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Fracture toughness of silicon nitride thin films of different thicknesses as measured by bulge tests

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Abstract

A bulge test setup was used to determine the fracture toughness of amorphous low-pressure chemical vapor deposited (LPCVD) silicon nitride films with various thicknesses in the range 40–108 nm. A crack-like slit was milled in the center of each free-standing film with a focused ion beam, and the membrane was deformed in the bulge test until failure occurred. The fracture toughness K_{IC} was calculated from the pre-crack length and the stress at failure. It is shown that the membrane is in a transition state between pure plane-stress and plane-strain which, however, had a negligible influence on the measurement of the fracture toughness, because of the high brittleness of silicon nitride and its low Young's modulus over yield strength ratio. The fracture toughness K_{IC} was found to be constant at 6.3 ± 0.4 MPa m$^{1/2}$ over the whole thickness range studied, which compares well with bulk values. This means that the fracture toughness, like the Young's modulus, is a size-independent quantity for LPCVD silicon nitride. This presumably holds true for all amorphous brittle ceramic materials.

Keywords: Thin films; Fracture toughness; Bulge test; Size effect; Silicon nitride

1. Introduction

The successful design of microelectromechanical systems and microchips requires the accurate knowledge of the mechanical properties of the materials used in thin films, which usually differ from those of their bulk counterparts. Not only are the fabrication processes different, leading for instance to different densities, but it has also frequently been reported that downscaling inherently leads to a modification of the mechanical behavior of materials. More precisely, a large number of experiments performed by different techniques such as bending tests [1,2], nanoindentation [3–5] and nanocompression [6–8] tests have concluded that there is an increase in yield strength with decreasing length scale. Yet little [9–11] is known about the possible size dependence of the fracture toughness and, as a consequence, the design of micro-systems usually relies on macroscopic values obtained from bulk samples. Such an approach is questionable, since it was recognized as early as the 1960s that the fracture behavior of two samples from the same material could strongly depend on their respective thickness. The reason for this is that, in a free-standing thin film, the material is almost in a perfect state of plane-stress, while in the bulk, it is rather in a state of plane-strain. As a result, the crack extends along different planes of maximum shear stress [12], requiring different amounts of energy.

The experimental model of Anderson [13] provides the simplest description of the variation in the measured fracture toughness with thickness. It considers that the measurement yields a plane-stress value $K_{IC}^{\text{plane-stress}}$ for thin samples and a plane-strain value K_{IC} for thick samples, and in this model a linear transition between both states is assumed (see Fig. 1).

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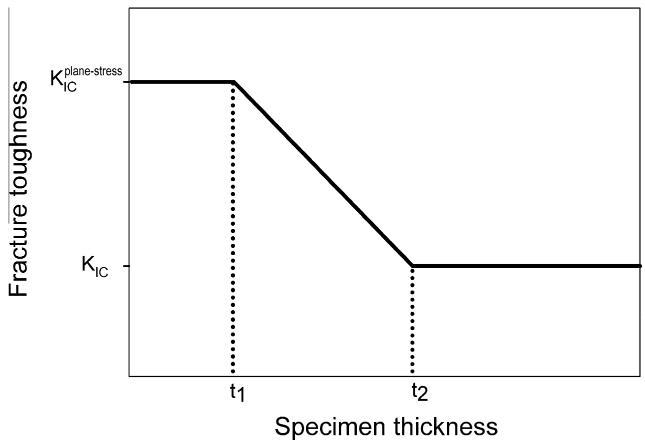

Fig. 1. Model from Anderson, arbitrary values.

The plane-stress state is found in samples thinner than the characteristic size of the plastic zone before failure. The corresponding critical specimen thickness is given by [12]:

$$t_1 = \frac{1}{3\pi} \cdot \frac{K_{IC}^2}{\sigma_Y^2} \tag{1}$$

where σ_Y is the yield strength of the material.

