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Experimental study on tensile bifurcation of nanoscale Cu film bonded to polyethylene terephthalate substrate

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ABSTRACT

Cu films are widely used in flexible electronic products. Tensile mechanical properties of the film determine product performance. In this paper, tensile experiments of sputtered Cu films on a polyethylene terephthalate (PET) substrate were carried out under an optical microscope. In the experiments, three changes took place under tension: uniform deformation, microcrack initiation and propagation, and microcrack saturation. The elastic modulus of the Cu film is 120 GPa and is independent of film thickness since the film is formed to be continuous in the nanoscale range. Film thickness is an important parameter to decide the tensile properties. The critical fracture strain, the interfacial bonding strength, and the crack spacing after saturation are related to film thickness. The critical strain and the interfacial bonding strength of the nanoscale Cu film tend to ascend then to descend as film thickness increases. The microcrack spacing is in direct proportion to film thickness after the microcrack saturates. The optimum thickness of the sputtered Cu films on the PET substrate is about 500 nm.
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1. Introduction

Flexible electronic products have a broad prospect of application, such as collapsible display, electronic skin, and amorphous thin film solar cell [1]. The fundamental difference between flexible electronic technology and traditional electronic technology is that in flexible electronic technology flexible substrates are used instead of traditional rigid substrates. Ductility and flexibility are thereby improved [2]. The substrate materials usually used in flexible electronic products are polyimide, poly-dimethylsiloxane, and polyethylene terephthalate (PET) [1]. Generally, there are two main failure modes for thin films [3]: one is bifurcation and fracture under tension loading, and the other is buckling and delamination under compression. The thin film devices used in flexible electronic products are mainly metal thin films bonded on polymer substrates. The film structure is subjected to repeated tensile testing, curling and folding during operation. The mechanical properties of the films will directly determine quality and service life of products. At present, many researchers have been devoted to the study of the mechanical properties of films bonded to flexible substrates and have yielded important information. Deformation of solids is homogeneous in the uniform strain, while local deformation or inhomogeneous deformation mode is called bifurcation. The theory of this bifurcation phenomenon in plastic solid field was first proposed by Biot [4] in 1965. Study on bifurcation problem for film deformation by Li and Suo [5–7] is worth mentioning. From 2004 to 2007, they carried out systematic theoretical study on the deformation bifurcation phenomena of film/substrate structure under tensile load, but further experimental validation and improvement are required. Many researchers [8–10] have proved that freestanding metal films usually ruptured at a small strain (<1%) and formed a neck within a narrow region. By contrast, the substrates could suppress necking and the fracture strain varies from 1% to several tens of percent. Some researchers [11–14] reported that the fracture strain of metal films bonded on substrates is less than 10%. For example, Lacour [12] suggested in his experiments that gold films on an elastomer substrate formed cracks at about 8% strain. Xiang [13] reported that the strain of well-bonding Cu films could reach 10% and discrete microcracks could form at a strain of 30%. Some research groups [15,16] reported in their experiments that the strain of supported metal films could reach 20%. Kang [15] reported that some of his aluminum films bonded on a polyimide substrate didn't form any crack under a strain of 20%. Lu et al. [17] have achieved elongations over 50% of polyimide-bonded Cu films without detectable fractures. Lu et al. [18] in 2010 performed uniaxial tensile tests on polyimide-supported copper films with thicknesses varying from 50 nm to 1 μm using a focused ion beam/scanning electron microscope and Intron 3342 tensile tester. The experimental research and numerical simulation showed that film thickness, grain size, crystallographic texture and adhesive property have a

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direct impact on the strength and deformation of thin films. Djaziri et al. [19] investigated the elastic–plastic behavior of nanostructured W thin films deposited on Kapton using X-ray diffraction (XRD) and digital image correlation (DIC) combined technique under equi-biaxial and non-equi-biaxial loading conditions. The elastic limit of the nanostructured W thin films was determined at a strain of about 0.5% obtained by XRD and DIC. The research group of Sun Jun [14] used electrical resistance and statistical microcrack density method to measure the critical strains of the Cu films with a thickness ranging from 60 nm to 700 nm. They reported that the critical strains of polymer-supported Cu films were simply dependent on the thickness, i.e., the thinner the film, the lower the critical strain. That means thicker film is better. But it was mentioned by Freund and Nix [20] that the film had a critical thickness. Current research on thin film tensile deformation bifurcation is still in its infancy. Theoretical model parameter selection, bifurcation models, deformational evolution rules, relationship between material parameters and deformation bifurcation require further experimental validation and improvement.

The Cu film has great prospect in the ultra-large-scale integrated circuit as an interconnect material. It has the advantages of low resistance rate, good heat conduction, small thermal expansion coefficient, and high melting point. It helps to improve work frequency of circuit and electroresistive migration ability [21–25]. However, the considerable stress in Cu films can cause holes, cracks or peeling in the membrane. The open or short circuit accident affects the application of Cu films. There is a scale effect within the micro or nanoscale. Therefore, research on tensile mechanical properties of Cu films is important. In this paper, tensile tests carried out for the Cu films sputtered on PET substrates under an optical microscope are presented. The morphology evolution rules of Cu films under tension are addressed. The relationships between critical strain, elastic modulus, saturated crack spacing, interfacial strength and film thickness are studied.

