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Using microfabricated devices to determine the fracture strength of materials

Measuring the fracture strength of brittle materials is often difficult, because alignment of the load with the test specimen is critical. Any off-axis misalignment of the load will produce fracture in an undesired mode. This report describes several micrometer-scale devices, in which the "load cell" is fabricated simultaneously with the fracture mechanics specimen, so that precise alignment is assured. The devices utilize the residual stresses contained within the materials, which are deposited onto Si substrates, to create the loading forces and determine the fracture strength. The devices are passive, meaning that the fracture results are obtained immediately upon fabrication, and no external loading is required. Stress concentrations are generated at notches, and the fracture strength is determined by the critical stress required for failure. A variety of devices have been fabricated from materials of interest for microelectromechanical systems devices, including polysilicon, silicon nitride, and aluminum, with both tensile and compressive residual stresses.

Keywords: Fracture strength; Residual stress; Microelectromechanical systems

1. Introduction

The fracture strength of brittle materials is typically difficult to measure because perfect alignment of the load with the intended tensile axis of the material is required. In ductile materials, small amounts of plastic deformation can occur to compensate for any misalignment of the load, but in brittle materials this is not possible, and mixed-mode fracture will occur instead of the desired mode. This is particularly true for micrometer-sized specimens, whose small sizes make alignment even more complex. However, to investigate the fracture strength of materials such as polycrystalline silicon (polysilicon) deposited by low-pressure chemical vapor deposition (LPCVD), the specimens are limited to these types of dimensions. Fortunately, microelectromechanical systems (MEMS) fabrication procedures can be used to create devices that integrate the loading apparatus with the fracture specimen, ensuring precise alignment between the two components. This is accomplished by fabricating both components simultaneously on the same Si substrate.

In addition, deposition conditions can often be tuned to create residual stresses in the materials to be studied. This has been demonstrated for films deposited by LPCVD [1] and sputtering [2], among other techniques. Through proper device design, these residual stresses can be exploited to generate the loads required to test the fracture specimens. It is highly desirable that the strength measurements be made using "passive" devices – devices that perform their function upon release* with no further applied loads. Then, visual inspection is all that is needed to obtain the results. Rapid determination of fracture strength could then be achieved. Previously reported devices that fit this description are clamped–clamped beams (beams that are fixed to the substrate on both ends) produced from materials that contain residual tensile stresses [3–5]. Upon release, the residual tension creates stresses within straight beams [3], notched beams [4], or beams with sharp pre-cracks [5]. If the stresses are high enough, the beams will fail catastrophically, and if not, no changes will be observed. Given accurate knowledge of the residual stress in the material, finite element analysis (FEA) can predict the stresses in the device. Then, upper and lower bounds can be established for the strength of the material.

However, for a given device geometry, the usable range of residual stresses can be small. For some materials, the residual stress can be tailored by varying the deposition conditions, but in other cases, this ability is limited. In this report, we describe several passive device designs that can be used to determine the strength of materials with a wide range of residual stresses, including devices with both tensile and compressive residual stresses.

2. Experimental details

The devices reported here were all fabricated using standard surface micromachining technology [6]. For example, the devices shown in Figs. 1 and 2 were fabricated by

(i) depositing a ≈ 4 µm film of SiO_2 via LPCVD onto a 100 mm-diameter, 500 µm-thick Si substrate,
(ii) depositing a ≈ 2 µm film of polycrystalline silicon (polysilicon) via LPCVD onto the SiO_2,
(iii) patterning the device using optical photolithography, (iv) etching the polysilicon in a Cl_2 plasma,
(v) removing the photoresist in aqueous H_2SO_4/H_2O_2,
(vi) releasing the devices in aqueous HF.

The HF release etch is timed so that the SiO_2 beneath the narrow beams is fully removed, while some SiO_2 remains

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* "Release" is the term used in the MEMS community to denote the last stage in device fabrication. This invariably involves dissolution of a sacrificial material, SiO_2 in the case of polysilicon devices using hydrofluoric acid as the dissolution agent, so as to leave a device freely suspended as a cantilever [6].

