THERMO-MECHANICAL CHARACTERIZATION OF MATERIALS AT MICRO/NANOSCALE UNDER BENDING

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ABSTRACT

This paper presents a new method for testing materials at the microscale at high temperature under bending in-*situ* in SEM. The proposed method consists of a straining stage with built-in force and displacement sensors attached to a heating stage inside the SEM. The sample is co-fabricated with the stage to eliminate any misalignment error. The method is applied to test the strength of single crystal silicon (SCS) micro-beams under bending. At room temperature, bending tests revealed strengthening of SCS compared to that under uniform tension. This strengthening is contributed by stress localization near the surface of the beams close to the anchors, and the stress gradient from the surface to the neutral axis. The study further reveals significant reduction in the Brittle to Ductile Transition (BDT) temperature of SCS micro-beams compared to their bulk counterparts.

INTRODUCTION

Recently, considerable efforts to explore new materials for micro/nano-electromechanical systems (MEMS) were undertaken to improve their functionality. Novel testing techniques were invented to be able to test these materials at the micro/nanoscale. Uniaxial tension, compression, nano-indentation and bending tests were implemented to understand their mechanical properties. Most of the reported bending tests were carried out using Atomic Force Microscope (AFM) [1,2] or nanoindentation [3,4] where load is applied at the free end of a cantilever beam or in the middle of a fixed-fixed beam/nanowire. However, there are several uncertainties in the directionality and the location of the applied load [5,6], as well as misalignment errors [7]. In addition, handling of the samples in this small scale becomes significantly difficult. Recently, 3D printed prototypes were fabricated as a proof of concept, where an indenter can be used to test multiple samples under bending [8] and single specimen can be subjected to pure bending [9]. In the current design the sample is co-fabricated with the stage to avoid these shortcomings. The chip-based stage, actuated by a piezo-actuator, applies bending moments on micro/nanoscale beam specimens. The new stage minimizes uniaxial state of stress in the specimen, but maximizes bending stress over a small volume such that high stresses can be reached within a small volume on the specimen without a premature failure by fracture. In addition, a new setup is designed to test the samples at high temperature in-situ inside the SEM chamber. The chip including the specimen can be heated up to 500°C. The BDT temperature of silicon is explored using the proposed chip and a new in-*situ* setup inside the SEM.

BENDING STAGE DESIGN

Figure 1 shows the basic operation of the proposed bending stage. The bending specimen with a rectangular cross section is placed transverse to the straining direction (fig.1.b). The specimen (in the center) is pulled from one end. The other end of the specimen is connected to the force sensing beams (fig.1.b). The long two parallel soft beams (supporting beams) allow the motion of one of the specimen's ends in the x-direction. They serve as a soft anchor for the sample, allowing translation while preventing rotation at the end. The specimen is co-fabricated with the stage to avoid any misalignment problems.

