

A comparative study of the strength of Si, SiN and SiC used at nanoscales

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Abstract—Microelectromechanical systems (MEMS) are being used in many critical applications that require very high stress levels. To properly design MEMS components, mechanical properties should be characterized testing relevant sized samples that are fabricated with the same procedures as the final structure. In this paper we compare the fracture strength statistics of 3 common MEMS materials, Si, SiC and SiN, with atomic force microscopy experiments.

Index Terms—MEMS, fracture, AFM, reliability, SiC, SiN

I. INTRODUCTION

ver the past decade, with the advances in integrated circuit (IC) fabrication methods, microsystem technology has attracted a significant commercial interest, and Microelectromechanical systems (MEMS) have been used in a wide variety of novel science and engineering products. The increasing tendency to create smaller MEMS devices that perform critical functions generally requires very high stress levels to be used. Hence, to use the full mechanical potential of a MEMS device, the failure mechanisms and the relation between environmental conditions and mechanical performance should be accurately characterized.

At MEMS size scales, the fracture properties of materials differ significantly from the well known bulk properties; they are influenced significantly by the sample size and often by small changes in the fabrication processes. As device dimensions decrease, the number of intrinsic volume defects such as dislocations and grain boundaries (for poly crystalline materials) decreases. Considering that fracture is related to the presence of surface and bulk defects [1,2], nanoscale materials may be able to sustain high stresses at low probability of failure. However, with decreasing sizes, the surface-to-volume ratio increases, surfaces and interfaces start to dominate material failure [3] and also the environmental conditions gain importance [4].

Hence, to properly design mechanical components for microsystems, fracture properties should be characterized testing relevant sized samples that are fabricated with the same procedures as the final structure.

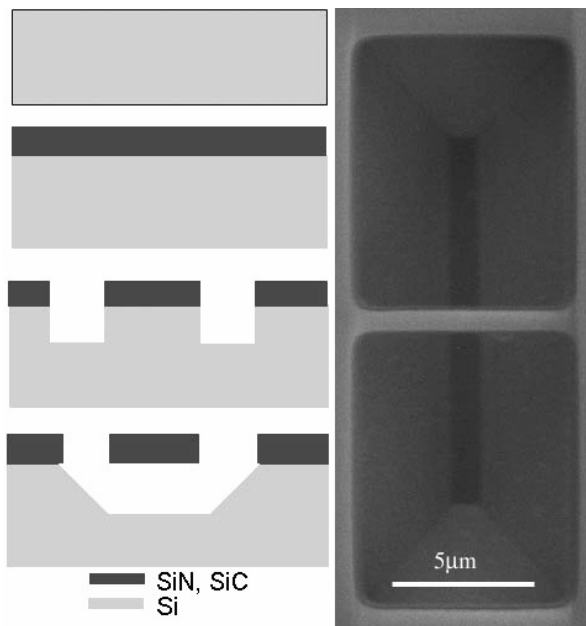
So far, numerous studies (with novel in-situ experimental procedures which generally consisted of modifications to well-established macroscale methods such as tensile and bending tests) have been performed to characterize the mechanical properties and fracture reliability of materials used in micro-nano size scales. The high displacement and load resolutions, necessary for nanoscale tests, can generally be accommodated by using commercially available piezoelectric actuators and high precision load cells. Yet, the handling of the test specimen remains as the major challenge; it is very difficult, if not impossible, to grip an individual micron-sized tensile test sample from both ends and to apply a load. A detailed review of previous experimental procedures can be found in [5].

Despite the large number of studies, due to differences in experimental methods, and the large scatter in observed values (for instance the measured strength of SiN films changes between 0.3 and 7 GPa [6]), it is still not possible to effectively compare the mechanical performance of different materials. In this study, we perform atomic force microscopy (AFM) experiments to characterize fracture strength statistics of SiN and SiC test beams. Using the same AFM method and similar sample shape and sizes, allows a direct comparison of the mechanical performance of different materials, which will be useful in determining the best material for different mechanical applications and also to validate the theoretical limits.

II. SAMPLE PREPARATION

The test samples are 200 nm thick (Si, SiC and SiN) doubly clamped beams that have an effective length changing between 5 to 8 microns and a width changing from 0.5 to 1 micron. The fabrication of the Si beams is explained in detail in [3]. The fabrication of the polycrystalline samples follows standard lithography and wet etching procedures and the basic steps are

Fig. 1. Schematic of the sample fabrication and an SEM micrograph of an 8 micron long SiN beam.

