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Nanomechanical characterization of porous anodic aluminum oxide films by nanoindentation

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ABSTRACT

Nanostructured thin films have gained great interests in recent years due to their significantly enhanced properties and great potential for various applications. Nanoindentation techniques are commonly used to measure nanomechanical properties of thin films and the surface layers of bulk materials. In this article, nanoindentation tests coupled with computer modeling were proposed to characterize nanostructured porous anodic aluminum oxide (AAO) films grown on glass. A three-dimensional nanoindentation model was developed. The Young's modulus and hardness were measured by nanoindentation using a Berkowich pyramidal sharp tip indenter. The effects of the substrate and porous structure of the film on the coating measurements were investigated. The dilemma for extracting the mechanical properties of a porous structural film in nanoindentation has been pointed out, and an alternative approach of combined modeling/experimental provided a justification by considering the porous structure of the film and minimizing the influence of the substrate. Computer modeling results were validated by experimental data.

1. Introduction

Nanostructured materials, thin films, and coatings offer the potential for significant improvements in physical properties, such as optical, electromagnetic, thermal, and mechanical properties. Nanoscale characterization is a critical step to understanding the properties of nanostructured materials that possess microstructural features about 100 to 1000 times smaller than conventional engineering materials. Surface morphology can be characterized using techniques such as scanning probe microscopy (SPM) (e.g., atomic force microscopy) and scanning electron microscopy (SEM). Thin-film cross section can be analyzed using transmission electron microscopy (TEM), SEM, X-ray and neutron scattering [1]. Direct measurement of the mechanical properties of thin films and nanostructured materials is particularly challenging, because traditional mechanical tests are not suitable in most cases. Scaling down the conventional mechanical tests has led to novel techniques that have enriched knowledge in nanomaterial properties, such as Young's modulus, hardness, yield strength, strain-rate sensitivity, strain-hardening rate, friction and wear resistance, and fracture strength [2]. Among these nanoscale-mechanical-characterization techniques, nanoindentation is particularly attractive.

Nanoindentation measurement is based on continuously monitoring the load and the depth of an indenter during an indentation process at the micro −/nano-scale. This technique has been significantly improved over the past 30 years and has become popular for assessing mechanical response at high spatial resolution. One of the advanced applications of nanoindentation is to determine the mechanical properties of thin films and coatings. In nanoindentation tests, the properties of a thin film may be measured without removing the film from the substrate as needed in other types of tests. By continuously recording the load and indenter displacement, a single test produces a load-depth curve that can be further analyzed to determine the mechanical properties of the material [3–59].

The main difficulty encountered in nanoindentation of thin films and coatings is to avoid probing the properties of the substrate and to separate the influence of the substrate from the measurement data. To achieve this, it is common to restrict the maximum depth of penetration in a test to no more than 10% of the film thickness, although research suggests that this rule has no physical basis [46,54,58]. On the other hand, controlling indentation penetration within 10% of the film thickness requires very small load and leads to great errors in the test data. A number of researchers have attempted to derive expressions that correlate measured thin film properties with substrate properties and composite properties (measured on coated substrate). The proposed models are based on the theoretical analysis or numerical simulation, taking into account a number of factors, such as the effects of the film thickness, the P–h behavior, the pile-up or indentation profile, and the indenter contact radius or pressure distributions [3,5–7,11,15–18,20–23,25–32,34,36–38,40–42,44,45,48,49,52,53,59]; the plastic zones in the film and substrate or laws of mixture of the area or volume fractions

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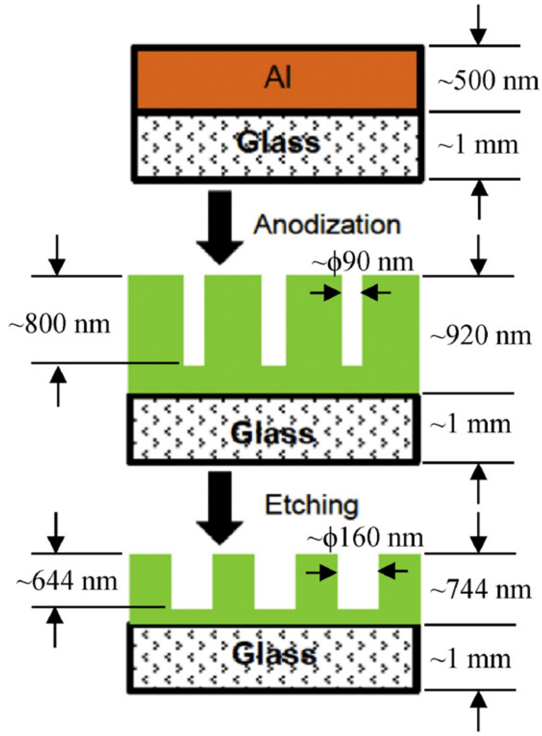

Fig. 1. Schematic diagram of fabrication procedure of porous anodic aluminum oxide [65].