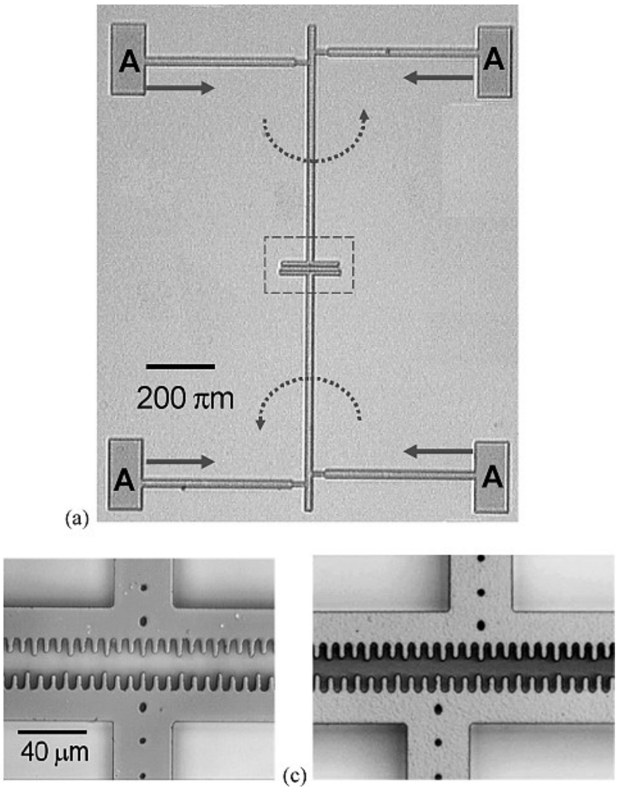

Fig. 1. Optical micrographs of a polysilicon rotating strain gauge. In (a), the anchors, labeled "A", are fixed to the substrate. If the polysilicon contains a compressive residual stress, it will expand, as indicated by the solid arrows, and the two central beams will rotate, as indicated by the dashed arrow. The vernier scale whose two parts are attached to the ends of the tow rotating beams is shown before release in (b) and after release in (c). The displacement after release is related to the residual stress in the polysilicon using finite element analysis, assuming a Young's modulus of polysilicon of 164 GPa [10].

beneath the large anchor pads at the ends of the beams, creating attachments to the substrate. The etch rate of polysilicon in aqueous HF is isotropic, ≈ 1 μm per minute. Device dimensions were determined using scanning electron microscopy (SEM). The residual stress of each material was quantified by laser wafer curvature measurements [7]. For the LPCVD polysilicon and silicon nitride films, the residual stresses were also determined using rotating microstrain gauges [8] fabricated on the same wafers, with good agreement between the two techniques. A polysilicon rotating microstrain gauge is shown in Fig. 1. Two-dimensional FEA was used to predict the stress concentrations at the notch roots.

3. Results and discussion

Figure 2 shows the device designed to investigate tensile strength for notched beams in materials with moderate tensile stresses. It consists of 500 μm long clamped–clamped beams with micromachined notches with root radii of 0.9 μm. The notches in the 13 beams increase in depth, a, from 3.2 μm to 15.2 μm, with 1 μm intervals. The device is fabricated from a 2 μm thick undoped polysilicon film deposited at 570 °C by LPCVD and subsequently annealed at 615 °C to produce a fine-grained microstructure [1] with a tensile residual stress of 318 MPa. Given the residual stress, the stress at each notch root is determined using finite element analysis. The SiO_2 remaining beneath the large anchor pads seen on both sides of the beams in Fig. 2a produces extremely stiff anchors. The fracture of any beam(s) does not affect the stress in the other beams.

