Thin Solid Films 536 (2013) 179–186

Contents lists available at SciVerse ScienceDirect

Thin Solid Films

journal homepage: www.elsevier.com/locate/tsf

The structure and properties of thin aluminum coatings

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ARTICLE INFO

Article history:
Received 28 December 2011
Received in revised form 15 March 2013
Accepted 21 March 2013
Available online 2 April 2013

Keywords:
Strength
Mechanical properties
Coating
Fragmentation
Buckling

ABSTRACT

Mechanical properties and structure of a thin aluminum coating were investigated. Experimental methods of measuring yield stress, strength and fracture strain of ultrathin coatings were developed. Yield stress, strength and fracture strain of aluminum increase with decrease in the coating thickness. The increase of yield stress and strength was explained by reduction of the crystal size and by strain-hardening of metal.

1. Introduction

Thin films are increasingly used in coatings, optical reflectors etc. [1]. Although the mechanical properties of submicrometer-thick films are critical for their use, there are currently few methods for measuring thin film properties. Conventional mechanical testing devices are not sensitive enough to measure the forces resulting from straining of submicrometer-thick films.

Scratch test [2–5] and indentation test [5–7] are sometimes used to characterize the properties of thin rigid films on a substrate. In the case of scratch test, scratches are made on the coated sample by a diamond indenter, which is drawn across the surface. By indentation test the Young's modulus E and hardness of thin metal films were determined. Both the hardness and the Young's modulus depend on the film thickness [8,9]. Unfortunately, the scratch and indentation tests do not allow to estimate the fracture strain and strength.

In addition to indentation test, the Young's modulus and the Poisson's ratio of metal coatings were determined by the X-ray diffraction [10].

At scratch test, adhesion strength may be calculated from the size of debonded zone and the load needed to remove the film from the substrate. Although quantitative evaluation is easily realized in such tests, the results are influenced by the strength of film and interface, by film thickness [8,9], and residual stress [11]. Adhesion strength was measured also by shear loading [12]. In the alternative approach adhesion is characterized by interface fracture toughness G [13,14]. Interface fracture toughness G represents the amount of energy needed to extend a crack by unit area along the substrate/coating plane. A thin rigid coating supported by a soft substrate under tension load can fracture into a number of parallel cracks oriented perpendicularly to the loading direction. The crack in the coating may turn by 90° and grow along the interface [13]. This process causes debonding of the coating. The coating debonds if the inequality $\sqrt{2EG/h}<\sigma_y$ is fulfilled, where σ_y, E and h are yield stress, Young modulus and the thickness of the coating respectively. If the coating thickness h exceeds some critical value h_c, the cracked coating debonds from the substrate. In contrast, thin coatings do not debond from the substrate. The critical thickness value h_c for the Pt/polyethylene terephthalate (Pt/PET) system was equal to 26 nm [13].

The Young's modulus of the coating or substrate was measured also [15,16] using spontaneous buckling of a rigid coating on a soft substrate under compressive or tensile load [17–21].

Mechanical, physical–chemical and other properties of thin films depend on their thickness [22–28].

Thus, the properties of thin films may be measured by different methods. However, there is a need for development of methods, especially for measurement of the coatings strength. The aim of this work is to develop methods for measuring fracture strain, strength and yield stress of thin aluminum coatings deposited on polymer films.

2. Experimental details

A commercial film of amorphous non-oriented polyethylene terephthalate (PET) was used as a substrate. The thickness of the film was

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0040-6090/$ – see front matter
http://dx.doi.org/10.1016/j.tsf.2013.03.050

100 μm. Samples, dumbbell in shape, were cut from the film. The gauge of samples was 6 × 20 mm^2.

Thin aluminum coating was deposited on the polymer films by thermal sputtering in vacuum in VUP-4 device. The thickness of the coating was changed by variation of time of metal deposition. Control measurements of the coating thickness were made by depositing an aluminum coating on a glass plate. After that, the coating was scratched with a sharp stick, which does not scratch the glass, and the depth of the scratch was measured by an atomic-force microscope. Fig. 1 illustrates the method [29]. The light area in the left part of the image is an aluminum layer, and the dark area on the right is the glass surface. The coating thickness was proportional to the time of metal deposition. The proportionality coefficient was found from Fig. 1, thus determining the thickness of the aluminum coating as a function of the deposition time.

Mechanical properties were studied with a standard "Instron-1122" test machine. The samples were tested in tension at temperature 20 and 90 °C at a cross-head speed of 10 mm/min. After elongation of the coated sample at temperature 90 °C (which is higher than the glass transition temperature T_g of PET substrate) it was cooled in the clamp of the test machine to room temperature.

The samples after elongation were investigated in the scanning electron microscope (SEM) "Hitachi S-520", working voltage is 20 kV, transmission electron microscope (TEM) "LEO 912AB", working voltage is 80 kV, and atomic-force microscope "Nanoscope-IIIa" in the contact and tapping modes, non-contact (fpN10S, length 100 μm, resonance frequency 254 KHz), and contact (fpC10S, length 250 μm, force constant 0.1 N/m) cantilevers were used manufactured by State Research Institute for Problems in Physics (Zelenograd, Russia). The average values of the width of coating fragments and the period of the microwave were determined from SEM and scanning probe microscopy (SPM) images using FemtoScan Online software [29].

