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Fracture characterization of brittle thin-films by membrane testing

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The continuous miniaturization process in the microelectronic industry, along with the introduction of Interlayer Dielectrics (ILDs) with poorer mechanical properties, makes necessary the development of characterization techniques to evaluate the mechanical performance of very thin films. This work presents a mechanical characterization technique for thin films based on membrane testing. Membranes, micromachined with anisotropic wet etching of Si, are tested to fracture using a nanoindenter to apply the load and register the provoked deflection. The technique is applied to the fracture characterization of two different ILDs with four thicknesses ranging from 100 nm to 500 nm. Combination of experiments and finite element simulations allows for the calculation of the strength of the materials from the fracture load. The technique permits to discriminate both ILDs and to establish clear thickness dependence: for both materials, 100 nm films show a significant lower strength while no effect of film thickness on strength is observed in the range between 200 and 500 nm. A sensitivity analysis of the outcome of the technique, the fracture stress, to the variability of the input parameters is presented, showing the robustness of the proposed approach: the experimental error in the fracture stress is smaller than the variation in the input parameters.

1. Introduction

As the microelectronic industry advances, the materials used in integrated circuits are mainly optimized from their electronic and optic point of view. Though, mechanical properties cannot be disregarded as the applied stresses increase (superposition of residual stresses, consequence of the deposition processes, stresses during packaging and in-service thermal cycling) [1–3]. Consequently, the use of low-*k* dielectric materials [4,5], with poor mechanical properties, can compromise the reliability of the chip. Therefore many different techniques have been developed to characterize the mechanical properties of the thin films and the properties of their interfaces [6], but these techniques have severe limitations as the thickness of the thin films is reduced.

Four-point bending (introduced by Charalambides et al. [7] and developed and extensively used by the group of Dauskardt [8,9]) is the reference testing method in the industry. In this test, a macroscopic notched sample (10 mm long) is subjected to bending to initiate a crack which kinks and propagates along the interface of interest. The main limitations of this technique are that (i) special specimens involving adhesives are required, thus increasing the throughput time and introducing variability in the results, and (ii) the cracks generated are of the order of millimeters and hence local properties cannot be determined (drawback for patterned structures). As an alternative method, Sánchez et al. [10] developed the cross-sectional nanoindentation (CSN), that was further developed and adapted for characterizing interfaces involving elasto-plastic materials by Elizalde et al. [11] and pattern films by Ocaña et al. [12]. In CSN a crack is initiated in the silicon underlying the structures of interest by nanoindentation, and the crack propagation along different interfaces is measured and used to characterize their adhesion energy.

Concerning the fracture characterization of thin films, several efforts have been made. "Channel cracking", developed by Huang et al. [13], is a technique extensively used in industry. In channel cracking a crack initiated from a scratch is propagated by bending the sample. The film fracture energy is calculated from the stress needed for crack propagation. Even though it is widely used in industry, the experimental errors usually linked to this technique are large. Indentation techniques have also been used to measure fracture toughness. A sharp tip (typically a Vickers, a Berkovich or a cube corner diamond) is pushed into bulk brittle materials. If the applied load reaches the critical value, radial cracking can occur. Using the maximum load and the crack length, fracture toughness can be calculated [14,15]. These techniques have been developed in order to obtain the fracture toughness of ceramic materials [16]. Finite element modeling is necessary to describe the complex stress and strain fields that appear under the indenter tip [17]. The main drawback of these techniques is that the crack patterns obtained depend on the system tested (thin film thickness, substrate properties, residual

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stresses, interfacial properties) which reduces the reproducibility and makes quantification very challenging.

There are several efforts in literature testing microsamples to characterize mechanical properties of small volumes. For instance Matoy et al. [18] used bending of cantilever beams (machined by etching processes) to calculate the fracture toughness of silicon based dielectric materials and found that it increased with decreasing cantilever thickness. As for thin films, bulge test was one of the first techniques introduced to calculate their mechanical properties. Vlassak and Nix, for instance, proposed a formulation allowing the use of this test to calculate the Young's modulus and residual stresses of thin films [19]. In this test, sample preparation is critical. The bulge test can also be used to determine the fracture toughness of thin films [20,21].

In this work, the possibility of testing membranes using a nanoindenter is studied. Brittle thin films are tested until they break so fracture properties can be obtained. The experimental load–displacement record together with finite element modeling can be used to extract the fracture stress of the thin film.

2. Experimental procedure

The samples studied in this work are blanket dielectric films of different natures and thicknesses. Fig. 1 sketches the stack on the samples. A Si (100) wafer is used as a support for the membranes. On one side of the wafer, a SiN layer is prepared through a simple lithography process, with convenient square opens (the largest being 1.5 × 1.5 mm and 2 × 2 mm) to allow for further micromachining (etching of the Si). On the other side, a continuous layer of SiN (50 nm) is deposited to stop the etching process and protect the Interlayer Dielectric (ILD) from the etchant. Finally the ILD layer to be characterized is deposited and a photoresist layer covers the ILD to protect it during sample handling. In the sample preparation process, the SiN layer has not been removed and its properties have to be taken into account to estimate the fracture properties of the target film.