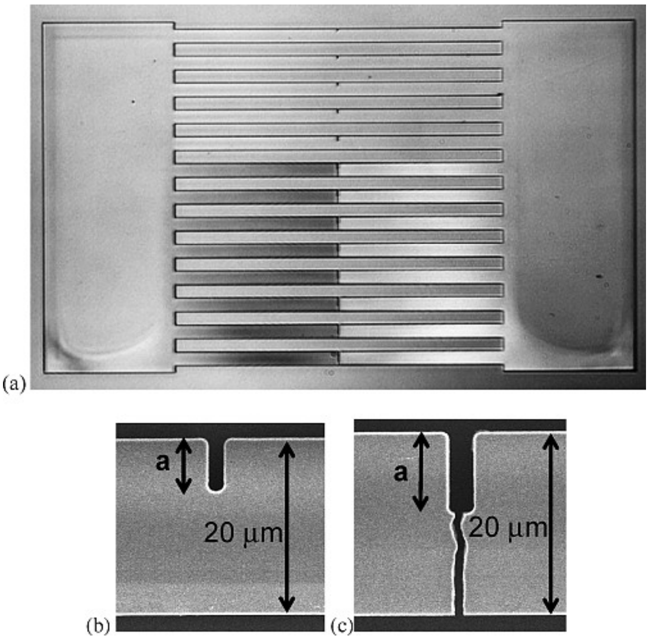

Fig. 2. (a) SEM micrograph of a polysilicon device comprising a series of 500 μm long clamped–clamped beams containing notches with increasing depths. (b) and (c) Higher magnification images of the 4th and 7th beams from the top.

As seen in Fig. 2a, the top 5 beams (with notch depths of 7.2 μm and smaller) survived after release, as illustrated by Fig. 2b, and the lower 8 beams (with notch depths of 8.2 μm and greater) failed after release, by Fig. 2c. Due to the stochastic nature of brittle failure and etching-induced flaws, several identical devices were fabricated and released. Out of 14 devices, the beam with $a = 8.2$ μm failed 11 times. Statistical analysis using the standard Weibull distribution for brittle fracture gave the fracture strength as 3.9 GPa. These results were taken immediately after release of the devices. With the same devices, delayed fracture could be studied as a function of time, humidity, temperature, or other factors.

Figure 3 shows the same device fabricated from a 1 μm thick sputtered Al film, with a residual tensile stress of 44 MPa. In this case, the fracture strength was determined to be 1.0 GPa. While the intact beam (Fig. 3b) shows a small amount of bending, the broken beam (Fig. 3c) does not. This implies that no plastic deformation occurred before fracture. Any plasticity would have altered the stresses in the beams. This could be investigated in greater detail with devices designed specifically for this purpose.

In Fig. 3, only the 2 beams with the deepest notches fractured upon release. If the residual stresses were slightly lower, none of the beams would have broken, and the fracture strength could not have been determined. For such low residual tensile stresses, the device shown in Fig. 4 has been designed. The beams are wider at the ends and narrower in the center. This effectively increases the stress in the central portion of the beams.

For the opposite case – very high tensile stresses – the device in Fig. 5 can be used. This device was fabricated from

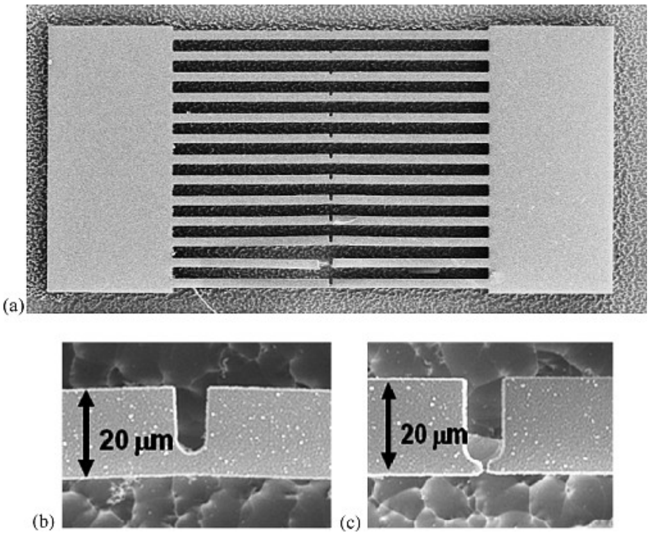

Fig. 3. (a) SEM micrograph of the same device shown in Fig. 1, made from aluminum. (b) and (c) Higher magnification images of the 11th and 13th beams from the top.

