THERMO-MECHANICAL BEHAVIOR OF SILICON AT NANOSCALE – AN IN SITU INVESTIGATION

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DISSERTATION

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Abstract

Materials at the micrometer and submicrometer scale exhibit mechanical properties that are substantially different from bulk materials. With the increasing miniaturization of devices, accurate characterization of micro/nanoscale materials and fundamental understanding of their deformation mechanism are essential to ensure their reliability and performance. The application of the micro/nano devices are not limited to room temperature as those small devices are often required to operate in high temperature environment. The mechanical properties of micro- and nano-materials are expected to be highly temperature dependent, even more than those of their bulk counterparts. One of such behavior is size dependent brittle-to-ductile transition (BDT) in single crystal silicon (SCS). Several experimental and computational studies suggest that SCS can plastically deform near or even at room temperature with reduction of sample size. However size dependent BDT in SCS is not conclusive as there are controversial experimental results in the literature, i.e., no plastic deformation until brittle failure of a sample irrespective of the sample size. The foremost reason for the relatively limited available data and little understanding of the mechanisms of size dependent BDT is the lack of comprehensive and robust in situ experimental techniques. In particular, there has been no technique to perform in situ deformation experiments in SEM or TEM while simultaneously controlling both the key parameters influencing BDT, namely temperature and specimen size.

In this study we have developed novel methods to explore mechanical and thermo-mechanical behavior of micro/nano scale materials with a special emphasis on in situ study. The in situ material testing offers an attractive feature in studying of micro/nano materials
as it provides direct structure-property relationship due to ultra high resolution of Electron Microscopy observations. Using this unique in situ measurement ability, we have unambiguously explored size dependent thermo-mechanical behavior in micro/nano scale SCS samples. Our experimental investigation has revealed single crystal silicon, well known brittle material at bulk scale, can plastically deform at substantially lower temperature than well known bulk BDT temperature. For example, we have observed about 31% reduction in BDT temperature for sample size 0.72µm with respect to its bulk counterpart. The stronger surface effects at the micro/nano scale give raise to this unusual behavior. Also, we have, for the first time, considered size dependent yield strength of silicon samples incorporated with stress gradient and material characteristic of silicon due to strong covalent bond. For theoretical study of the size dependent yield behavior, we have employed an isotropic elastic continuum based model. The model shows that stress concentration due to dislocation pile-up decreases by up to 82% with larger stress gradient in a sample. Also, the model predicts that size dependence becomes more important for materials with large Peierls stress like SCS. We have experimentally confirmed substantial increase in yield strength with sample size using SCS samples.
To my family
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Table of Contents

List of Tables .................................................................................................................... ix
List of Figures ..................................................................................................................... x
List of Symbols and Abbreviations .................................................................................... xvi

Chapter 1 Introduction ....................................................................................................... 1
  1.1 Background ................................................................................................................. 1
  1.2 Organization of the Dissertation .................................................................................... 6

Chapter 2 Challenges in In Situ Uniaxial Mechanical Tests ............................................ 8
  2.1 Source of Nonuniform Stress ....................................................................................... 8
    2.1.1 Transverse misalignment ...................................................................................... 10
    2.1.2 Rotational misalignment ...................................................................................... 12
  2.2 Figures ........................................................................................................................ 16

Chapter 3 In Situ Mechanical Testing at Micro/Nanoscale: Theory ................................ 19
  3.1 Microscale tensile/compression stage ......................................................................... 19
    3.1.1 Force sensor ......................................................................................................... 20
    3.1.2 Finite Element Analysis ...................................................................................... 22
  3.2 Specimen with a Self-Aligning mechanism ................................................................ 24
    3.2.1 Hinge-like mechanism ....................................................................................... 24
    3.2.2 Analytical model .................................................................................................. 25
    3.2.3 Numerical model .................................................................................................. 26
  3.3 Conclusion .................................................................................................................... 29
  3.4 Figures ........................................................................................................................ 31

Chapter 4 In Situ Mechanical Testing at Micro/Nanoscale: Experiment ......................... 42
  4.1 In situ uniaxial testing apparatus ................................................................................ 42
    4.1.1 Experimental calibration for a force sensor ......................................................... 43
  4.2 Influence of misalignment ............................................................................................. 44
    4.2.1 Compliance of the stage ...................................................................................... 44
    4.2.2 Gripping mechanism ............................................................................................ 46
  4.3 Uniaxial loading .......................................................................................................... 48
    4.3.1 Self-aligning mechanism ..................................................................................... 48
4.3.2 Self-aligning sample ........................................ 50
4.4 Conclusion ..................................................... 52
4.5 Figures ......................................................... 54

Chapter 5  A novel method for in situ thermo-mechanical testing for micro/nanomaterials .......................... 65
5.1 A novel SiC MEMS stage ....................................... 65
  5.1.1 Temperature sensor: bimetallic type ..................... 67
  5.1.2 Analytical model ......................................... 68
  5.1.3 Numerical model ......................................... 70
5.2 In situ uniaxial measurement at high temperature ............... 72
  5.2.1 In situ temperature measurement ....................... 73
  5.2.2 In situ mechanical test of SCS samples at high temperature 74
5.3 Conclusion ..................................................... 77
5.4 Figures ......................................................... 78

Chapter 6  Size and temperature dependent BDT behavior in SCS .... 89
6.1 Experimental methods ......................................... 90
  6.1.1 A microdevice for thermo-mechanical measurement ...... 90
  6.1.2 SCS bending samples .................................... 91
6.2 Experimental Results and Discussions ......................... 92
  6.2.1 Thermomechanical response of SCS bending samples .... 92
  6.2.2 Mechanism for size and temperature dependent BDT ...... 94
6.3 Conclusion ..................................................... 97
6.4 Figures ......................................................... 98

Chapter 7  Stress Gradient Plasticity in SCS ..................... 102
7.1 Effect of Stress Gradient and Size on Plasticity in SCS ........ 103
  7.1.1 Stress gradient plasticity in SCS ....................... 103
  7.1.2 Dislocation density with stress gradient ............... 106
  7.1.3 Effect of stress gradient on stress concentration of dislocation pile-up 108
7.2 In situ thermo-mechanical experiment ........................ 111
  7.2.1 Force-displacement relation: elastic deformation ....... 111
  7.2.2 Force-displacement relation: elastic-plastic deformation 113
  7.2.3 Yield Strength Measurement ............................ 115
7.3 Conclusion ..................................................... 115
7.4 Figures ......................................................... 117

Chapter 8  Conclusions and Future Research .................... 122
8.1 Conclusion ..................................................... 122
8.2 Recommended future work ................................... 124
8.3 Discussion on potential research: Materials in Extreme Environments 125
8.4 Figures ......................................................... 127

References ........................................................ 128
List of Tables

3.1 Comparison between the hinged and plain samples at $f^* = fl^2/(EI) = 20$. 28

7.1 The summary for the values of $\tau_{peierls} = \tau_{peierls}/\tau_N$ and $l^* = l/b$ for $\tau_s^* = \tau_s/\tau_N = 1.5$ .......................... 110
List of Figures

2.1 The scenarios of misalignment between loading, $f$, and a specimen: (a) ideal loading, (b) transverse misalignment, and (c) rotational misalignment. The degree of misalignment for transverse and rotational misalignments are quantified by $c$ and $\theta_o$, respectively. The free body diagrams of the representative cases are shown in (d), (e), and (f) for ideal loading, transverse misalignment, and rotational misalignment, respectively. .................................................. 16

2.2 Normalized non-uniform stress error $\frac{h}{6c}c_{TM}$ as a function of $x^*$ at the top of the sample with variation of $f^* = fL^2/(EI)$ from 0 to $10^4$. .................. 17

2.3 Non-uniform stress error $c_{RM} = \sigma_B/\sigma_I$ at the bottom of the sample as a function of $x^*$ for rotational misalignment $\theta_o$ with variation of (a) $0 \leq \theta_o \leq \pi/12$, (b) $1/100 \leq h^* \leq 1$, and (c) $10^{-4}\% \leq \varepsilon_I \leq 5\%$. .................. 18

3.1 The schematic of the uniaxial testing stage: (a) 3-dimensional solid model and (b)-(c) grips on the stage and a sample before and after assembly. .... 31

3.2 Solid models to study influence of stage compliance and gripping between the sample and the stage on misalignment errors: (a) overall view of one-to-one scale solid modeling of the tensile stage for FEA, (b) zoom-in view of the sample upon loading, and (c) the schematic of the initial gripping condition. Dotted line in (c) shows the tapered profile of the specimen. .................. 32

3.3 The schematic of gripping errors: (a) inclined sidewall profile, (b) full-engagement due to critical force $f_{cr}$, (c) asperity contact, and (d) full-engagement. The magnitude of gripping errors is defined by $\theta_g$. ................................. 33

3.4 The sequences of the FEA results for a tapered specimen: (a) the strain evolution with incremental increase of the pin-hole distance (a1)-(a5) and (b) the corresponding stress-strain response at the mid-length of the sample. $\sigma_{ave}$ is the average stress of the top and bottom of the sample. ................................. 34

3.5 The schematic of the loading mechanism from side view: (a) initial loading and (b) reverse of bending in the sample with increase of loading. The asymmetric location of the sample, near the top of the stage, and the tapered surface on the sample induce $M_{st}$ and $M_{sp}$, respectively. $M_{sp}$ and $M_{st}$ reduce the surface contact angle, $\theta_g$, between the specimen and the grip continuously and result in a shift of contact toward the bottom. ................................. 35

3.6 The schematic of the self-aligning specimen: (a) overall 3-dimensional view, (b) top view, and (c) side view. GP indicates parts that are engaged by grips. 36
3.7 The schematic of samples: (a) a hinged sample and (b) a sample without any hinge. The load is applied on both the samples with misalignment $c$.  

3.8 Analytical results of the hinged and plain samples subjected to the transverse misalignment. In (a), deformed shape of the hinged and plain samples is shown for $f^* = \frac{f^2}{(EI)} = 10, c^* = 0.1$, and $l_1^* = 1/3$ with variation of $I^* = I/I_1$. The red line represents deformed shape of a plain sample from Eq. 2.12. In (b), $\eta = (d^2X^*/dx^2)/(d^2X^*/dx^2)$ is shown as a function of $I^*$ for $0 \leq l_1^* = l_1/l \leq 0.4$ at $x^* = 1/2$ upon loading with $f^* = 10$ and $c^* = 1/10$. $\eta$ directly compares bending moments in the hinged and plain samples.  