Fig. 2 shows the back side of a sample with the square opens in the SiN layer. Two different ceramic ILDs (ILD-1 and ILD-2) have been tested in 4 different thicknesses each (100, 200, 350 and 500 nm).

2.1. Sample preparation

Membranes have been prepared by anisotropic wet etching with a 30% KOH dissolution at 90 ºC, which removes the Si. A special mounting jig has been used to make sure that just the back side of the sample is exposed to the KOH bath. This way, the etching process starts in the opens drawn in the SiN layer and progresses through the Si until it is stopped at the top SiN layer.

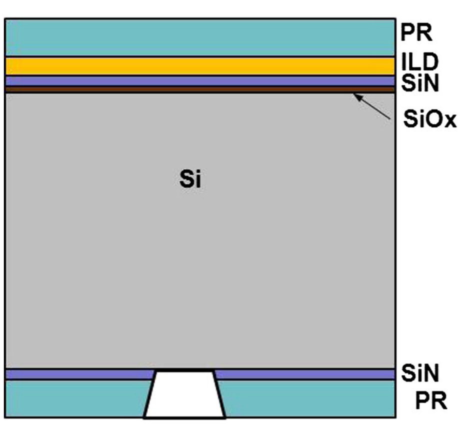

Fig. 1. Sketch of the general stack of the samples. All the samples have photoresist deposited on both sides protecting the thin films. SiN is deposited on one side (bottom) to select the places where the etching process is taking place, and on the top side to stop the etching process.

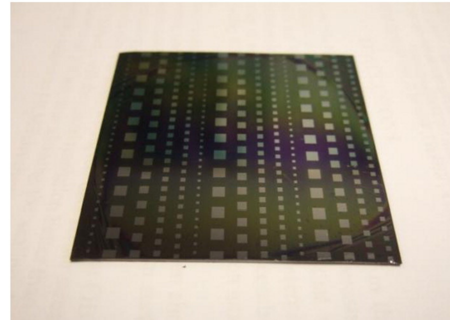

Fig. 2. Back side of back-patterned sample as received.

As a first step, the sample surface is etched for 1 min using HF to eliminate the native oxide layer formed on the opens due to air-contact. After this step, the sample is rinsed with deionized (DI) water.

The chemical reactions that govern the silicon KOH etching are presented below:

$$Si + 2OH^- + H_2O = SiO_3^= + 2H_2\uparrow$$
$$SiO_3^= + 2H_2O = SiO_3H_2\downarrow + 2OH^-.$$

The global process is described as follows:

$$Si + 3H_2O = SiO_3H_2\downarrow + 2H_2\uparrow.$$

As the global reaction equation predicts, there is some amount of hydrogen emerging from the etching process. This hydrogen comes out in the form of bubbles, which helps in monitoring the process (once the etching process has arrived at the SiN layer, the bubbles disappear).

The etching process takes between 7 and 8 h. Once it is completed, in order to remove the thin SiO_x layer between the Si and the SiN, a further etching step is applied using buffered oxide etching (6 parts 40% NH_4F and 1 part 49% HF). This etchant has a high selectivity for the silicon oxide compared to the nitride.

After the micromachining process, the sample is carefully cleaned, rinsing it several times in DI water. Once clean and dry (iso-propanol and a N_2 draft are used to help in removing the remaining water), the photoresist layer is removed by dipping the sample in acetone at 40 °C for 10 min.

Following this procedure two sizes of membranes are obtained. Taking into account the anisotropy of the etching (the new surface forms an angle of 54.7° with the surface of the sample, because KOH etching rate for Si is 400 times higher in the <100> directions than in the <111> directions) and the thickness of the Si wafer (750 μm), only the two biggest sets of openings, 1.5 × 1.5 mm and 2 × 2 mm, result in membranes, with a size of 460 × 460 μm and 960 × 960 μm membranes are obtained, respectively. All the membranes are composed of the corresponding ILD (of variable thickness for different samples) and a SiN layer 50 nm thick. Fig. 3 sketches the anisotropy of the etching process.

2.2. Testing

Samples prepared following the above mentioned procedure are tested to fracture. The tests are performed at the TriboIndenter™ (Hysitron Inc., USA), using a conical tip (with a curvature radius of 1.86 μm), recording the applied load and the displacement of the tip. The sample is placed with the openings facing down on the indenter stage (Fig. 4a), allowing the free deflection of the membrane. The transparency of the film makes it possible to easily position the indenter at the aimed point (Fig. 4b).

The spatial resolution of the position of the table in X–Y is better than 0.5 μm, allowing performing tests in a very repetitive way. Tests are conducted under displacement control up to the fracture of the membranes