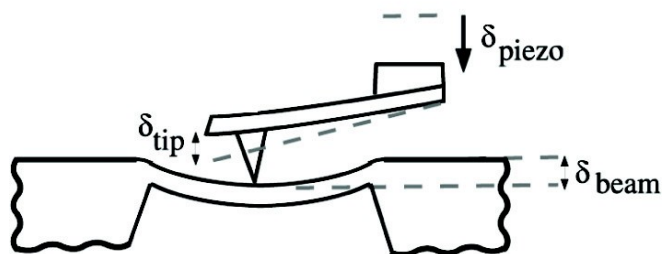


outlined in Fig. 1. Briefly, a 200 nm thick layer of SiN or SiC is deposited on a (100) oriented Si substrate with low pressure chemical vapor deposition (LPCVD). The film is patterned through photolithography and the beam shape is formed in the deposited layer through dry etching. The dry etch was continued further to create 5 micron deep trenches in the Si substrate. The wafers were diced and individual chips were then immersed in a 25% potassium hydroxide solution at 72°C for 5 minutes to completely release the test structures. The rounded beam corners and the small undercut originating from the wet etch helped to avoid geometric stress singularities and ensured that maximum stresses always occur at the center of the beam.

III. EXPERIMENTAL PROCEDURES

In our tests, we use AFM as a nanomanipulation tool. The test consists of using an uncoated, stiff, single-crystal Si AFM cantilever (Veeco TAP525) to push against the center of the doubly clamped test beams, bending them to the point of fracture, as sketched in Fig. 2. As a first step in the experiment, the position of the AFM cantilever tip relative to the test beam and the load application point must be determined. For this purpose, the beam is imaged by a contact mode scan and the geometric center of the beam (where the load is applied) is determined from the AFM image. Using the piezo controls, the cantilever tip is offset to the beam center accurately. Next, the beam is loaded. When the piezo scanner is extended by δ_{piezo} , the AFM cantilever pushes

Fig. 2. Schematic of an AFM cantilever loading the test beam.



against the beam, and the beam center is deflected by δ_{beam} , while the cantilever undergoes an upward displacement of $\delta_{cantilever}$.

The cantilever deflection, $\delta_{cantilever}$, is monitored with a split photodiode detector. Laser light from a solid state diode is reflected off the back of the AFM cantilever and collected at a position sensitive photodetector. The deflection of the cantilever results in a change in the output signal, which is used to measure deflection. Hence, before each experiment (every time a cantilever is attached to the piezo scanner), the relation between $\delta_{cantilever}$ and the photodetector signal, must be calibrated. Further details of the calibration procedures, can be found in [3].

The applied force, F , and the deflection of the beam, δ_{beam} , during loading are calculated as:

$$\delta_{beam} = \delta_{piezo} - \delta_{cantilever} \quad (1)$$

$$F = k\delta_{beam}, \quad (2)$$

Where, k is the stiffness of the AFM cantilever. Once the load and displacement at the fracture point was known, the strength of each beam is calculated through the simple beam theory, or in the case of large displacements, is inferred from a finite element simulation. The fracture strength was defined as the maximum stresses corresponding to failure load.

The main advantage of the AFM method is that it does not require a complex set-up. Any AFM with a closed-loop scanner and commercially available cantilevers can be used to repeat the tests in different environments. However, before testing, some design considerations should be taken into account to address the limitations of the method.

The deflection measurements are affected by the compliance of the AFM cantilever, the compliance of the tested sample and the limitations of the photodetector read-out. To be able to monitor the load and deflection of the test beams, the AFM cantilever used in the experiment has to be much stiffer than the cantilevers used for imaging. The calculated stiffness of a

Fig. 3. Weibull fracture probabilities for successive experiments for SiC, SiN and single crystal Si nanobeams.

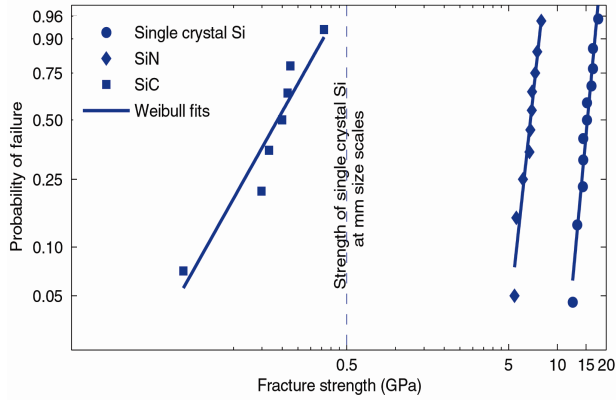


TABLE I
ESTIMATED WEIBULL PARAMETERS

Material	σ_0 (GPa)	m
SiC	0.23	1.86
SiN	7.08	9.64
Si	15.8	11.3

typical cantilever used in the experiments was approximately 260 N/m. On the other hand, no useful information could be extracted from an infinitely stiff cantilever.