3.9 FEA results for the hinged sample and the plain sample with $l_1^* = l_1/l = 1/3, I^* = I/I_1 = 9$, and $\theta_g = 0.037$ rad. In (a), the transition from $\alpha < 0$ to $\alpha > 0$ is observed near $\varepsilon_1 \approx 1.5\%$ for the plain sample where $\alpha = d\varepsilon_{\text{bottom}}/d\varepsilon_{\text{top}}$. Figure (b) shows the zoomed in view of (a). The transition for the hinged sample occurs between $\varepsilon_1 \approx 0.1\%$.  

3.10 Analytical results for the hinged sample and the plain sample subjected to the eccentricity $c = h/2$ with $l_1^* = l_1/l = 1/3$ and $I^* = I/I_1 = 9$ (refer Fig. 3.7). In (a), the transition from $d\varepsilon_{\text{bottom}}/d\varepsilon_{\text{top}} < 0$ to $> 0$ is observed near $\varepsilon_1 \approx 5\%$ for the plain sample. Figure (b) shows the enlargement of (a). The transition for the hinged sample occurs about $\varepsilon_1 \approx 1.5\%$.  

3.11 $\epsilon_m = \sigma_B/\sigma_1$ at the top of the samples for the hinged and plain samples at $x^* = l_1^*$ and $= 1/2$ as a function of $f^*$ when $c = h/2$.  

4.1 The schematic of fabrication flow: (a) Al deposition and photoresist (PR) spin-coating on both sides of a silicon wafer, (b) patterning PR layers by lithography on both sides, (c) patterning the Al films by wet etching, (d) ICP-DRIE etching to make the grooves of grips, (e) to release free-standing structure, and (e) removal of the Al film and PR.  

4.2 The calibration of the force sensor. The solid circles show experimentally measured force-displacement response. The experimental data is fitted by $f = \bar{k}\delta + \bar{k}_3\delta^3$ and linear and nonlinear spring constants are obtained. Also, the spring stiffness of the force sensing beams is predicted by a linear ($\bar{k}\delta$) and a nonlinear ($\bar{k}\delta + \bar{k}_3\delta^3$) approximations (with $\bar{k}$ and $\bar{k}_3$ predicted from geometry and elastic property of single crystal silicon).  

4.3 SEM images of the tensile stage and the specimen: (a) overall view of the stage, (b) a specimen mounted on the grips, (c) image tracking marks on the specimen (dotted circles) and on the stage (solid circle on grip and dash-dot circle on force sensing gauge), and (d) a image of an assembly using the micromanipulator (FIB omniprobe). The specimen is glued to the manipulator by Pt deposition. After the assembly, the specimen is released from the manipulator by ion milling.  

4.4 Experimental results for a 30$\mu$m-long specimen: (a) stress-strain response and (b) strain ratio between $\varepsilon_{\text{bottom}}$ and $\varepsilon_{\text{top}}$. In (a) $E_{\text{mean}} = \sigma/\varepsilon_{\text{ave}}$ where $\varepsilon_{\text{ave}} = (\varepsilon_{\text{top}} + \varepsilon_{\text{bottom}})/2$.  

4.5 A SEM image of tapered sidewall profile due to ICP-DRIE.
4.6 SEM images of the modified tensile stage: (a) overall view of the stage and the specimen, (b) 90µm-deep groove of a grip, (c) specimen with strain measurement gauges, (d) image tracking marks on the strain measurement gauges (dotted circle) and on the stage (solid circle on grip and dash-dot circle on the force sensing gauge).

4.7 Experimental and analytical results for a 540µm-long specimen with cross section area \( A = 77\mu m^2 \). Stress-strain response and strain ratio between \( \varepsilon_{\text{bottom}} \) and \( \varepsilon_{\text{top}} \) from experimental measurement are shown in (a) and (b).

4.8 (a) The schematic of the grips and sample. (b) The strain of the sample and stage from the side view (see (a)) by using a finite element analysis as in the previous chapter.

4.9 The self-aligning specimen: (a)-(b) the schematic for top view and side view, (c) a SEM image of the sample loaded on the stage, and (d) zoom in view of the sample.

4.10 (a)-(b) Schematics of self-aligning and plain specimens. (c) Transverse deformation \((\chi)\) of the specimens due to \( f \) with eccentricity \( c \) as a function of axial coordinate of the specimens \((x)\). Applied loads are corresponding to 0.01%, 0.05% and 0.2% strains. (d) Normalized bending stress by uniaxial loading as a function of \( x \). The dimension of the specimen in (c) and (d) is \( h_{\text{gauge}}/l = 1/50 \) and \( l_{\text{hinge}}/l = 1/3 \).

4.11 The experimental and analytical results for a self-aligning sample. The gauge length is 50µm with cross section area 90µm². Stress-strain response and strain ratio between \( \varepsilon_{\text{bottom}} \) and \( \varepsilon_{\text{top}} \) from experimental measurement are shown in (a) and (b). The strain ratio by using analytical model in Section 3.2 is shown in (c). (b1) and (b2) are the specimen with \( I^* = 1.5 \) and \( = 23 \), respectively, where \( I^* = I_1/I \). Note that stress-strain responses upon loading and unloading designated by dotted lines in (a) follow linear relation consistently.

5.1 Three dimensional schematics of the SiC stage: (a) overall view of the SiC stage (b) experimental setup for in situ uniaxial tests, (c)-(d) are enlargement of C in (b) which show a sample before and after assembly with the stage, respectively. In (a), zoom-in view of a bi-metal type temperature sensor is shown. In (b), the stage is thermally isolated from an SEM holder sample holder by a macor heat insulator. The metal pillars are connected to copper wires so that electrical potential can directly be applied to the stage for resistive heating.

5.2 (a) The schematic and theoretical prediction of the SiC-Pt temperature sensor with temperature. (b1) the 3-dimensional schematic of bimorph sensors in series for the resolution enhancement, (b2) an FEA result for mechanical displacement of the sensor at 700°C, and (b) deformation-temperature at A, B, C, and D. In (b2), red, yellow, green, and blue indicate small to large displacement of the sensor, respectively.
5.3 (a) The schematic of the SiC stage for electrical connection through the stage and (b) corresponding lumped model. Electrical power cross (c) U-beams, (d) the frames of the SiC stage, and (e) the sample. In (f), the electrical power at the sample as a function of $R_{DE}$ is shown. The indicated data point in (f) corresponds to the maximum electrical power in (e).  

5.4 A finite element analysis for the SiC stage with metal pillars and a macor frame: (a) electrical (b) thermal, and (c) mechanical responses of the system at 50 V.  

5.5 FEA analysis (a) Variation of temperature across the sample length at 50V (between A and B) and (b) temperature at the sensor at C and at the ends (at A and B) of the sample as a function of input voltage (V).  

5.6 Fabrication flow of a SiC stage for high temperature in situ uniaxial tests. From a 256 $\mu$m-thick SiC wafer in (a), trench and through holes are created by using laser milling from the back side of the wafer to reduce height of a temperature sensing beam and to create alignment marks for the front side fabrication (see (b) and (f)). In (c), all inner structures, such as supporting beams, force sensing beams, and U-beams, of the SiC are fabricated by laser milling from the front side of the wafer. In order to align the backside trench with the temperature sensing beam on the front side, the alignment marks in (f) are used. During laser milling, SiO$_2$ deposition occurs (see (c) and (g)) which is cleaned by HF wet etching as shown in (d) and (h). The grooves of grips for sample gripping are etched and Pt is deposited to make a bimetal temperature sensor in (e) by using focused ion beam (FIB) etching and deposition, respectively. Finally, the outer frame of the SiC stage is cut by laser milling.  

5.7 The images of the SiC stage, SCS sample, and linear stage: SEM images of (a) overall view of the SiC stage, (b) the SCS sample and (c) zoom-in view of a built-in bimaterial sensor with Pt-Si junctions, and (d) the image of the SiC stage on a linear stage where I, II, III, and IV designate the SiC stage, piezoaetuator, water cooling block, and macor frames, respectively.  

5.8 In situ calibration of a temperature sensor: (a) crucible temperature with increase in ESEM heater temperature and (b) displacement of a bimaterial sensor on the SiC stage as a function of temperature. We melt a piece of tin (b1) near one of two grips and glass (b2) near U-beams.  

5.9 Uniaxiality of loading during in situ mechanical testing.  

5.10 Stress-strain responses of the SCS sample at different temperatures (room temperature to 403$^\circ$C).  

5.11 (a) Temperature at the piezoactuator with and without water cooling as a function of temperature at the SiC stage and (b) temperature at the stage (circle) and sample (square) with applied voltage.
6.1 Experimental method for in situ thermo-mechanical test: (a) an SEM image of the MEMS stage for in situ test, (b) zoom-in view (Area C) of the silicon bending sample on the MEMS stage, (c) the schematic of the bending sample, (d) a force($f$)-displacement($\delta$) relation, (e) a schematic of a cantilever beam loaded by $F$, and (f1)-(f2) zoom-in view of area A in (b). During in situ test, the sample displacement($\delta$) and applied force($f$) are measured by analyzing high resolution SEM images as in (d). Displacement measurement is done without exposing the bending arms to electron beams during in situ test by measuring $\delta$ away from the bending arms (Area A in (b)).

6.2 Force-displacement responses of single crystal silicon samples with variation of sample size and temperature: (a) $h = 8.7\, \mu m$, (b) $h = 1.5\, \mu m$, and (c) $h = 0.72\, \mu m$. As shown in (d) for all sample sizes, the plasticity is observed at lower temperature with reduction of sample size.

6.3 The SEM images of the SCS bending sample: (a) initial configuration without any mechanical loading, (b) zoom-in view of Area A, (c) plastic deformation of the bending arm after complete unloading of the sample, and (d) zoom-in view of Area B after material failure of the sample. Note that permanent curvature after failure in (d) indicates considerable plastic deformation with respect to the reference lines in (b) and (d), respectively.

6.4 Mechanism for size and temperature dependent brittle-to-ductile transition (BDT) in silicon. (a) the schematic of the dislocation nucleation from the sources in the volume and at the surface. (b)-(c) theoretical predictions of the temperature for BDT in the sample as a function of the radius of the cylinder and the ratios of activation energies for dislocation nucleation from surface and bulk, and the ratio of the constants $f_S/f_V$.

