. The finite element method is a numerical method which is commonly used for various multi-physics problems lately. FEM methodology has provided an effective means to explore and predict the behavior of high-performance thin film systems. Therefore, the use of FEM design could reduce the total number of experimental trials, while still maintaining a high accuracy of analysis. In this paper, *Ansys 15* was used to analyze external stress within each layer. Various outputs and characteristics of the thin films and substrates mechanic such as failure strength, stresses, temperatures, etc. can be predicted by using FEM without doing any experiment.

Boundary conditions were set according to the rings radius of the ring-on-ring test machine. The supporter ring radius is 21 mm; indenter ring is 9.5 mm. *Shell 181* element was used to model layered structure [13,14]. Glass substrate was fixed within all 6° of freedom with nodes which were modeled according to supporter ring.

Since, nanostructured material properties are quite different from bulk conditions, Young's Modulus (E) is calculated via analytical calculations shown below [12].

$$E_i^p = E_i/(1-\nu_i^2) \quad (1)$$

where E^p represents plane strain modulus and ν shows Poisson's ratio.

$$D^m = \sum_{i=1}^{n} E_i^p t_i \left[h_{i-1}^2 + h_{i-1}t_i + \frac{t_i^2}{3} - z_n^m \left(h_{i-1} + \frac{t_i}{2} \right) \right] \quad (2)$$

D^m shows flexural rigidity of layer; t and h values are shown in Fig. 1.

$$z_n^m = z_{nr} = z_{n\theta} = \frac{\sum_{i=1}^{n} E_i^p t_i \left(h_{i-1} + \frac{t_i}{2} \right)}{\sum_{i=1}^{n} E_i^p t_i} \quad (3)$$

Radial and tangential neutral surfaces are equal and it is redefined as Z_n^m.

The maximum stress is supposed to occur within the inner radius of the indenter ring. In addition radial and tangential stresses are equal to each other. Therefore, the stress value for each layer

Table 1
Young's Modulus and Poisson ratio of materials.

Material	Poisson ratio	Literature Young's Modulus [GPa] [9]	Calculated Young's Modulus [GPa]
Ta_2O_5	0.23	140	1180
TiO_2–SiO_2	0.18	85	550

can be calculated by:

$$\sigma_{ri} = \sigma_{\theta i} = \frac{-E_i P (z - z_n^m)}{8\pi D^m (1-\nu_i)} \left[2\ln\left(\frac{l_2}{l_1}\right) + \frac{(1-\nu)\left(l_2^2 - l_1^2\right)}{(1+\nu)R^2} \right]$$

$$(r \le l_1 \quad \text{and} \quad i = 1 \quad \text{to} \quad n) \quad (4)$$

σ, P, l_1, l_2, R indicate stress, applied force, indenter ring radius, supporter ring radius and sample radius respectively. The calculated Young's Modulus can be used in FEM simulations.

Since, the applied force P can be read from test machine and σ is assumed according to glass ultimate tensile strength which is 70 MPa, Young's Modulus of thin films can be calculated by using Eqs. (1)–(4). Material constants are shown in Table 1. Materials are assumed isotropic. Since, the thickness of films is rather smaller than the size of glass substrates, the linear mixed rule cannot be used to calculate the elastic modulus of films quantitatively. However, many studies reported the X-ray diffraction and indentation methods for measuring the elastic modulus of thin films, and the elastic modulus is between 200 and 1000 GPa for different kinds of inorganic films [7].

3. Experimental procedure

Two types of glass substrates were used in these experiments. Indented (300 g applied for 5 s by Shimadzu Microhardness Test Equipment) glass (ŞİŞECAM Glass of 8 mm × 8 mm × 3.2 mm) and Corning glass 2947 was used for the mechanical and the optical characterizations respectively. Prior to the deposition of film on the glass substrate, Corning glasses were first flushed with a liquid detergent and then washed with de-ionized water. Next, the surface is washed ultrasonically in acetone, methanol and ethanol separately. ŞİŞECAM Glasses were cleaned by and industrial washing system (WV–120/90 FinnSonic Washing System) containing 5 separate tanks. First tank contains 3–10% sodium hydroxide solution (pH: 9), second tank deionized water, third tank 2–4% phosphoric acid solution (pH: 1), fourth and fifth contains deionized water. Tanks were set to 60 °C and glass samples were first dipped in first tank for 9 min, than rinsed in second tank, and dipped in third tank for 9 min and rinsed in second, fourth and fifth tanks. Drying of the samples were done at 100 °C in CRD-90 FinnSonic drying oven for 8–10 min.

The SiO_2–TiO_2 hybrid sol was prepared according to Ref. [15]. The molar ratio of TiO_2 to SiO_2 is 5%. For Ta_2O_5 sol, 1.8 ml tantalum ethoxide was dissolved together with 35 ml ethanol and 0.5 ml acetic acid and stirred for 30 min. Meanwhile another solution was prepared with 15 ml pure water and 0.5 ml acetic acid and were stirred for a 10 min. 0.75 ml of this solution was added into 7.5 ml ethanol and was stirred for 20 min. 2.5 ml of the last mixture was added into the first solution and were stirred slowly for 18 h with a magnetic stirrer at room temperature. All films were deposited on glass substrates by a sol–gel technique. All as-deposited films were heated at 120 °C for 30 min in order to get a film formation on the glass substrates.

The piston-three-balls test has been standardized as ISO 6872. (ISO 6872:2008 specifies the requirements and the corresponding test methods for dental ceramic materials for fixed all-ceramic and