IV. PRELIMINARY RESULTS

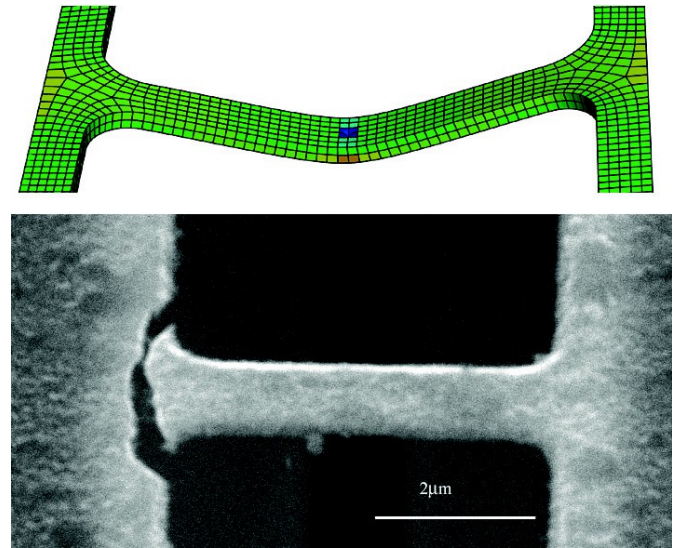
An ideal, defect-free material would always fracture at the same stress, due to breaking of bonds every time it is loaded. The failure of a real material however, is dominated by randomly distributed defects and hence the fracture properties must be treated statistically. The fracture strength of brittle materials is generally characterized by a Weibull probability distribution. Weibull distribution assumes “weakest-link failure”: i.e. the whole sample fails when a critically sized defect is encountered anywhere in the sample.

The corresponding cumulative probability of failure is a function of the stress, σ , and is expressed as

$$P_f(\sigma) = 1 - \exp(-(\sigma/\sigma_0)^\rho), \quad (3)$$

where σ_0 is the Weibull strength, and ρ is the Weibull shape parameter. The Weibull strength, σ_0 is the strength corresponding to an approximate 60% failure probability.

Fig.4. a) The shape of the deflected beam obtained by finite element analysis (colors indicate stress levels), b) a fractured SiC test beam.



Similarly, ρ controls the scatter of the distribution, with large scatter corresponding to small ρ .

After each set of experiments, the calculated fracture strengths were ordered, and each of the N strength values are assigned a failure probability $P_f(i)$ for $i=1..N$.

$$P_f(i) = (i-0.5)/N \quad (4)$$

The Weibull parameters σ_0 and ρ were then extracted by least-squares fitting the formula given in equation (3) to the assigned failure probabilities in equation (4). Fig. 3 shows the failure probability distribution of the polycrystalline SiN and SiC test beams in comparison with the failure probability of single crystal Si, tested previously and reported elsewhere [3]. The estimated Weibull parameters are summarized in Table 1.

V. DISCUSSION OF THE RESULTS

Our experiments show that both SiC and SiN samples fracture at stresses that are considerably lower than the very high strength values observed for single crystal Si. As seen in Table 1, the strength of the SiN is 60% less than that of the Si, measured at same length scales using the same AFM method. The SiC beams fracture at a much lower value, corresponding approximately to 1% of the single crystal Si strength. Similarly, there's a significant scatter in the observed strength values of the SiC samples while the least scatter is observed in Si.

The estimated strength of SiN is slightly higher than the previously reported values [6]. However, the estimated

Weibull strength of the SiC is even lower than the typical strength values observed at mm lengths scales, implying that the polycrystalline SiC structures fabricated using the same deposition parameters are not reliable for high-stress applications.

As shown in Fig. 4, all of the carbide samples, unlike the SiN beams, fractured from the corners. As indicated in section 2, the geometric properties of the beams were chosen to be free of major stress singularities. Hence, the fracture at the corners is related to either a process induced defect (that is continuously repeated) or the grain boundary acting as an infinitesimal crack. To further investigate the effects of grains, and possibly to improve the strength statistics, tests will be performed on SiC films with modified crystalline structures obtained using different annealing conditions.

In the present paper, we focused exclusively on the strength of structures that were not subjected to any external effects. Yet, to accurately determine the reliability of practical MEMS devices, the environmental conditions under which they operate should be taken into account. To characterize the effects of humidity on the strength statistics, samples that are currently being treated in a humidity chamber with 90% relative humidity, will be tested using the same experimental procedures.

VI.SUMMARY

In this paper, using atomic force microscope as a mechanical tester, we compared the fracture strength statistics of single crystal Si and polycrystalline SiC and SiN nanostructures. Our preliminary results showed, as expected, that both polycrystalline materials fracture at stresses that are lower than the strength of single crystal Si. However, the calculated strength of the SiC samples were much lower than expected, and the values had considerable scatter, indicating that at the present form they would not be the best candidates for high stress mechanical applications.

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