[8,13,14,16–19,25,30,36,42,51]; the response of the elastic and plastic deformation of both the film and substrate and the film–substrate composite associated with the indentation [4,6–11,23,24,26,32,41,45,52]; the various shapes/types of the indenters (such as cylindrical [9,14,44], conical [5,10,14,44,46], spherical [3,8,15,17,22–24,28], Vickers [9,29,37,43], and Berkovich [6,9,23,30,37,41,43,45,48,59] indenters); the different film–substrate combinations for which the substrate is either harder or softer than the film [8,11,16,35,36]; the film and substrate having different yield strengths or Young's moduli or hardness [10,11,47]; the features regarding interface, structure, failure, time and energy, and porosity of the films [8,12,20,33,37,39,43,46,48,49,51,54,59]. The transitional functions represent the connection of the composite response of the film/substrate system to the individual responses of the film and the substrate and are sensitive to the mechanical properties of film and the substrate. Even with well controlled and systematic nanoindentation experiments, which can provide valuable insights on the material properties and help practical designs, accurate prediction of the material properties is impossible without parallel modeling effort. Therefore, finding a way to overcome the limitation of the minimum penetration and to separate the influence of the substrate from the measurement data is a key to successfully characterizing thin films and coatings by nanoindentation.

Among many important thin-film applications, antireflection coatings in solar panels are highly desired to obtain high energy-conversion efficiency. Common antireflection coatings on glass include multiple thin layers that need precise control of the film thickness and hence are expensive [60–64]. Porous anodic aluminum oxide (AAO), a mixture of air and alumina, has the potential to meet the requirements. By tuning the

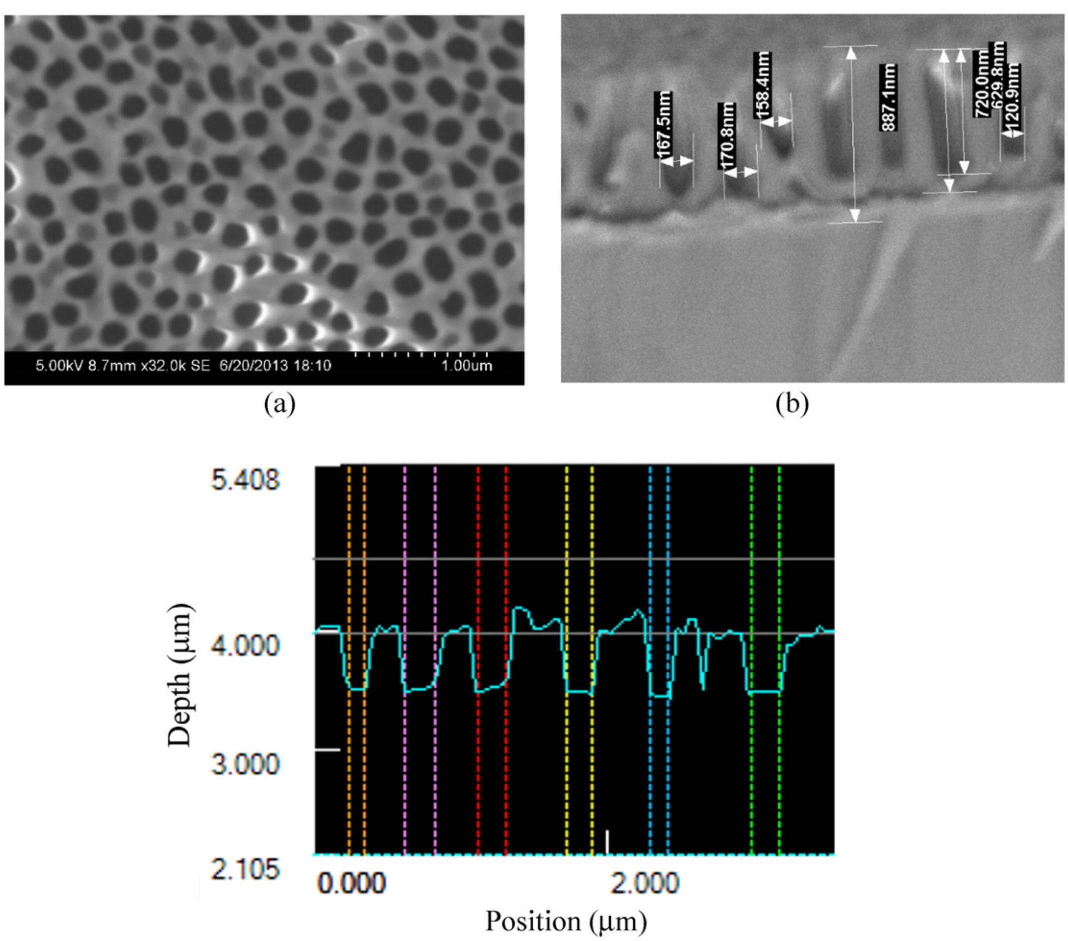

Fig. 2. The resulted AAO coating sample: (a) SEM image of the surface morphology, (b) SEM image of the cross section, and (c) 3D laser microscope image of the cross section porosity profile.