3. Theoretical background

Fig. 2 shows SEM micrographs of a coated PET elongated at 90 °C (2a) and at room temperature (2b). The sample 2a was elongated at temperature higher than the glass transition temperature T_g (75 °C) of PET substrate. The sample 2b was elongated at room temperature, i.e. below T_g. The light bands, perpendicular to the elongation axis, are the coating fragment bands. The dark bands show PET substrate made visible due to opening of cracks in the coating. The coating in Fig. 2 is fragmented on several bands of approximately equal width. In addition, the coating is folded so that the surface is wavy, and the wave crests are parallel to the tension direction. Spontaneous formation of a wave on an initially smooth surface is described in [17,18]. In contrast, if a sample was elongated at temperature below T_g of the polymer substrate (Fig. 2b), the wave-like folding do not appear. In this case only the coating fragmentation is observed.

Both the coating folding and coating fragmentation are quite periodic. Both structures appear if the following conditions are fulfilled [17,18]: 1) the coating is much thinner than the substrate and 2) the elastic modulus of the coating is much higher than that of the substrate. 3) the coating is adhered to the substrate. The first two conditions are fulfilled for a polymer with a metal coating at temperature higher than the glass transition temperature of the polymer substrate. The coating does not debond from the substrate if it is thin and its thickness is lower than some critical value determined by the debonding fracture toughness.

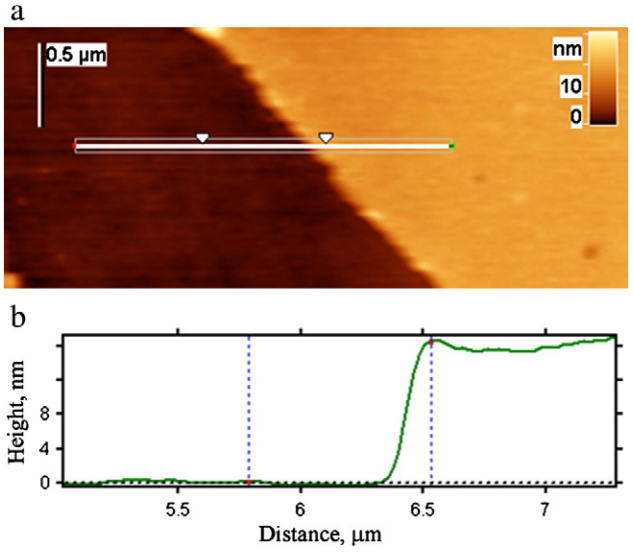

Fig. 1. SPM-image and a cross-section of a glass plate coated with scratched thin Al film.

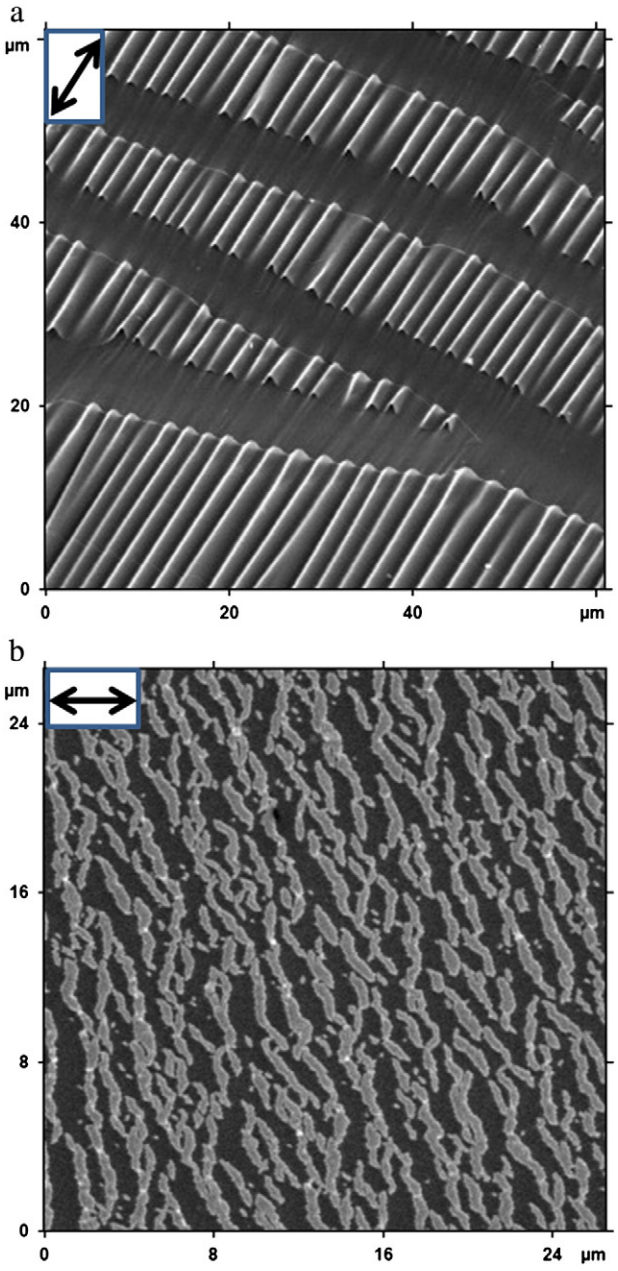

Fig. 2. SEM microphotographs of PET coated with 10 nm gold film and elongated by 50% at (a) 90 °C and (b) room temperature.