7.1 The schematic of surface dislocation nucleation in SCS. The SCS sample with the length ($L$) and thickness ($d$) is shown in (a). The sample is subjected to pure bending moment $M$. The zoom-in view (b) of area C in (a). Dislocations nucleate from A and move along slip plane (A-B). $s$ is a coordinate along the slip direction from A. $\Delta L$ is a mean average distance between slip bands. (c) shows stress state in the sample due to $M$. In (d), the resolved shear stress along $s$-coordinate is shown.

7.2 (a) and (b) show dislocation density $n^*(s) = n(s)/P$ for $\tau_{cr}/\tau_s = 0$ and $= 1$ cases within $1 \geq s/(l_{cr}/2) \geq -1$, respectively, where $P = 2(1-\nu)/Gb$. (a) shows nondimensional resolved shear stress ($\tau_{Lead}^* = \tau_{Lead}/Q$) at the leading dislocation of dislocation pile up ($s = -l_{cr}/2$) as a function of stress gradient ($1 \geq \tau_{cr}/\tau_s \geq 0$) where $Q = (1-\nu)\tau_s^2l_{cr}/Gb$. (b) shows nondimensional yield stress ($\tau_s^* = \tau_s/\tau_N$) as a function of non dimensional sample size ($l^* = l/b$) with variation of nondimensional Peierls stress ($\tau_{peierls}^* = \tau_{peierls}/\tau_N$).
7.4 The schematic of bending sample: (a) overall structure of the sample with eight bending arms, (b) free body diagram of the upper part of the sample (see Area A in (a)), (c) bending stress state at cross section p in (b) due to elastic and elastic-plastic deformation of the sample, (d) the sample geometry at the designated area in (b). .......................................................... 120

7.5 (a) Measurement of yield strength for SCS samples using a least squares fitting between the experimental data and the elastic-plastic force-displacement model for $h = 720nm$ sample. (b) shows yield stress as a function of sample size using the least squares fitting............................................. 121

8.1 Further investigation of the surface effect: (a) a unpassivated sample, (b) SiO$_2$ passivation with the cracks, and (c) Si$_3$N$_4$ passivation........................................... 127
List of Symbols and Abbreviations

MEMS microelectromechanical systems
NEMS Nanoelectromechanical systems
SCS single crystal silicon
SEM scanning electron microscope/microscopy
TEM transmission electron microscope/microscopy
BDT brittle-to-ductile transition
Chapter 1

Introduction

1.1 Background

With the expanding applications of small scale mechanical systems such as MEMS, NEMS, and bio-MEMS[1–6], mechanical behavior of micro/nanoscale materials has become increasingly relevant. But because of the size dependence of material behavior, bulk material properties cannot be directly extrapolated to micro and nanoscale. The differences in the mechanical behavior of macro and micro/nanoscale specimens arise primarily due to the changes in the material deformation mechanisms. Investigations have revealed several unconventional mechanisms, especially in the plastic deformation of metals, at the micro and nanoscale. Some prominent examples include dislocation channeling in thin metal films on substrates [7], dislocation starvation [8] and dislocation nucleation/escape [9] in submicron single crystal specimens. In addition, reduction in microstructural size leads to unusual properties such as plastic strain recovery irrespective of specimen size [10, 11]. More comprehensive details on the size dependent material properties and underlying mechanism of the material deformation are discussed in [12–15].

In order to explore the size dependent material properties, several methods such as resonance test[16, 17], bending test[18, 19], bulge test[20], nanoindentation test[21, 22], and uniaxial test[23–25] have been proposed. A detailed review of these methods can be found elsewhere [26–28]. Among the various material testing methods, one of the unique advantages of uniaxial tension and compression tests is that they provide a direct measure of mechanical properties such as Young’s modulus without any apriori model. However, car-
rying out uniaxial tests at the micro/nanoscale involves several challenges including: (1) fabrication and handling of a free-standing specimen, (2) application of small forces, (3) high resolution for stress and strain measurements, (4) gripping of the samples, and (5) uniaxiality of loading. Ruud et al.[29] and Sharpe et al.[30] overcame these challenges by using macro-scale instruments to apply load on microscale samples. However, the large size of their instruments precluded in situ mechanical testing in analytical chambers such as scanning or transmission electron microscopes (SEM or TEM). In situ testing is especially attractive for micro/nanoscale samples because one can monitor the overall macroscopic response while simultaneously observing the underlying deformation mechanisms and thus establish the structure-property relationship. In this work we give special attention to such in situ uniaxial mechanical testing of micro/nanoscale materials in SEM/TEM.

For in situ testing of nanoscale thin films in SEM/TEM, Haque et al. [31] developed a MEMS based uniaxial testing stage. In their MEMS stage, tensile force is applied on the specimen by imposing a displacement at one end of the stage using a piezoactuator while the other end is held fixed. The stress-strain response is monitored by taking a series of in situ SEM or TEM images. Using the stage, Haque et al. observed reduced elastic modulus, nonlinear elasticity, lack of work hardening, and low failure strain in aluminum and gold thin films with average grain size $< 50$ nm [31]. Zhu et al. [32] studied size dependent mechanical response of nanowires in TEM, also using a MEMS testing platform. In this case, load is induced by a comb drive electrostatic actuator or an in-plane thermal actuator and measured by integrated capacitors. This MEMS stage was used by Agrawal et al. [33] to study fracture properties of ZnO nanowires. They found that the fracture strains of these nanowires are about five times larger than those of bulk or thin film ZnO. Kiener et. al. [34] carried out tensile testing of miniaturized single crystal copper specimens in an SEM to study size-dependent crystal plasticity. They used a tungsten grip to apply deformation on dog-bone shaped samples and observed an increase of the flow stress with decreasing diameter. The dog-bone shaped samples and the tungsten grip were both fabricated by
focused ion beam milling.

For in situ compression of material samples in SEM, Uchic et. al. [24, 35] developed a micropillar compression test. The material sample, a circular micropillar, was fabricated by focused ion beam milling and loaded by a nanoindenter with a flat diamond tip. The stress-strain response was directly obtained from the nanoindenter. By employing this method, a substantial increase in flow stress for single crystal gold micropillars was observed [36, 37]. The authors argued that the observed hardening in the submicron scale samples is due to dislocation starvation whereby dislocations rapidly leave the sample before multiplication can occur. Thus, further plastic deformation requires nucleation of new dislocations leading to an increase in flow stress. Note that micropillar experiment requires careful precaution during sample preparation by using FIB milling, one of common methods. For example, ion beam milling can result in artificial defects in the test sample such as implantation of gallium ion, formation of an amorphous layer, and surface roughing which may alter material responses. [38] More extensive studies on the effect of FIB milling on the material response during microcompression test can be found in [39–42].

Among the various uniaxial in situ testing methods outlined earlier, the MEMS stage developed by Haque et al. [31] for thin films included an active self-aligning mechanism to ensure uniaxial loading. They suppressed the effect of misalignment errors by cofabricating the stage and the sample and introducing U-beams and support beams that automatically align the sample with the loading direction. This self-aligning mechanism reduced misalignment errors by 6 orders of magnitude (18° loading alignment error is reduced to $1.33 \times 10^{-5}$ degrees misalignment at the grips [23]).

Following the work of Haque et al. [31], Han and Saif developed a simpler procedure [43], consisting of fewer lithography and etching steps, to fabricate MEMS tensile testing stages which had the added feature of measuring the electrical properties of thin film samples [44]. Utilizing these stages Rajagopalan et al. [10] showed that nanocrystalline metal films (grain size 50-100 nm) recover a substantial portion of their plastic strain after deformation.
They explained this unexpected recovery in terms of inhomogenous deformation caused by an interplay between microstructural size and heterogeneity at the nanoscale [45]. In situ XRD experiments on bulk nanocrystalline aluminum specimens have confirmed that this unusual strain recovery is intrinsic to microstructurally heterogenous nanocrystalline metals, irrespective of their geometry and dimension [11]. While the MEMS based uniaxial testing methods by Haque et al and Han et al [31, 43] have many advantages, the method is restricted to thin film materials that can be cofabricated with the loading stage. Hence it limits the choice of materials and sample geometries that can be tested. In this work, we will present a novel in situ method to overcome these limitations (see Chapter 3 and 4).

As discussed so far, size dependent material properties at room temperature have been extensively studied by various in situ methods [24, 27, 32, 34, 46, 47]. However, the application of the micro/nanoscale devices is not limited to room temperature, but they may be subject to high temperatures, e.g., in microturbines [48], micropower generators [49], and sensors/actuators [50] in automobile and aerospace applications. At the micro/nanoscale, it is expected that material response depends on size and temperature.

One of such thermo-mechanical properties is the reduction in brittle-to-ductile transition (BDT) temperature with size. For example, Nakao et al. [51] carried out three points bending tests directly on a hot plate by using microscale single crystal silicon (SCS) samples (4µm in thickness) and observed significant reduction in BDT temperature [51]. The fracture toughness of a microscale silicon sample increased rapidly as temperature approached 70°C while BDT for bulk SCS occurs at about 550°C [52]. Their postmortem observation of the fractured surface in SEM revealed that BDT is triggered by a rapid increase in dislocation density and subsequent motion of dislocations. Furthermore, Han et al. [53] carried out in situ tensile strain test in TEM and reported ductile deformation of SCS nanowires with diameter less than 60nm at room temperature. Östlund et al. [54] carried out micropillar compression test with variation of sample diameter and observed ductility in SCS samples with <300-400nm in diameter.
MD simulation results also indicate size dependent plasticity in SCS. For example, some computational studies suggest that free surfaces are the source of dislocation nucleation in SCS at the nano scale [55–57]. Interestingly, Kang and Cai [55] predicted that single crystal silicon can deform plastically at room temperature when sample diameter is less than 4nm. But the authors pointed out that the critical diameter in experiment can be much larger than their prediction due to the extremely high strain rate in MD simulation (about 13 orders of magnitude higher than in the experiment) as BDT in SCS is found to be sensitive to strain rate[52].

Although size and temperature dependent BDT in SCS has received increasing attention in the literature, BDT behavior in SCS is not fully understood. For example, Zhu et al [58] tested SCS nanowires by uniaxial tension where no ductility was observed even for nanowires 16 nm in diameter. Here the authors argued that large plastic deformation in Han et al work [53] may be due to strong electron beam irradiation during TEM observation. Also, unlike micropillar compression tests, bending tests of silicon beam with a thickness 255nm [18], and silicon nanowire with diameter between 200nm and 300nm [19] did not show any plastic deformation at room temperature. Note that the measured bending strengths are 17.53GPa (200nm in width) in [18] and 18.3GPa (120nm in diameter) in [19], respectively, while yield stress for SCS micropillars in [54] is about 2.5GPa~4GPa for 250nm in diameter at room temperature.