The lower thickness bound for plane-strain fracture toughness testing has been found experimentally to be [14]

$$t_2 = 2.5 \frac{K_{IC}^2}{\sigma_Y^2} \tag{2}$$

There are some more refined theories [15], but the model of Anderson offers a sufficient approximation for engineering and design purposes. However, it should be noted that it does not predict the ratio between K_{IC} and $K_{IC}^{\text{plane-stress}}$, and thus the slope of the transition. While there is currently no model that can predict it accurately, the coarse approximation provided by Broek and Vlieger [16] can be used to assess the order of magnitude of the transition:

$$\frac{K_{IC}^{\text{plane-stress}}}{K_{IC}} = \sqrt{1 + \frac{\varepsilon_f \cdot E}{12 \cdot \sigma_Y}} \tag{3}$$

where ε_f is the fracture strain of the material, and E its Young's modulus. This equation implies that the transition is negligible for an ideal brittle material or for a moderately brittle material with a moderate Young's modulus over yield strength ratio.

As an alternative to the extrapolation of values from those models, direct measurements of the fracture toughness can be performed on thin samples thanks to advanced techniques such as micro-cantilever deflection [17,18] or bulge testing.

2. Bulge testing

The bulge test has long been used to determine the Young's modulus and internal stress of thin films [19–21]. More recently, since the advent of focused ion beam (FIB) technology, its use has been extended to the measurement of fracture toughness [22,23]. A special sample preparation technique is required, where a sharp pre-crack is introduced into the center of a rectangular membrane, in the direction of the long axis.

The membrane is then deformed by the pressure p of a gas, creating a deflection h in the center of the membrane, as illustrated schematically in Fig. 2. From those two values, the lateral stress σ_{xx} and strain ε_{xx} in the center of the membrane can be calculated according to the analytical model of Vlassak et al. [24,25]:

$$\sigma_{xx} = \frac{pb^2}{2th} \quad \text{and} \quad \varepsilon_{xx} = \varepsilon_{0,xx} + \frac{2h^2}{3b^2} \tag{4}$$

where t is the thickness, $2b$ is the width, and $\varepsilon_{0,xx}$ is the lateral pre-strain of the free-standing film.

Because of the geometry of the membrane, the material in its center cannot deform along the long axis, thus yielding the relationship

$$\sigma_{xx} = \frac{E}{1 - \nu^2}(\varepsilon_{xx} - \varepsilon_{0,xx}) + \sigma_{0,xx} \tag{5}$$

where E is Young's modulus, ν is Poisson's ratio, and $\sigma_{0,xx}$ is the lateral pre-stress of the membrane.

In the present study, the measurements are carried out with a self-developed bulge test setup described elsewhere [26]. This bulge test setup can be used inside an atomic force microscope (AFM), so that in situ observations of loaded membranes are possible. The membrane is pressurized by nitrogen gas, and its deflection is recorded with a laser autofocus displacement sensor. In an experiment, the pressure continuously increases until the lateral stress σ_{xx} causes the extension of the crack, leading to the failure of the specimen. Provided that this happens in a quasi-brittle way, the conditions for linear elastic fracture mechanics are satisfied, and the fracture toughness can be calculated according to Irwin [27]:

$$K_{IC} = \sigma_{xx,failure}\sqrt{\pi \cdot a} \cdot Y\left(\frac{a}{W}\right) \tag{6}$$

where a is the crack half length, W is the length of the membrane, and $Y(\frac{a}{W})$ is a geometry factor equal to 1 for this simple membrane condition.

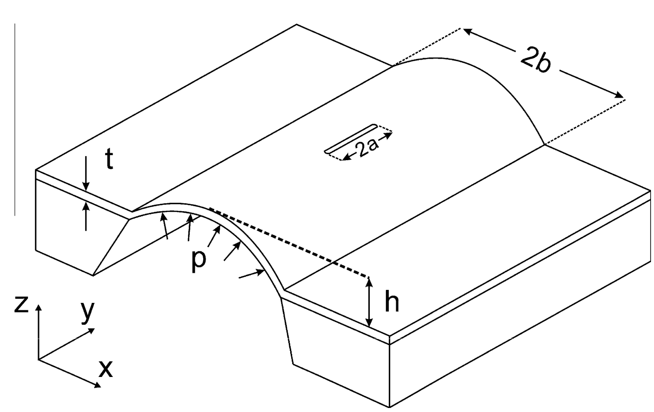

Fig. 2. Perspective view of the central section of a long rectangular membrane with a pre-crack during a bulge test.