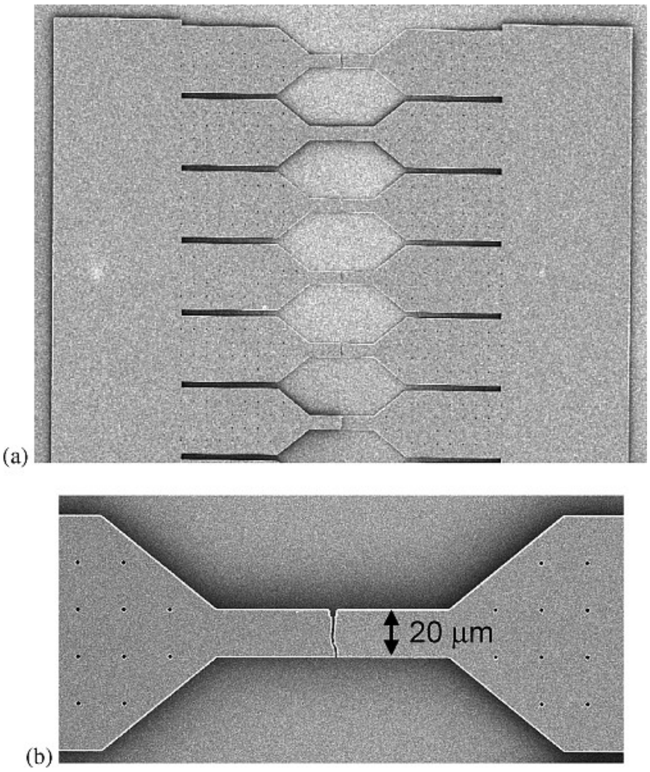

Fig. 4. (a) SEM micrograph of a device with tapering beams. The total beam length is 500 µm. (b) Higher magnification image showing the center of the top beam. The release holes are visible.

LPCVD Si_3N_4 with a residual tensile stress of 800 MPa. In a similar manner but opposite to the device of Fig. 4, the central portion of the beams, where the notches are located, are wider than the ends. This reduces the stress concentrations at the notch roots. An additional complication for this device is that the stress at the notch roots can be greater after partial release. That is, after the HF has dissolved the release oxide from beneath the sides of the beams, but a thin strip of SiO_2 remains beneath the central axis of the beam, the stress at the notch root can actually be higher than after the beam is fully released. To prevent this situation, the release holes visible in Fig. 5b were designed so that the last release oxide to be removed is located directly opposite the notch. FEA analysis shows that this geometry effectively shields the notch root from high stresses during the release process. The Si_3N_4 devices reveal a fracture strength of 6 GPa.

Finally, for materials with compressive residual stresses, the "Theta" specimen [9] can be used, in which compressive loading and Poisson expansion of a brittle sample shaped like the Greek letter theta leads to tensile stresses in the cross beam sufficiently large to cause tensile failure. An example is shown in Fig. 6a. In this device design, the connections between the anchors and the rest of the structure are small, to mimic the standard test for bulk materials and to better approximate the point load boundary condition. However, we find that after release, the devices had broken at these connection points, due to the fragility inherent to the brittle materials.

Therefore, the device shown in Fig. 6b was designed, which is the MEMS equivalent of the Theta specimen. Because the release holes are difficult to see in Fig. 6b, the anchors have been labeled. Upon release, the material expands due to its residual compression. The wide, long beams on the sides of the devices expand more than the thin, short beams in the center, leading to a tensile stress in the central beams. The notches are naturally located within the central beams. These devices were fabricated from LPCVD polysilicon deposited at 615 °C, with a columnar microstructure [1] and a compressive residual stress of -300 MPa.

From the notch depths required for failure, the fracture strength was determined to be 3.0 GPa. This is smaller than that measured for tensile fine-grained polysilicon. It is possible that the columnar microstructure creates a greater surface roughness and larger surface flaws when etched with