One of the main reasons for limited experimental data and controversies in the literature is due to the lack of a robust and rigorous in situ method for thermo-mechanical measurement. For unambiguous experimental investigation of size dependent BDT behavior in SCS, it is essential to control not only sample size, but also sample temperature as BDT is intrinsically sensitive to temperature. In this work we will introduce a novel thermo-mechanical in situ method with simultaneous control of two key parameters, sample size and temperature, (see Chapter 5) and using the method, we will resolve the controversy in the literature (see Chapter 6 and 7).
1.2 Organization of the Dissertation

In Chapter 2, we consider challenges in mechanical test with an emphasis on micro/nanomaterials. The significant portion of this chapter is taken from *In situ uniaxial mechanical testing of small scale materials - a review* by Kang et al. [59].

In Chapter 3, we theoretically consider a novel MEMS stage and sample design to overcome the challenges outlined in Chapter 2. The content of this chapter was published in Journal of Micro-Electro-Mechanical Systems which is entitled *A novel method for in situ uniaxial tests at micro/nano scale-Part I: Theory* [46]. Kang independently performed analytical study with the guidance from Prof. Saif.

In Chapter 4, we experimentally study the MEMS stage and the self-aligning mechanism built-on samples. The content of this chapter was published in Journal of Micro-Electro-Mechanical Systems which is entitled *A novel method for in situ uniaxial tests at micro/nano scale-Part II: Experiment* [47]. Kang carried out most of experiment and data analysis with initial collaboration with Dr. Han under the supervision of Prof. Saif.

In Chapter 5, we present a SiC based MEMS stage for in situ thermo-mechanical test. The content of this chapter is from *A SiC MEMS Apparatus for In Situ Uniaxial Testing of Micro/Nanomaterials at High Temperature* (IOP select paper) on Journal of Micromechanics and Microengineering [60] and *In Situ Thermo-Mechanical Testing for Micro/Nanomaterials* on MRS Communications [61]. Kang independently carried out this work under the supervision of Prof. Saif.

In Chapter 6, we experimentally show size dependent brittle-to-ductile transition (BDT) in single crystal silicon (SCS) using the SiC MEMS stage. We hypothesize this unusual behavior is due to surface dominant dislocation nucleation process with reduction in sample size. The content of this chapter is under review for journal publication. Kang led the research efforts with the guidance of Prof. Saif.

In Chapter 7, we study size dependent yield strength of SCS incorporated with stress
gradient and unique material characteristics of SCS. This work has been submitted for peer review process. Kang carried out modeling and experiment with Prof. Saif.

In Chapter 8, we summarize the major findings from this work and discuss future research topics.
Chapter 2

Challenges in *In Situ* Uniaxial Mechanical Tests

For uniaxial mechanical test in both tensile and compressive modes, the alignment between the sample axis and the loading direction is an important criteria to ensure uniform stress across the cross section of the sample. Misalignment introduces bending, and hence often unaccounted non-uniformity in stress on the sample. Sources of such misalignment include gripping error for tensile test and non-parallel compression plates for compressive tests. Macroscopic uniaxial test instruments have evolved by carefully considering the issue of misalignment and by introducing appropriate hinge mechanisms for self alignment [62]. This important issue has only received limited attention for uniaxial testing methods of micro/nano scale samples as we discussed in Chapter 1.

In this Chapter, we consider the alignment between the sample axis and loading direction, one of the key requirements for uniaxial tests to ensure uniform stress across the sample cross section with special emphasis on the micro/nano scale measurement. We will show that off-axis loading strongly influences the stress state of micro/nanoscale samples and can lead to large errors in data interpretation even when the deformation is within the elastic regime.

2.1 Source of Nonuniform Stress

Consider the representative loading scenarios (see Fig. 2.1) for uniaxial tests to explore possible sources of misalignment and their influence. Figure 2.1) shows (a) ideal uniaxial loading, (b) transverse misalignment, and (c) rotational misalignment.

In the following analysis, we assume that materials are linear elastic and strains are small
in order to highlight sources of misalignment errors and their influence up to maximum elastic
deformation of a sample. Furthermore, we assume sample deformation is small and the slope
of a deformed sample with respect to the undeformed shape is much smaller than unity. The
sample is held at the ends by the rigid frames of a loading stage. The frame on the right
moves horizontally to apply load on the sample while the left frame is fixed. The normal
stress at any point of specimen cross-section due to loading, $f$, is

$$\sigma_x = \sigma_I + \sigma_B \quad (2.1)$$

$$= \frac{f}{A} + \frac{My}{I} \quad (2.2)$$

where $\sigma_I, \sigma_B, A, M, y,$ and $I$ are uniaxial stress, bending stress, cross-sectional area, bending
moment, vertical coordinate of the point from its neutral axis, and moment of inertia. In
order to quantify non-uniform stress in the specimen, we define a non-uniform stress error
$e_m$ by

$$e_m = \frac{\sigma_B}{\sigma_I} \quad (2.3)$$

$$= \frac{\varepsilon_B}{\varepsilon_I} \quad (2.4)$$

where $\varepsilon_I$ and $\varepsilon_B$ are strains due to $\sigma_I$ and $\sigma_B$, respectively. Because strain measurement
is generally available at the surfaces of the specimen, we consider $\sigma_B$ and $\varepsilon_B$ at the top or
bottom of the sample or equivalently at $y = \pm h/2$ where $h$ is the height of the sample.

Let the apparent elastic modulus be $E_{app} = \sigma_I/\varepsilon_m$ where $\varepsilon_m = \varepsilon_I + \varepsilon_B$ is the strain
measured on the surface of the sample. From Eq. 2.3, $\varepsilon_m = \varepsilon_I + e_m \varepsilon_I$ and the error in elastic
modulus measurement is

\[
e_E = \frac{(E - E_{\text{app}})}{E} = 1 - \frac{\varepsilon_1}{\varepsilon_m} = \frac{e_m}{1 + e_m}
\]

where \( E \) is the elastic modulus of the specimen. Note that the error can be large when \( \varepsilon_m \to 0 \), i.e., when \( \varepsilon_1 \) and \( \varepsilon_B \) cancel each other. However, in order to take account of the non-uniform stress in the sample, \( E_{\text{mean}} = \sigma_{\text{I}}/\varepsilon_{\text{ave}} \) can be used for the accurate measurement of elastic modulus for linear elastic material response where \( \varepsilon_{\text{ave}} = (\varepsilon_{\text{top}} + \varepsilon_{\text{bottom}})/2 \). \( \varepsilon_{\text{top}} \) and \( \varepsilon_{\text{bottom}} \) are strains measured at the top and bottom of the sample.

### 2.1.1 Transverse misalignment

Here the sample is subjected to a load \( f \) with eccentricity \( c \). Such misalignment may be contributed by the asymmetric gripping of the sample as shown in Fig. 2.1(b). Both ends of the sample are clamped by grips which are connected to the loading frames by hinges. The width and axial length of the sample are designated by \( b \) and \( l \). To load the sample, the right loading frame moves away from the fixed frame and the load \( f \) is measured by a force sensor.

Consider the corresponding free body in Fig. 2.1(e). Let the curve DPG represent the shape of the neutral axis of the sample due to load \( f \). As a result of the eccentricity of loading, the sample is subjected to not only longitudinal loading but also bending. Let \( x \) be the distance along the undeformed sample and \( \chi(x) \) be the transverse deformation of the sample. We assume that the curvature of the deformed sample at any cross section \( P \) depends only on the magnitude of the bending moment. Then, the moment-curvature
relation becomes\cite{63}

\[ M(x) = \frac{EI}{\rho(x)} \tag{2.8} \]
\[ \approx EI \frac{d^2 \chi(x)}{dx^2} \tag{2.9} \]

where \( \rho(x) \) is radius of curvature due to bending. The curvature is approximated by \( d^2 \chi/dx^2 \) due to the assumption of the small slope of the deformed sample with respect to unity. The moment at \( x \) is given by

\[ M(x) = f(\chi(x) - c). \tag{2.10} \]

From Eq. 2.8 and 2.10, the normalized moment-curvature relation becomes

\[ \frac{d^2 \chi^*(x^*)}{dx^{*2}} = f^* \chi^*(x^*) - f^* c^* \tag{2.11} \]

where all lengths are normalized by the length, \( l \), of the sample (\( x^* = x/l, \chi^* = \chi/l, \) and \( c^* = c/l \)) and \( f^* = f l^2 / (EI) \) is the normalized load. Note that \( f^* = 12\varepsilon_1 / (h^*)^2 \) for a sample with rectangular cross section, \( A = bh \), where \( h^* = h/l \). From now on we only consider a sample with rectangular cross section. The solution for Eq. 2.11 with boundary conditions \( \chi^*(x^* = 0) = 0 \) and \( \chi^*(x^* = 1) = 0 \) is

\[ \chi^*(x^*) = c^* - \frac{c^* \cosh \left( \frac{1}{2} \sqrt{f^*} (2x^* - 1) \right)}{\cosh \left( \frac{1}{2} \sqrt{f^*} \right)} \tag{2.12} \]

where \( y^* = y/l \). From Eq. 2.3, 2.8, and 2.12, the non-uniform stress error \( e_m^{TM} \) at \( y = \pm h/2 \) is

\[ e_m^{TM} = \frac{\sigma_B}{\sigma_1} \]  
\[ = \pm \frac{6c^* \cosh \left( \frac{1}{2} \sqrt{f^*} (2x^* - 1) \right)}{h^* \cosh \left( \frac{1}{2} \sqrt{f^*} \right)} \]  

11
The non-uniform stress error at the top of the sample \((y = h/2)\) is normalized by \(\frac{h}{6c}\) and is shown in Fig. 2.2 as a function of \(x^*\) for \(0 \leq f^* \leq 10^4\). As \(f^*\) increases, the effect of non-uniform stress is reduced and is limited to only near the boundaries. For a free standing thin film sample with length \(\sim 100 \mu m\), thickness \(\sim 0.1 \mu m\), width \(\sim 1 \mu m\), and loaded to a strain of 0.1%, we have \(f^* \sim 10^4\) and the strain becomes uniform within 5% of the length of the sample from the edge. For a short sample with length \(\sim 10 \mu m\) and thickness \(\sim 1 \mu m\), we have \(f^* \sim 10^{-1}\) when loaded to 0.1% strain and the non-uniform stress is observed along entire gauge length. Even when the short sample is loaded to 1% strain, \(f^* \sim 1\), the cross section stress is highly non-uniform. For \(f^* \sim 10^2, \frac{h}{6c} e_{TM}^m \approx 0\) near \(x^* = 1/2\), but the magnitude of \(\frac{h}{6c} e_{TM}^m\) is 0.1357 at \(x^* = 0.2\) and 0.8 which indicates that the effect of non-uniform stress along gauge length must be carefully considered (see Fig. 2.2).