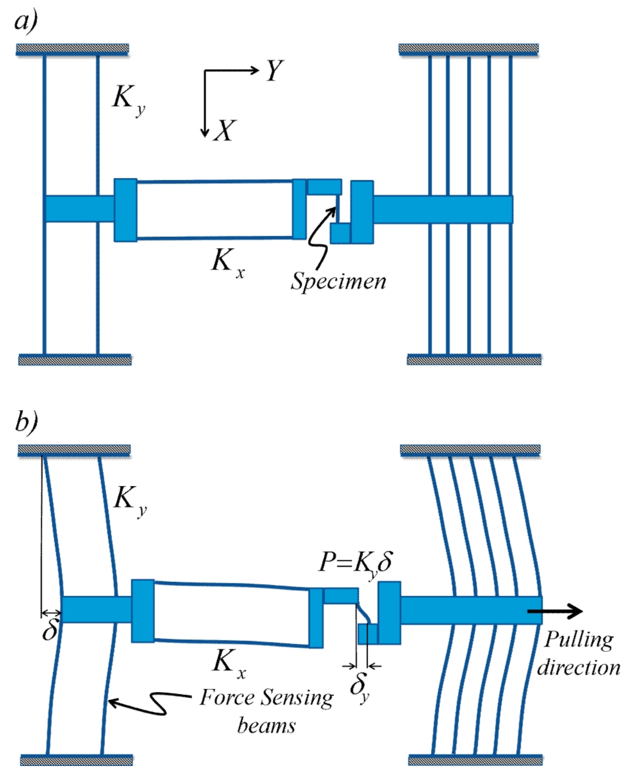

Figure 1 a) The proposed bending stage (no load applied) with the specimen in the center, and three sets of beams (springs), and b) loading the bending stage with all the beams deformed, where the deflection of the force sensing beams is used to measure the applied load ($P=K_y\delta_y$)

A schematic of the stage operation (fig.2) shows how the force sensing beams and the supporting beams (K_y and K_x, respectively) are deformed during loading. An analytical model was developed to attain the relationship between the applied load on the specimen and the total deformation. Equation 1 shows the relation between the applied load, elastic modulus of elasticity, moment of inertia, stiffness of the supporting beams, stiffness of the force sensing beams, and total deformation of the specimen along y-direction denoted by *P, E, I,* K_x, K_y, and δ_y, respectively. In addition, a finite element model of the stage was built to select the dimensions of the force sensing beams for specimens with different thicknesses. It was also used to calculate the stress values at the anchors.

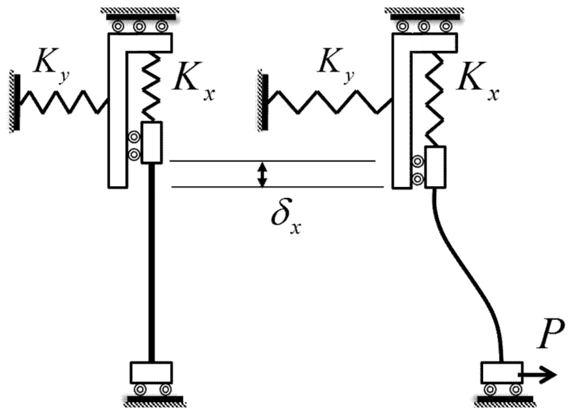

Figure 2 A schematic of the bending stage before and during loading (Deflections not to scale)

$$\frac{\pi^2}{128}\left(K_x+\frac{EI\pi^2}{2L^3}\right)\delta_y{}^3+\frac{P}{3}\delta_y{}^2+\frac{EI\pi^2}{8L}\delta_y-\frac{PL^2}{\pi^2}=0 \qquad (1)$$

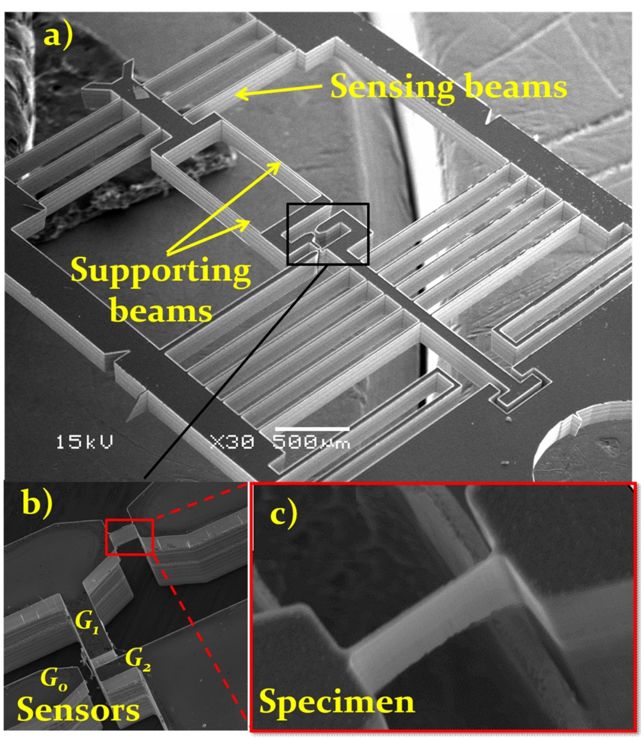

Figure 3 a) The fabricated bending stage with the force transmission mechanism, b) a zoom-in-view of the specimen and gauges, and c) the specimen

STAGE MICROFABRICATION

The microfabrication process flow is shown in fig.4. Two different masks are used during the fabrication. The backside mask doesn't have the specimen pattern, while the top mask has different specimen sizes. The etching rate used varies between 1 to 14 µm/min. The depth of the specimen is controlled by the first etching process. All devices were fabricated from 200 µm thick wafers. Samples with few microns thickness (>2µm) and depth (20-50µm) were fabricated.