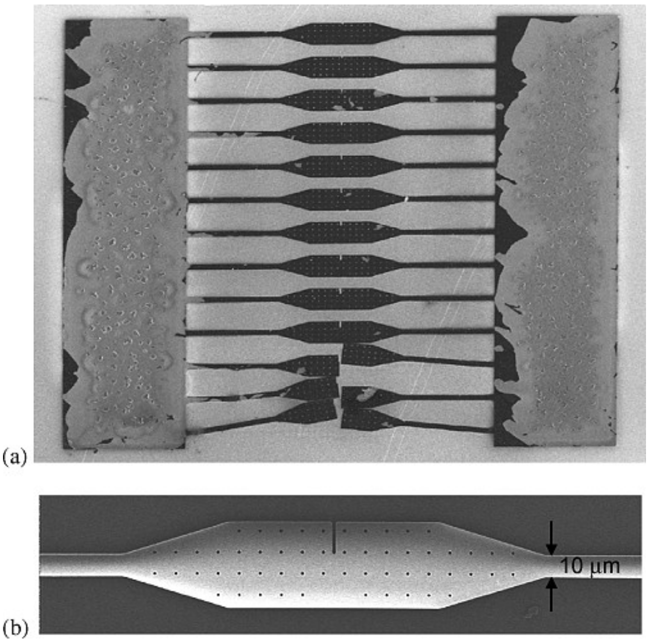

Fig. 5. (a) SEM micrograph of a Si_3N_4 device with beams whose width increases in the center. The total beam length is 500 µm. (b) Higher magnification image showing the center of one beam. The release holes are visible.

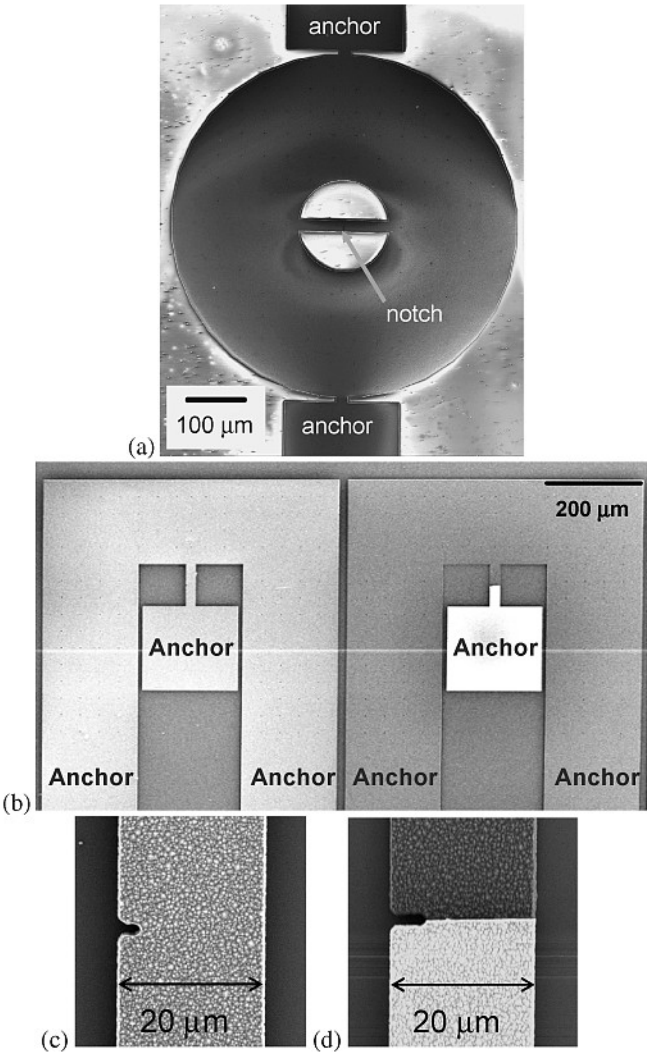

Fig. 6. (a) SEM micrograph of a microfabricated polysilicon Theta device. The connections to the anchors were found to be too fragile. (b) SEM micrograph of two alternate devices for determining fracture strength in compressive materials. (c) and (d) Higher magnification images showing the notched area of the two devices in (b). The device with the greater notch depth has failed.

Cl_2 plasma. It is also possible that these devices were inadvertently over-etched, which resulted in higher surface roughness.

4. Summary

Passive MEMS devices have been designed for determining the fracture strength of brittle materials. They appear also to be useful for metallic thin films. These devices use the residual stresses contained within the structural MEMS materials to generate stress concentrations at micromachined notch roots. By fabricating devices with a range of notch depths, in conjunction with FEA, the critical stress required for fracture can be determined. Various devices have been fabricated to measure the fracture strengths of materials with widely varying residual stresses, including devices with both tensile and compressive residual stresses.

5. Dedication

AHH is still grieving about the untimely early death of Rowland Cannon, who was his good friend and wonderful colleague for over forty years.

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