For macroscopic samples the magnitude of the non-uniform stress error, \(6c/h\), in Eq. 2.13 is likely \(\ll 1\) since the absolute misalignment \(c\) depends on the precision of the loading machine and the sample geometry. Hence, the error due to misalignment might be negligible. However, for micro- and nano-scale samples, it is difficult to achieve very small \(c\) with respect to \(h\). Therefore, misalignment error becomes important and a measure of misalignment (e.g. asymmetry in surface strains) becomes essential for the interpretation of load deformation data. For example, a seemingly small misalignment \(c = h/10\) with \(f^* \ll 1\) results in a nonuniform stress error \(e_m \approx 0.6\) and \(-150\%\) error \((E_{app} \approx 2.5E)\) in elastic modulus measurement from Eq. 2.5.

### 2.1.2 Rotational misalignment

Though mechanical testing of nanowires is not the main focus of the present work, here we consider the influence of the rotational misalignment in Fig. 2.1(c) for complete analysis on the misalignment scenarios at the micro/nano scale. Such misalignment may arise during placement of a sample, for example a nanowire, on the loading stage. The sample is glued to the stage (e.g., by metal deposition [32]). Even with careful handling, placement of the
nanowire by using a micromanipulator may result in small rotational misalignment, $\theta_0$, with respect to the loading axis as shown in Fig. 2.1(c). The sample is loaded by moving the right frame along the horizontal direction while the left frame is held fixed. The force, $f$, along the horizontal direction, i.e., horizontal component of the force on the sample end, is measured by a force sensor. Let us assume that there is no deformation of the glue or sliding between the sample and the glue during loading.

Now consider the free body of the sample in Fig. 2.1(f) where $x$ is the distance along the undeformed sample and $\chi(x)$ is the transverse deformation with the origin at K. The sample is rotated by $\theta_0$ with respect to the horizontal direction. The glued boundary at N restricts the sample end from rotation and constrains its motion along the horizontal direction. This fixed boundary condition results in a reaction force $R$ and moment $M_o$ at N.

Let the curve KPN in Fig. 2.1(f) represent the shape of the neutral axis of the sample during loading. The moment at any cross section of the sample is given by

$$M(x) = F_x(l - x) - F_x(\chi_l - \chi(x)) + M_o$$  \hspace{1cm} (2.15)

where $\chi_l$ is transverse deformation of the sample at $x = l$, $F_x$ and $F_\chi$ are the components of the force along $x$ and $\chi$ directions, respectively. Thus, $F_x = f \cos \theta_0 - R \sin \theta_0$ and $F_\chi = f \sin \theta_0 + R \cos \theta_0$. Let $u(x) = F_x x / (AE)$ be axial stretch of the sample at $x$.

From Eq. 2.8 and 2.15, the normalized moment-curvature relation is

$$\frac{d^2 \chi^*}{dx^*} (x^*) = F_x^* (\chi^* (1) - \chi^* (x^*)) - F_\chi^* (1 - x^*) + M_o^*$$  \hspace{1cm} (2.16)

where $F_x^* = F_x l^2 / (EI)$, $F_\chi^* = F_\chi l^2 / (EI)$, and $M_o^* = M_o l / (EI)$. The solution for Eq. 2.16 for the boundary conditions $d\chi^*/dx^* (x^* = 0) = 0$ and $d\chi^*/dx^* (x^* = 1) = 0$, and given $F_x$,
\[ F_x, \text{ and } M_o \text{ is} \]

\[ \chi^* (x^*) = \chi^*_l - \frac{F^*_x (1 - x^*) + M^*_o}{F^*_x} + \frac{F^*_x \sinh \left( \frac{1}{2} \sqrt{F^*_x} (1 - 2x^*) \right)}{\left( F^*_x \right)^{3/2} \cosh \left( \frac{1}{2} \sqrt{F^*_x} \right)} \]

(2.17)

where \( \chi^*_l \) is transverse deformation due to \( f \) as shown in Fig. 2.1(f). There are four unknowns, \( \chi^*_l, F^*_x, F^*_\chi, \text{ and } M^*_o \), which need to be solved from additional boundary conditions. The conditions \( \chi^* (0) = 0 \) and \( \chi^* (1) = \chi^*_l \) give \( M^*_o = -\frac{F^*_x \tanh (\sqrt{F^*_x} / 2)}{\sqrt{F^*_x}} \) and \( \chi^*_l = \frac{(\sqrt{F^*_x F^*_\chi} - 2F^*_\chi \tanh (\sqrt{F^*_x} / 2)) / (F^*_x)^{3/2}}{F^*_x h^*} \). At \( x^* = 1 \), we have a geometrical constraint that the stage moves only along the horizontal direction which gives the relation between longitudinal and transverse deformation of the sample at N. If the stretch, \( s = \int_0^1 (\sqrt{1 + d^2 \chi^* / dx^2} - 1) dx \), of the sample due to bending alone is small such that \( s / u \ll 1 \), then \( u^* (1) / \chi^* (1) \approx \tan \theta_o \). This gives the relation between \( F^*_x = f^* \cos \theta_o - R^* \sin \theta_o \) and \( F^*_\chi = f^* \sin \theta_o + R^* \cos \theta_o \) as follows

\[ \frac{12 F^*_x (\sqrt{F^*_x} - 2 \tanh (\sqrt{F^*_x}))}{(F^*_x)^{5/2} (h^*)^2} - \tan (\theta_o) = 0. \]

(2.18)

From Eq. 2.3 and 2.17, the nonuniform stress \( \varepsilon_m \) for rotational misalignment with \( \sigma_1 = F_x / (bh) \) is

\[ \varepsilon_m^{\text{RM}} = \pm \frac{6 (d^2 \chi^* / dx^2)}{F^*_x h^*} = \pm \frac{n}{m} \sqrt{\frac{3}{m \varepsilon_1}} \sinh \left( \frac{1 - 2x^*}{h^*} \right) \sqrt{3m \varepsilon_1} \]

(2.19)

where \( m = \cos \theta_o - \lambda \sin \theta_o, n = \sin \theta_o + \lambda \cos \theta_o, \) and \( \lambda = R^* / f^* \) (\( \lambda \) can be obtained by using Eq. 2.18). We validated the assumptions on \( d\chi / dx \) and \( s / u \) in the analysis. The maximum slope of the deformed sample occurred at \( f^* = 10.7895 \) where \( d\chi / dx = 0.01778 \) and \( s / u \sim 10^{-3} \) for \( \theta_o = 15^\circ \) and \( \varepsilon_1 = 5\% \). Equation 2.18 and 2.19 are used to evaluate \( \varepsilon_m^{\text{RM}} \) at the bottom of the sample as a function of \( x^* \) (Fig. 2.3(a)-(c)) for parameters \( \theta_o, h^*, \) and \( \varepsilon_1 \). Figure 2.3(a) shows that stress non-uniformity is minimum at the mid length of the sample and is maximum at the boundaries. \( \varepsilon_m \) increases with \( \theta_o \) as expected. Figure 2.3(b) indicates the importance of \( h^* \) for uniaxial tension test. For example the effect of nonuniform stress is limited to within 5\% of the total gauge length near the boundaries for
$h^* \sim 1/100$. Finally, Fig. 2.3(c) shows that for given $h^* = 1/10$, the overall uniformity of the stress along the gauge length improves as $\varepsilon_1$ increases.

For nanowire samples, the radius to length ratio is much less than 1/10 and hence uniform stress along gauge length can be achieved. However, increase of $\varepsilon_1$ induces strong edge effect such that the maximum magnitude of $e_m$ is substantially increased near $x^* = 0$ and $= 1$ (Fig. 2.3(c)). Hence, for fracture strength measurement, the edge effect should be properly considered. Note that $h^*$ is of order $10^{-3}$ (or $f^* = 12 \times 10^6\varepsilon_1$) for the thin films used in Ref. [31] and, hence, uniaxial loading is guaranteed even with transverse and rotational misalignment.

In the present chapter, we showed that for a given misalignment error (due to gripping or otherwise), the bending stress on the sample or the deviation from the uniform stress (= load/area) scales inversely with the sample size. Thus, at small scale, the error can be large and may result in large local stress and strain gradients, premature apparent yielding, and non-uniform strain hardening which complicates the interpretation of the results. In order to overcome this intrinsic challenge in mechanical measurement of the small samples, we will introduce a MEMS based testing stage and sample design with built-in self-aligning mechanisms that ensure uniaxial tests on small samples in the following chapters. In Chapter 3, we will theoretically explore the question of stress uniformity in the context of the new stage and sample. Then in Chapter 4 we will address the same question experimentally by carrying out in situ studies.
2.2 Figures

Figure 2.1: The scenarios of misalignment between loading, $f$, and a specimen: (a) ideal loading, (b) transverse misalignment, and (c) rotational misalignment. The degree of misalignment for transverse and rotational misalignments are quantified by $c$ and $\theta_o$, respectively. The free body diagrams of the representative cases are shown in (d), (e), and (f) for ideal loading, transverse misalignment, and rotational misalignment, respectively.
Figure 2.2: Normalized non-uniform stress error \( \frac{h}{6c} \epsilon_{m}^{TM} \) as a function of \( x^* \) at the top of the sample with variation of \( f^* = f l^2 / (EI) \) from 0 to \( 10^4 \).
Figure 2.3: Non-uniform stress error $\varepsilon_{\text{RM}} = \sigma_R/\sigma_1$ at the bottom of the sample as a function of $x^*$ for rotational misalignment $\theta_o$ with variation of (a) $0 \leq \theta_o \leq \pi/12$, (b) $1/100 \leq h^* \leq 1$, and (c) $10^{-4}\% \leq \varepsilon_1 \leq 5\%$. 
Chapter 3

In Situ Mechanical Testing at Micro/Nanoscale: Theory

As discussed in Chapter 2, the alignment between the sample axis and loading direction is one of the key requirements for uniaxial tests to ensure uniform stress across the sample cross section. At micro/nano scale, the effect of nonuniform stress must be carefully considered since the error due to the misalignment is inversely proportional to sample size. Although sources of misalignment and their influence on mechanical testing of micro/nano scale samples have been considered for micropillar compression tests [64], such issues in tensile testing have received little attention in the literature. Also, as outlined in Chapter 1, Haque et al. [31] minimized the misalignment errors by cofabricating the stage and the sample and by introducing a self alignment mechanism built-into the stage. However, the method is restricted to thin film materials that can be cofabricated with the loading stage. Their test sample cannot be prepared separately from the stage. Hence in this chapter, we present a novel stage with gripping mechanism so that an independently fabricated sample can be tested without limitation on sample material and dimension. In addition, we propose a sample design with built-in self-aligning mechanism for uniaxiality of loading during mechanical test at micro/nano scale.

