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Fixtureless superlayer-driven delamination test for nanoscale thin-film interfaces

Jiantao Zheng, Suresh K. Sitaraman *

Computer-Aided Simulation of Packaging Reliability (CASPaR) Laboratory, The George W. Woodruff School of Mechanical Engineering, Georgia Institute of Technology, 813 Ferst Drive, Atlanta, GA 30332-0405, USA

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Abstract

A fixtureless delamination test has been developed to measure the interfacial fracture toughness of patterned nanoscale thin films on a substrate. The driving energy for delamination propagation is supplied by a highly stressed superlayer deposited on top of the nanoscale thin film. The amount of energy available for delamination propagation is changed by depositing an etchable thin release layer with varying width between the nanoscale thin-film strip and the substrate. By designing a decreasing area of the release layer, it is possible to arrest the delamination at a given location, and the interfacial fracture toughness or critical energy release rate can be found at the location where the delamination ceases to propagate. For titanium film with a thickness of 90 nm, the results show that the interfacial fracture toughness of titanium/silicon ranges from 3.45 J/m^2 to 5.70 J/m^2 when the mode mixity increases from 6.8° to 38.4°. The methodology presented in this paper is generic in nature, and can be used to measure the process-dependent interfacial fracture toughness of various micro and nanoscale thin films on a substrate.

Keywords: Adhesion; Interfaces; Interfacial fracture toughness; Silicon; Titanium

1. Introduction

Microelectronics, optoelectronics, MEMS/NEMS, and other applications contain several layers of thin-film materials with dissimilar properties bonded together. These thin films could delaminate under external loading during operation or intrinsic stresses during fabrication and processing. The criterion for delamination propagation in such thin films is that:

$$G > \Gamma(\psi) \tag{1}$$

where G is the available energy release rate (ERR) for delamination propagation, Γ is the interfacial fracture toughness, and ψ is the mode mixity, which is related to the ratio of shear to normal stresses at the crack tip. Unlike cohesive fracture toughness, interfacial fracture toughness is a function of mode mixity, and typically, interfacial fracture toughness increases with mode mixity. Thus, delamination is less likely to occur when the mode mixity is higher; that is, when the loading along the interface is mostly shearing.

With the film thickness scaling down to nanoscale, understanding and quantifying the interface characteristics is challenging. For example, in microelectronics back-end processes, the thickness of the metal interconnects and the insulating dielectric is generally 100–500 nm thick, and the barrier film used between the metal and the dielectric is normally 5–10 nm [1]. Handling such nanoscale thin films and measuring the interfacial parameters is cumbersome. Therefore, appropriate test methods are needed to characterize the interfacial fracture for these and other interfaces consisting of nanoscale thin films. Ability to handle nanoscale thin films, ability to create representative interfacial conditions, ability to extract fracture parameters from the given test conditions, ability to address a wide range of mode mixities, etc. are some of the challenges associated with interfacial fracture toughness measurement.

A number of test methods [2,3] are currently available for the measurement of interfacial fracture toughness. Some of these test methods include symmetric double cantilever beam [4],

* Corresponding author.
E-mail address: suresh.sitaraman@me.gatech.edu (S.K. Sitaraman).
URL: http://www.me.gatech.edu/caspar (S.K. Sitaraman).

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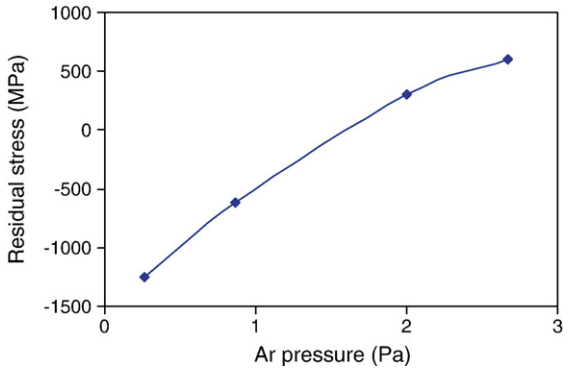

Fig. 1. Residual stress in Ti–W (10% Ti, 90% W) as a function of the Argon pressure during physical vapor deposition.