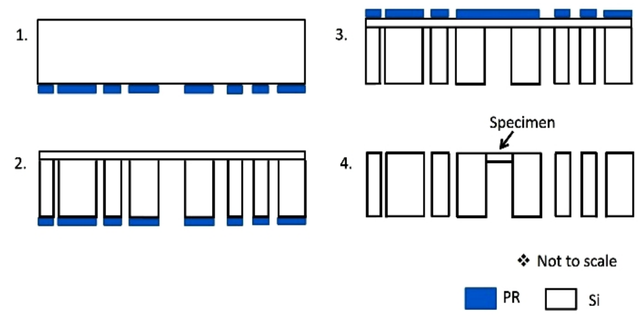

Figure 4 Fabrication process flow 1) patterning the backside, 2) DRIE etching, 3) Patterning the top side, and finally 4) DRIE etching the top side.

MICROMECHANICAL TESTING

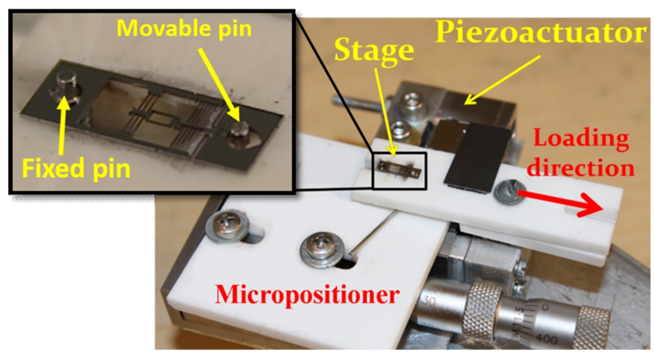

Figure 5 Testing setup showing the piezoactuator for stretching the bending chip and the micropositioner for adjusting the position of the chip before testing

In order to be able to measure the force and deflection of the specimen, sensor gauges were added at a close proximity from the specimen. Fig.3.b shows the gauges, where the change in distance between G_1 and G_0 is used to measure the load applied. The change in distance between G_1 and G_2 provides the total deflection of the specimen. The setup (fig.5) is used to test the stage at room temperature. Before testing, the micropositioner drags the stage until both pins touch the walls of the holes. The piezoactuator pulls one end of the stage during testing inside the SEM. The actuator is connected to a power source through a feedthrough on the back side of the SEM. The whole setup is placed inside the SEM and the specimen lies at a close proximity from the pole piece to be able to capture high resolution images of the gauges during the test (fig.6). For high temperature testing, a heater is used to heat the sample from the back, which allows sample temperature to reach 500°C. Thermocouples are connected to the stage to measure the temperature close to the specimen location.

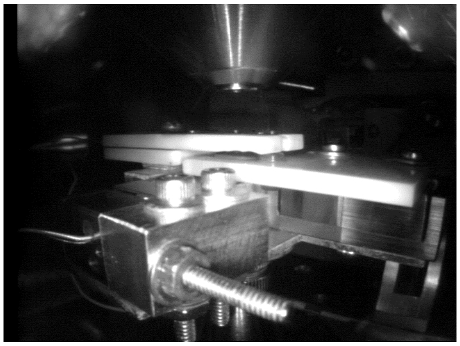

Figure 6 Test setup inside the SEM chamber during the bending test

RESULTS AND DISCUSSIONS

Guided by the analysis, the bending stage and the sample are designed and co-fabricated from a 200μm thick SCS wafer using lithography and DRIE as shown in (Fig. 4). The chip is stretched by applying voltage to the piezo-actuator inside scanning electron microscope (SEM) to bend the SCS specimen (32x39x2.5μm^3) at different temperatures. Images of the gauges were taken during the experiment to be analyzed later using image correlation software for load and deflection measurements. The elastic modulus of SCS along <110> at room temperature is found to be 169.9±5.8 GPa, in good agreement with literature [10]. The stress vs deformation shows a linear relation until fracture (fig.7). Samples showed brittle fracture usually near one of the anchors where the stress is maximum (fig.8).