3.1 Microscale tensile/compression stage

The MEMS stage allows testing of independently fabricated samples with an in-built gripping mechanism. This is a significant improvement over the stage presented in [23] where the sample needed to be cofabricated with the stage. The overall dimensions of the novel MEMS
based uniaxial testing device are small enough to perform *in situ* uniaxial tests in SEM and TEM. The 3-dimensional solid model of the tensile stage is shown in Fig. 3.1(a). It has two grooves which serve as grips for a dog bone shape sample as shown in Fig. 3.1(b)-(c). Beams at A-A in Fig. 3.1(a) serve as a force sensor and at B-B as a support for the sample. These beams are compliant in the in-plane transverse direction, but stiff in other directions due to high depth to width ratio (150/10 ∼ 150/60). A dog-bone specimen fits into the grips (see Fig. 3.1(c)) and then is loaded by deforming the stage using a piezoelectric actuator with two pillars that go through the holes of the stage.

As in [23], upon loading, the support beams transfer the deformation to the specimen. The U-beams suppress any misalignment between the direction of loading at pillars and at the grips by 6 orders of magnitude as discussed in Ref [23] (18° loading alignment error is reduced to 1.33×10^{-5} degrees misalignment at the grips). The specimen and the force sensing beams are in series. Therefore, the load in the specimen is obtained from the deformation of the beams (A-A) with respect to the force sensor gauge. The stiffness of the beams is calibrated by a weighting scale. Note that force resolution of the force sensor can be achieved by decreasing the width of the force sensing beams. Stretch in the sample is obtained from image analysis. After the calibration of the force sensor, the uniaxial testing stage and the specimen can be assembled by a micromanipulator. SEM or TEM images can be taken to measure strain and stress of the specimen simultaneously. Though the stage is capable of both tension and compression tests, the present chapter focuses on the former.

### 3.1.1 Force sensor

Here, we consider the effect of geometric nonlinearity of the force sensing beams due to their transverse deformation δ along the loading direction (Fig. 3.1). In our previous work [23], displacement of the force sensing beam was small up to fracture of the thin film samples due to the small cross-section so that force-displacement relation can be captured by a linear relation. However, for three dimensional microsamples the deformation of the force sensing
beams becomes large and, therefore, the beams not only bend, but also stretch during deformation. Hence, force($f$)-displacement($\delta$) relation becomes

$$f = \bar{k}\delta + \bar{k}_3\delta^3$$ (3.1)

where $\bar{k}$ and $\bar{k}_3$ are linear and nonlinear spring constants. The linear spring constant of $N$ pairs of force sensing beams at A-A in Fig. 3.1(a) is $\bar{k} = 16NE_{SB}b_{SB}h_{SB}/(l_{SB})^3$ where $E_{SB}$, $b_{SB}$, $h_{SB}$, and $l_{SB}$ are Young’s modulus, width, depth, length of the force sensing beams, respectively [65].

The strain of the beams associated with the stretching is approximated by

$$\varepsilon_{SB} = \frac{\Delta l_{SB}}{l_{SB}}$$ (3.2)

$$\approx \sec \phi - 1$$ (3.3)

$$\approx \frac{\phi^2}{2}$$ (3.4)

where $\phi$ is the small angle of the sensor beam with respect to the rest position. Hence, the force-strain relation of the beam stretching is [66]

$$F_S = 2\varepsilon_{SB}E_{SB}b_{SB}h_{SB}\phi.$$ (3.5)

From Eq. 3.2 and 3.5

$$F_S = E_{SB}b_{SB}h_{SB}\phi^3$$ (3.6)

$$\approx 8E_{SB}b_{SB}h_{SB}(\delta/l_{SB})^3$$ (3.7)

since $\phi l_{SB}/2 \approx \delta$ for small $\phi$ and hence $\bar{k}_3 = 8NE_{SB}b_{SB}h_{SB}/l_{SB}^3$. This nonlinear term in the force-displacement relation in Eq. 3.1 must be properly considered for large deformation of the beams. In Chapter 4, we will discuss experimental procedure for force sensor calibration.
3.1.2 Finite Element Analysis

Here we consider the sources of misalignment due to the interaction between the sample and the stage as an assembled system and their influence on the stress state in sample cross section. Though we previously considered the representative misalignment errors, we assumed the loading frame was rigid and hence these results fail to predict the sources of misalignment which may be induced due to the compliance of the stage.

In order to allow the deformation of both the sample and the stage, we employ a finite element analysis. The one-to-one scale solid models for the stage and the microtensile stage are separately created and assembled using Pro/ENGINEER. The commercial package ANSYS 9.0 is used to simulate the system as shown in Fig. 3.2(a). Half the system is analyzed due to symmetry (asymmetry of the system about the half plane is not considered). Three pairs of contact surfaces are utilized to mimic actual surface contacts: one pair between the T-shaped beam and the frame and two pairs between the specimen and the grips. For FEA, Young's modulus (169 GPa, $<110>$ direction), the Poisson's ratio (0.25), and friction coefficient (0.2) [67] for single crystal silicon are used. We found FEA results are insensitive to variation of friction coefficient between 0.1 and 0.5 since no slip was observed between the stage and sample due to the small taper angle (3 degree) on the sample. Linear isotropic material behavior is assumed for the analysis. The left pinhole in Fig. 3.1(a) was constrained by fixed boundary condition while the right one was longitudinally pulled by prescribed displacement to load the specimen. Since our focus is on the sources of misalignment errors due to the compliance of the stage and their effect on the non-uniformity of stress in the sample, we use a sample with large bending stiffness, i.e., $h/l \sim 1$.

We first consider shallow grips where the depth of grips is $1/5$ of stage depth. The grip contacts the entire height of the sample with a friction coefficient of 0.2 so that loading is perfectly aligned with the sample initially. Upon loading, a moment on the stage, $M_{st}$, is induced due to the asymmetric location of the sample, near the top of the stage. Hence, even
though the sample is initially aligned with the loading axis, the bending of the stage results in shifting of the load from the center of the sample toward the bottom. For example, when $\varepsilon_{\text{top}} = 0.2$, we find the non-uniform stress error $e_m = 52\%$ at the mid-length of the sample. From Eq. 2.5, the corresponding error in elastic modulus measurement is about 33.3% when strain at the bottom of the sample is considered while $\varepsilon_m = 100\%$ when $\varepsilon_{\text{top}}$ is used. With larger strain $\varepsilon_{\text{top}} = 0.6$, the non-uniform stress error is still about 50%.

Next, we consider the effect of gripping. Misalignment errors due to the gripping are mimicked by the tapered surface on the sample as shown in Fig. 3.2(c). This tapered surface represents any inclined sidewall profile of either the sample or the grips and any asperity contact between the sample and the grips as illustrated in Fig. 3.3. Figure 3.4(a) shows the strain evolution of the specimen with incremental increase of the stretch in the stage at the pinholes. The corresponding average stress, $\sigma_{\text{ave}} = (\sigma_{\text{top}} + \sigma_{\text{bottom}})/2$, and strain at the top and bottom are shown in Fig. 3.4(b). Initially the bottom is in compression while the top is in tension. With further increase of the load, the slope, $d\sigma_{\text{ave}}/d\varepsilon_{\text{bottom}}$, changes its sign, from negative to positive, whereas $d\sigma_{\text{ave}}/d\varepsilon_{\text{top}}$ increases rapidly. During the early phase of loading ($d\sigma_{\text{ave}}/d\varepsilon_{\text{bottom}} < 0$), the load is aligned with the upper edge of the sample as schematically shown in Fig. 3.5(a). The transitions in $d\sigma_{\text{ave}}/d\varepsilon_{\text{bottom}}$ and $d\sigma_{\text{ave}}/d\varepsilon_{\text{top}}$ are explained by a shift in the loading axis due to the bending in the sample ($M_{\text{sp}}$) and in the stage ($M_{\text{st}}$). As shown in Fig. 3.5(a)-(b), $M_{\text{sp}}$ and $M_{\text{st}}$ reduce the surface contact angle, $\theta_g$, between the specimen and the grip continuously. When the loading exceeds some critical value ($f_{\text{cr}}$), such that the angle becomes zero, a shift in the loading axis occurs which leads to the simultaneous transitions in $d\sigma_{\text{ave}}/d\varepsilon_{\text{bottom}}$ and $d\sigma_{\text{ave}}/d\varepsilon_{\text{top}}$. Thus the elastic modulus measured from either the top or the bottom strain will be erroneous. In order to take account of the non-uniform stress in the sample, $\sigma_{\text{ave}}/\varepsilon_{\text{ave}}$ can be used for the accurate measurement of elastic modulus for linear elastic material response where $\varepsilon_{\text{ave}} = (\varepsilon_{\text{top}} + \varepsilon_{\text{bottom}})/2$. 
3.2 Specimen with a Self-Aligning mechanism

Here we propose a novel design for a specimen with a built-in hinge-like mechanism. Though the MEMS stage has U-beams which suppress any misalignment on the stage as discussed in Ref [23], simulation results in Fig. 3.4 showed that grips may induce rather large misalignment errors. In experimental uniaxial tests, the grips may introduce in-plane and out-of-plane misalignment with respect to the stage and it is difficult to quantify or control these errors due to the small scale. Hence, the hinge-like self-aligning sample is essential to suppress both in-plane and out-of-plane misalignment during uniaxial testing.