2. Experiments

2.1. Specimens

Nanoscale Cu films with different thickness are bonded to 0.12 mm PET substrates. The films are fabricated by a direct-current (DC) magnetron sputter-deposition system at room temperature. Prior to deposition of the metal films, the substrates are polished and ultrasonically cleaned with acetone and methanol. The sputtering conditions are: base pressure at 1×10^{-4} Pa, working-gas pressure at 1 Pa Ar, sputtering power at 20 W, bias voltage at −20 V, and sputtering rate at about 20 nm/min. These deposition conditions are similar to those used in Lu et al. [17]. Film thickness is controlled by varying the sputtering time. The samples are divided into groups by film thickness. There are two kinds of samples in each group, one is Cu films on Si substrates and the other is Cu films on PET substrates. Thickness of the films on Si substrates is calibrated using the surface profile device and taken as the value on PET substrate among the same group samples. The film sample size is shown in Fig. 1. The middle of the specimen is the observation area. The frames on both sides of the specimen are designed in order to reduce distortion caused during installation. The frames of the specimen are cut before the experiment. Elongation can be checked according to the fracture at the frames after stretching. Due to relative flatness before sputter deposition, the curvature of PET substrate is zero. The curvature radius of the film–substrate composite is about 432 mm after sputter deposition. According to Stoney's formula [26]

$$\sigma = \frac{E_s {h_s}^2 \kappa}{6 h_f (1 - \nu_s)} \tag{1}$$

where the subscripts "f" and "s" denote the thin film and substrate, respectively, $E_s = 0.78224$ GPa is the Young's modulus, $v_s = 0.35$ is the Poisson's ratio, h is the thickness, and κ is the change in system curvature. The residual compressive stress of the film–substrate composite is about 13 ± 2 MPa. The magnitude of the stress is generally small in comparison to the stress levels applied in the composite.

2.2. Experimental setup

Uniaxial tensile test has features of simple structure and high reliability. It can clearly reflect material deformation process and material performance parameters can be obtained. The objective parameters are proportional to the sample thickness. The error caused by inaccurate thickness measurement is smaller than that from bending test. The experimental results from the tension test are easy to explain. Manufacture, processing and testing of the freestanding film sample whose thickness is less than 3 μm are very difficult. If Si material is used as the frame, this approach of pattern-making and etch preparing is very complex. The supported films may cause non-uniform deformation and bifurcation phenomenon under tension. Therefore, the uniaxial tensile test method is used to study the bifurcation behavior of the films. The specimen is loaded with a small displacement. Pictures are finally recorded after five minutes. The deformation morphology and microcrack expansion of the films under tension are recorded by the pictures.

3. Experimental results and discussion

When the film/substrate structure is stretched, stress concentration on the film will cause local stretch, which needs accommodation space. According to volume conservation, this space is unstable for the film/substrate structure. When the tensile strain reaches the critical strain, the film will be thinner in the region of stress concentration and local necking occurs, which is called bifurcation phenomenon. Once necking is formed at a local spot, further deformation will be localized at the necking region as the tensile strain increases. Fractures will also occur at the necking region. The observation scope and resolution of the optical microscope are affected by natural light wavelength. The max resolution is 200 nm. Due to restriction of the microscope, it is difficult to capture the critical bifurcation point. But microcracks can be observed on the film surface and the mechanical characterization of the thin film can therefore be inferred.

The nanoscale Cu films bonded to the substrates experience three stages during tension (as shown in Figs. 2 and 3): uniform deformation, microcrack initiation and propagation, and microcrack saturation. Force–displacement curve is selected rather than stress–strain curve, because the stress is not uniformly distributed. The microcrack propagation direction is almost perpendicular to the loading direction. The microcracks are expanding until intersecting with another microcrack during tension. The microcracks look like furrows. The crack density is not uniform because of defects on the prepared films.

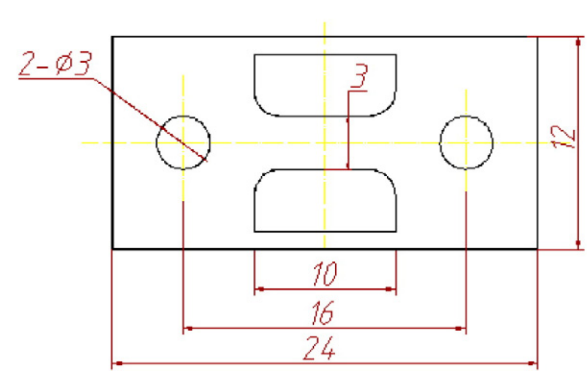

Fig. 1. Film sample size (units: mm).

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