Brazil nut sandwich [5], elastic-plastic peel test [6,7], four-point bend beam [8], sandwiched beam [9], micro-scratch technique [10,11], blister test [12], and nanoindentation test [13–15]. Although a number of test methods are available, in one way or another, the existing test methods fail to meet one or more of the criteria, listed below.

(a) An interfacial fracture toughness test should ideally account for the energies accompanying fracture beyond that of surface energy and be able to determine how ψ affects Γ; (b) the test sample should be prepared using the actual fabrication method to be able to create a representative interface; (c) the test sample dimensions should be representative of the actual size used in the application; (d) the test method should simulate the actual usage stress conditions as closely as possible (both in sample preparation and crack development) and the test method should be able to cover a wide range of mode mixity; (e) deformations should be elastic or the plastic deformation is negligible so that the problem can be easily modeled; (f) the test method should preferably lead to an analytical solution to extract fracture parameters; and (g) the test should be easy to perform, repeatable, and efficient.

2. Experimental details

2.1. Stress-engineered superlayer in the fixtureless delamination test

Before discussing the fixtureless delamination test, it is essential to discuss the concept of stress-engineered superlayer, and how the superlayer can be used to provide the energy for delamination propagation without using external fixtures and mechanical loads.

Intrinsic stresses can be intentionally introduced into thin-film metals by changing the deposition condition, such as the argon pressure during the direct current (DC) magnetron sputtering [16]. Windischmann [17] used atomic peening model as well as grain boundary relaxation model to explain such stress engineering in thin-film metals. When the argon pressure is low in the sputtering chamber, the target metal atoms collide less with the argon ions, and therefore, due to less scattering, the target metal atoms tend to deposit in a condensed pattern on the substrate. Due to this condensed deposition, the interatomic distance is less than the equilibrium spacing, and thus, compressive intrinsic stress is induced in the deposited metal layer. On the contrary, if the sputter chamber argon pressure is higher, the target metal atoms collide more with the argon ions, and therefore, the metal atoms tend to deposit on the substrate in a coarser pattern. Due to the larger interatomic distance than the equilibrium spacing, tensile intrinsic stress is created in the deposited thin-film metal. Fig. 1 shows an example for Titanium–Tungsten (Ti–W) deposition where we were able to introduce stresses ranging from −1.25 GPa to +0.6 GPa in the Ti–W layers by changing the sputter chamber argon pressure from 0.27 to 2.7 Pa. Similar results have already been demonstrated by others [16] for various other materials. Thus, by controlling the argon pressure in the sputter chamber, a uniform tensile stress or a stress gradient can be induced by depositing the metal layer-by-layer, starting with an intrinsic compressive stress and gradually changing it to an intrinsic tensile stress.

Such a stress-engineered layer is called a “superlayer”, and the tendency for the superlayer is to peel off from the substrate due to the presence of a tensile stress or a compressive-to-tensile stress gradient. If the superlayer is to be deposited on top of a substrate with another thin-film material, the superlayer will try to peel off the underneath thin-film material layer from the substrate as shown in Fig. 2.

Utilization of a stress-engineered superlayer to propagate an interface delamination was first described by Bagchi et al. [18]. However, their test has a serious shortcoming: if the thin-film material does not delaminate from the substrate upon the application of the superlayer, their test will call for the processing of another substrate with the thin-film material and the deposition of a thicker superlayer to facilitate the delamination propagation. If the thin-film material layer does delaminate, their test will call for the processing of one more substrate and the deposition of a thinner superlayer to determine the lower bound for the interfacial fracture toughness. Such a trial-and-error repeated processing approach is tedious and could take several weeks to obtain one interfacial fracture toughness data for a given mode mixity. Because of this serious drawback of multiple processing of substrates, their work [19] has not been actively pursued beyond 1996. Modi and Sitaraman [20] improved the method of Bagchi et al. using just one substrate with columns consisting of several thin-film strips to determine the interfacial fracture toughness. By determining which of the strips delaminated and which of the

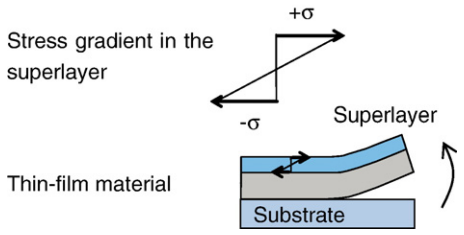

Fig. 2. Delamination of the thin-film material under stressed superlayer.