3.2.1 Hinge-like mechanism

Fig. 3.6(a)-(c) which consists of gripped parts, a specimen, and self-aligning beams. The dimensions of different parts are defined by $W_i, H_i$ and $L_i$ where $i = 1, 2, \text{and } 3$ correspond to the specimen, self-aligning beam 1, and self-aligning beam 2, respectively. The first design criterion is $W_1 H_1 < W_2 H_2$ and $W_1 H_1 < W_3 H_3$ which guarantee that the mean stress in the self-aligning beams is always smaller than that in the specimen. The other criterion is that the bending stiffness of the self-aligning beams is much smaller than that of the specimen about desired direction. For instance, beam 1 and beam 2 are designed to be compliant in the out-of-plane and in-plane bending directions, respectively. Hence, the ratios of bending stiffness between the specimen and the self-aligning beam 1 are

$$\frac{k_1}{k_2} = \frac{W_1 H_1^2 L_2^3}{W_2 H_2^2 L_1^3} \gg 1 \quad \text{and} \quad \frac{\hat{k}_1}{\hat{k}_2} = \frac{W_1^3 H_1 L_2^3}{W_2^3 H_2 L_1^3} \ll 1$$

for out-of-plane and in-plane bending stiffness, respectively. Likewise, the ratios of bending stiffness of the specimen to the self-aligning beam 2 are $k_1/k_3 \ll 1$ and $\hat{k}_1/\hat{k}_3 \gg 1$. Hence, the self-aligning beams behave as hinges. Further detail on the design criteria for the self-aligning beams is discussed in Chapter 4.
3.2.2 Analytical model

Bending stresses in the sample with and without the self-aligning mechanism are compared analytically. Only one hinge is considered in the sample for simplicity. They are referred to as a hinged sample and a plain sample, respectively. The samples are subjected load $f$ with the eccentricity $c$ as shown in Fig. 3.7(a). The moment of inertia and length of the hinge are designated by $I_1$ and $l_1$ (Fig. 3.7) where the total length of the sample is $l$. In the following analysis, we assume that the sample is linear elastic with small deformation. With the normalized coordinates ($x^* = x/l$ and $\chi^* = \chi/l$), the normalized geometry of the sample ($h^* = h/l, l_1^* = l_1/l$, and $l_2^* = l_2/l$), and the normalized parameters ($f^* = f/l^2/(EI), c^* = c/l$, and $I^* = I/I_1$), the normalized moment-curvature relations for a hinged sample with transverse misalignment, $c$, are

$$
\begin{cases}
-\frac{d^2\chi_1^*(x^*)}{dx^2} = f^*I^*(c^* - \chi_1^*(x^*)), 0 < x^* < l_1^* \\
-\frac{d^2\chi_2^*(x^*)}{dx^2} = f^*(c^* - \chi_2^*(x^*)), l_1^* < x^* < 1/2
\end{cases}
$$

(3.8)

with boundary conditions $\chi_1^*(x^* = 0) = 0$, $\chi_1^*(x^* = l_1^*) = \chi_2^*(x^* = l_1^*)$, $\frac{d\chi_1^*}{dx^*}(x^* = l_1^*)$, and $\frac{d\chi_2^*}{dx^*}(x^* = l_1^* + l_2^*) = 0$. $\chi_1$ and $\chi_2$ for a hinged sample from Eq. 3.8 are

$$
\begin{cases}
\chi_1^*(x^*) = \frac{c^*e^{-\sqrt{T^*}x^*}c_3(a_1b_1 + a_2b_2)}{a_1c_1 + a_2c_2} \\
\chi_2^*(x^*) = \frac{c^*e^{-\sqrt{T^*}x^*}(a_2b_4 + a_1b_3 - 2\sqrt{T^*}c_4)}{a_1c_1 + a_2c_2}
\end{cases}
$$

(3.9)

where $a_1 = 1 + \sqrt{T^*}$, $a_2 = 1 + \sqrt{T^*}$, $b_1 = e^{\sqrt{T^*}(1+2\sqrt{T^*}l_1^*)} - e^{\sqrt{T^*}(2l_1^*+\sqrt{T^*}x^*)}$, $b_2 = e^{2a_1\sqrt{T^*}l_1^* - e\sqrt{T^*(1+\sqrt{T^*}x^*)}}$, $b_3 = e^{\sqrt{T^*}(2l_1^*+x^*)} + e^{\sqrt{T^*(1+2\sqrt{T^*}l_1^*+x^*)}}$, $b_4 = e^{\sqrt{T^*(1+x^*)} + e\sqrt{T^*(2a_1l_1^*+x^*)}$, $c_3 = e\sqrt{T^*(1+x^*)} - 1$, and $c_4 = e\sqrt{T^*(1+l_1^*+\sqrt{T^*}l_1^*)} + e\sqrt{T^*(l_1^*+\sqrt{T^*}l_1^*+2x^*)}$. From Eq. 2.12 and 3.9 the ratio, $\eta$, of the curvatures between the hinged and plain samples, $(\frac{d^2\chi_1^*}{dx^2})/(\frac{d^2\chi_2^*}{dx^2})$, at $x^* = 1/2$ is

$$
\eta = \frac{4e^{\frac{3}{2}\sqrt{T^*(1+2a_2l_1^*)}}\sqrt{T^*}\cosh(\frac{1}{2}\sqrt{T^*})}{a_1a_3 + a_2a_4}
$$

25
where $a_3 = e\sqrt{r} + e^{2a_2l^*_1}\sqrt{r}$ and $a_4 = e^{2\sqrt{r}l^*_1} + e^{\sqrt{r}(1+2\sqrt{r}l^*_1)}$.

From Eq. 3.9, the shape of the sample due to $f^* = fl^2/(EI) = 10$ is shown in Fig. 3.8(a) for $l^*_1 = l_1/l = 1/3$ and $c^* = c/l = 0.1$ with incremental increase of $I^* = I/I_1$. The reduction in curvature or equivalently in non-uniform stress along the gauge length ($l^*_2$) is clearly observed for example between $I^* = 1$ and $I^* = 9$.

In order to systematically evaluate the reduction in non-uniform stress, we directly compare curvature of the hinged and plain samples, given by $\eta = (d^2\chi^*_2/dx^*_2)/(d^2\chi^*/dx^*_2)$ at $x^* = 1/2$, where both the samples have the same cross-section along the gauge length and are subjected to the same transverse misalignment error as shown in Fig. 3.7(a)-(b). $\chi^*$ and $\chi^*_2$ are given in Eq. 2.12 and 3.9, respectively. $\eta$ is shown in Fig. 3.8(b) for $f^* = 10$ and $c^* = 1/10$. Stress non-uniformity approaches zero exponentially as $I^*$ increases and the convergence rate is highly dependent on normalized length of the self-aligning beams. For example, using dimensions, $l_1 = 40 \mu m$, $l_2 = 10 \mu m$, $I = 10 \mu m^4$, $I_1 = 2 \mu m^4$ and $\sigma_1 = 350$ MPa, bending ratio $\eta$ is about 20% (or 80% reduction in bending) at the mid length of the sample.

### 3.2.3 Numerical model

We finally study the self-aligning mechanism shown in Fig. 3.7 numerically by using FEA. The conditions and procedure discussed in Section 3.1.2 are used here with the exception that the depth of grips is up to mid-height of the stage which minimizes $M_{st}$. The geometry of a sample is $l^*_1 = 1/3$ and $I^* = 9$ with tapered sidewall ($\theta_g = 0.037$ rad). Figure 3.9 shows $\varepsilon_{top}$ versus $\varepsilon_{bottom}$ at the mid-length of the hinged and plain samples. The two curves start with negative slope, i.e., $d\varepsilon_{bottom}/d\varepsilon_{top} < 0$ (for the hinged sample see Fig. 3.9(b)) and they approach 1 after the transition from $d\varepsilon_{bottom}/d\varepsilon_{top} < 0$ to $> 0$. However, quantitatively the onset of transition is clearly distinctive as they occur within $\varepsilon_{top} \approx 0.1\%$ for the hinged sample and $\varepsilon_{top} \approx 1.5\%$ for the plain sample. With the minimized moment on the stage ($M_{st} \approx 0$), the grips predominantly move along the longitudinal direction. Hence, maximum
bending deformation of the sample is geometrically restricted by $\theta_g$ as schematically shown in Fig. 3.9.

In order to compare the numerical results with analytical model, the sample with $l_1^* = 1/3$ and $I^* = 9$ is used again. In the FEA, the loading axis initially occurs at the top of the sample due to the taper at the gripped face and hence the transverse misalignment $c^* = h^*/2$ is used for the analysis. The strains at the top and the bottom of the sample can be obtained from $\varepsilon_{top,bottom} = \sigma_1/E \pm (h/2)(d^2\chi(x)/dx^2)$ and Eq. 3.9. Figure 3.10 shows $\varepsilon_{top}$ versus $\varepsilon_{bottom}$ of the sample with incremental increase of load $f$. We find similar qualitative behaviors as was obtained in FEA analysis. However, the onset of transition occurs near $\varepsilon_{top} \approx 1.5\%$ for the hinged sample and $\varepsilon_{top} \approx 5\%$ for the plain sample while they were observed near 0.1\% and 1.5\%, respectively, from FEA results in Fig. 3.9. This discrepancy is due to the geometrical constraint $\theta_g$ in FEA model where the sample bending is limited by the full engagement of the gripped faces. This results in a shift of the load towards the center of the sample which reduces misalignment. In the theoretical analysis, the misalignment is kept fixed.

The analytical and FEA results in Fig. 3.9 and 3.10 indicate the $\varepsilon_{bottom}$-$\varepsilon_{top}$ curve is highly sensitive to the boundary conditions at the grips which are difficult to control and measure in the micro/nano scale tests. However, the self-aligning sample significantly reduces the influence of misalignment in the $\varepsilon_{bottom}$-$\varepsilon_{top}$ curve, i.e., $\varepsilon_{bottom}/\varepsilon_{top} \approx 1$ under both the boundary conditions analyzed above which allows accurate measurement of elastic material properties of small scale samples even without considering the influence of the grips on the non-uniform stress of the sample.