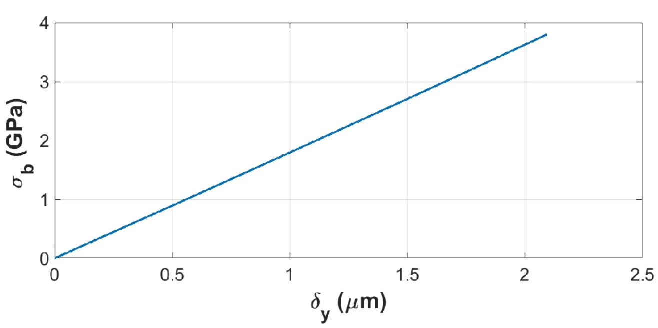

Figure 7 Bending stress measured at room temperature (brittle fracture)

The load-deflection relation during loading shows plastic deformation at 400°C, which is below the BDT temperature of bulk SCS (Fig. 9). Slip traces appeared on the sidewalls of the bent specimen. For the sample beam axis along [0 -1 1], slip occurs along four similar slip systems (e.g. (1 -1 1)[-1 0 1]), all with Schmid factor $1/\sqrt{6}$.

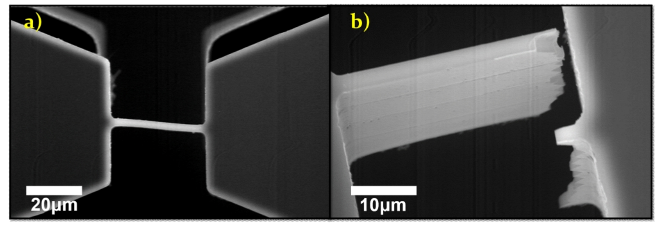

Figure 8 a) Specimen during loading, and b) broken sample (brittle fracture)

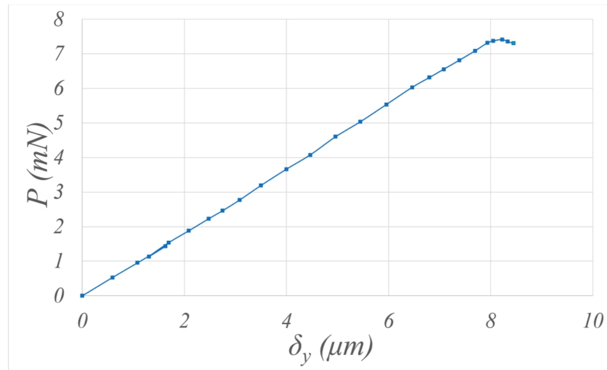

Figure 9 load-deformation relation for 2.5 μm thick sample at 400°C

CONCLUSIONS

The proposed on-chip bending stage offers a new method to test materials under strain gradient. To avoid handling and misalignment problems, the sample is co-fabricated with the stage. Microscale samples were tested at different temperatures using a new in-situ high temperature test setup inside SEM. The new stage maximizes the bending stress at the anchors and minimizes the uniaxial tension by implementing a new mechanism. The failure stress values at room temperature were found to be higher than the reported uniaxial tension test results. The high temperature tests revealed the size dependence of the BDT in silicon.

ACKNOWLEDGEMENTS

This project was funded by the National Science Foundation (NSF) award No.1562694. The single crystal silicon samples were fabricated in Micro-Nano-Mechanical-Systems Laboratory at University of Illinois Urbana Champaign (UIUC). The bending test was performed in the Microscopy Suite at Beckman Institute for Advanced Science and Technology at UIUC.

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