In order to compare the reduction in non-uniform stress for the hinged and plain samples with $l_1^* = 1/3$ and $I^* = 9$, the non-uniform stress error $e_m = \sigma_B/\sigma_1$ at the top of the sample is shown in Fig. 3.11 as a function of $f^* = f l^2/(EI)$. For the hinged sample, $e_m$ approaches zero much faster than for the plain sample. The significant reduction in the stress non-uniformity along the gauge length is also observed for the hinged sample as $e_{m,hinge}$ at $x^* = 1/2$ and $= l_1^*$ are almost overlapping each other. For the plain sample, on the other
Table 3.1: Comparison between the *hinged* and *plain samples* at $f^* = f l^2 / (EI) = 20$.

<table>
<thead>
<tr>
<th></th>
<th>$e_m (%)$</th>
<th>$e_E$ (%) at top</th>
<th>$e_E$ (%) at bottom</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Hinge</strong></td>
<td>5.7</td>
<td>5.4</td>
<td>6.0</td>
</tr>
<tr>
<td><strong>Plain</strong></td>
<td>81</td>
<td>45.0</td>
<td>449.5</td>
</tr>
</tbody>
</table>

At $x^* = 1/2$

<table>
<thead>
<tr>
<th></th>
<th>$e_m (%)$</th>
<th>$e_E$ (%) at top</th>
<th>$e_E$ (%) at bottom</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Hinge</strong></td>
<td>4.4</td>
<td>4.2</td>
<td>4.6</td>
</tr>
<tr>
<td><strong>Plain</strong></td>
<td>63.4</td>
<td>38.8</td>
<td>173.2</td>
</tr>
</tbody>
</table>

hands, substantial difference is consistently observed between $e_m^{\text{plain}}$ at $x^* = 1/2$ and $l_1^*$.

For example, consider a sample with $l = 180 \, \mu m$, $h = 5 \, \mu m$, $b = 5 \, \mu m$, $I^* = 9$, and $l_1 = 60 \, \mu m$. Table 3.1 summarizes the non-uniform stress error $e_m$ and the corresponding error $e_E$ in elastic modulus measurement at the top and bottom of the sample at $f^* = 20$ (or equivalently $\varepsilon_1 = 0.13\%$). For the *hinged sample*, the maximum $e_E$ is 6.0% and variation of the error along the gauge length and due to choice of the surface for strain measurement, either the top or bottom, is significantly smaller compared to the *plain sample*. Also, in order to achieve $e_m = 0.044$ at $x^* = 1/2$ with the *plain sample*, much larger normalized load $f^* = 96.78$ (or equivalently $\varepsilon_1 = 0.62\%$) is required and still relatively large stress non-uniformity along the gauge length is observed with $e_m = 0.117$ at $x^* = l_1^*$ (or $e_E = 13.3\%$ at the bottom or 10.5% at the top). Note that further improvement in uniformity of loading can be achieved with increase of $I^*$ and $l_1^*$ in the *hinged sample* as shown in Fig. 3.8.

We have extensively explored the parameter space for design of a self-aligning specimen and shown significant reduction in non-uniform stress not only across cross-section of a sample but also along gauge length due to the novel specimen. Hence, by using the sample with two self-aligning hinges in Fig. 3.6, the influence of unaccounted misalignment errors is eliminated or minimized even when the grips simultaneously induce in- and out-of-plane bending.
3.3 Conclusion

In uniaxial tension and compression tests, the axis of loading is often misaligned with the sample axis. Misalignment error may originate from multiple sources including asymmetric gripping. Misalignment error results in non-uniform stress on the cross section of the sample as well as along its gauge length. More importantly, the degree of misalignment may change in a given experiment with increasing load due to the shift of the location of the load at the grip. The shift is due to the compliance of the sample and/or the loading stage. This results in a nonlinear relation between the measured applied load and strain at a point on the surface of the sample, even though the strains are small and the material is linear elastic.

In order to carry out uniaxial test on small scale samples, we propose a novel MEMS stage with in-built grips. The stage allows in situ tension and compression tests of micro/nano scale dog-bone shape samples. Numerical simulation of a tensile test using the stage shows that the compliance of the stage may lead to 100% error in elastic modulus measurement. When we additionally introduce the irregular topography at the sample grip, the stage and sample compliance results in varying point of contact between the grip and stage along the height of the sample with increasing load. This inherent nonlinearity gives rise to the variation of $d\sigma/d\varepsilon$ with increasing load in linear elastic sample which challenges the elastic modulus measurement. In order to overcome the misalignment error, we propose a novel design for the specimen with self-aligning hinges. We conduct detailed analytical and numerical simulation of tensile test on the hinged sample using the MEMS stage. The analysis shows that the hinges suppress misalignment errors significantly within the entire gauge length of the sample. For example, the error in elastic modulus measurement is reduced to 4.2∼6.0% while a similar sample without the hinge gives 38.8∼450.0% error. We show that the error can be further suppressed by increasing compliance of the hinges which ensure pure uniaxiality of loading for the micro/nano scale uniaxial tests. In the following chapter, we will study the effect of misalignment experimentally in microscale samples. We
will then demonstrate the applicability of the new stage and the sample by measuring elastic modulus of single crystal silicon samples.
3.4 Figures

Figure 3.1: The schematic of the uniaxial testing stage: (a) 3-dimensional solid model and (b)-(c) grips on the stage and a sample before and after assembly.
Figure 3.2: Solid models to study influence of stage compliance and gripping between the sample and the stage on misalignment errors: (a) overall view of one-to-one scale solid modeling of the tensile stage for FEA, (b) zoom-in view of the sample upon loading, and (c) the schematic of the initial gripping condition. Dotted line in (c) shows the tapered profile of the specimen.
Figure 3.3: The schematic of gripping errors: (a) inclined sidewall profile, (b) full-engagement due to critical force $f_{cr}$, (c) asperity contact, and (d) full-engagement. The magnitude of gripping errors is defined by $\theta_g$. 

\[ \text{Asperity} \]
\[ \text{Grip} \]
\[ \text{Specimen} \]
\[ \text{Undercut Taper} \]
\[ \theta \]
Figure 3.4: The sequences of the FEA results for a tapered specimen: (a) the strain evolution with incremental increase of the pin-hole distance (a1)-(a5) and (b) the corresponding stress-strain response at the mid-length of the sample. $\sigma_{ave}$ is the average stress of the top and bottom of the sample.
Figure 3.5: The schematic of the loading mechanism from side view: (a) initial loading and (b) reverse of bending in the sample with increase of loading. The asymmetric location of the sample, near the top of the stage, and the tapered surface on the sample induce $M_{st}$ and $M_{sp}$, respectively. $M_{sp}$ and $M_{st}$ reduce the surface contact angle, $\theta_g$, between the specimen and the grip continuously and result in a shift of contact toward the bottom.
Figure 3.6: The schematic of the self-aligning specimen: (a) overall 3-dimensional view, (b) top view, and (c) side view. GP indicates parts that are engaged by grips.
Figure 3.7: The schematic of samples: (a) a *hinged sample* and (b) a sample without any hinge. The load is applied on both the samples with misalignment $c$. 
Figure 3.8: Analytical results of the \textit{hinged} and \textit{plain samples} subjected to the transverse misalignment. In (a), deformed shape of the \textit{hinged} and \textit{plain samples} is shown for $f^* = f l^2 / (E I) = 10$, $c^* = 0.1$, and $l_1^* = 1/3$ with variation of $I^* = I/I_1$. The red line represents deformed shape of a \textit{plain sample} from Eq. 2.12. In (b), $\eta = (d^2 x^2 / dx^2) / (d^2 x^* / dx^2)$ is shown as a function of $I^*$ for $0 \leq l_1^* = l_1/l \leq 0.4$ at $x^* = 1/2$ upon loading with $f^* = 10$ and $c^* = 1/10$. $\eta$ directly compares bending moments in the \textit{hinged} and \textit{plain samples}. 

\[ I = \frac{I}{I_1} = 1 \]

\[ l_1^* = \frac{l_1}{l} = \frac{1}{3} \]
Figure 3.9: FEA results for the hinged sample and the plain sample with \( l_1^* = l_1/l = 1/3 \), \( \bar{I}^* = I/I_1 = 9 \), and \( \theta_g = 0.037 \) rad. In (a), the transition from \( \alpha < 0 \) to \( \alpha > 0 \) is observed near \( \varepsilon_1 \approx 1.5\% \) for the plain sample where \( \alpha = d\varepsilon_{\text{bottom}}/d\varepsilon_{\text{top}} \). Figure (b) shows the zoomed in view of (a). The transition for the hinged sample occurs between \( \varepsilon_1 \approx 0.1\% \).
Figure 3.10: Analytical results for the hinged sample and the plain sample subjected to the eccentricity $c = h/2$ with $l_1^* = l_1/l = 1/3$ and $I^* = I/I_1 = 9$ (refer Fig. 3.7). In (a), the transition from $d\varepsilon_{\text{bottom}}/d\varepsilon_{\text{top}} < 0$ to $> 0$ is observed near $\varepsilon_1 \approx 5\%$ for the plain sample. Figure (b) shows the enlargement of (a). The transition for the hinged sample occurs about $\varepsilon_1 \approx 1.5\%$. 
Figure 3.11: $e_m = \sigma_B / \sigma_1$ at the top of the samples for the hinged and plain samples at $x^* = l_1^*$ and $= 1/2$ as a function of $f^*$ when $c = h/2$. 
Chapter 4

In Situ Mechanical Testing at Micro/Nanoscale: Experiment

In Chapter 3, we explored the effect of misalignment on uniaxial tests theoretically. We then proposed a novel design of a micro stage and a sample with self aligning mechanisms to suppress misalignment errors. Uniaxial tests were simulated numerically using the new stage and the sample. The analysis revealed that, irrespective of the degree and the source of the misalignment, its effect can be suppressed on the entire gauge length, and elastic modulus can be measured with less than 1% error.

In the present chapter, we explore the effect of misalignment error experimentally. Using a MEMS based tensile stage, we will show that even small misalignment error, often unavoidable at micro scale, can result in large non-uniform stress on the gauge cross section, unwanted non-linearity in testing and large error in measured elastic modulus unless the data is interpreted appropriately. We also experimentally demonstrate that the proposed sample design which was theoretically considered in Chapter 3 significantly minimize or eliminate any misalignment during in situ mechanical tests.

4.1 In situ uniaxial testing apparatus

Here we experimentally fabricate the uniaxial testing stage and sample shown in Fig. 3.1 and 3.6, respectively. The detailed introduction for the stage and sample can be found in Chapter 3. For the microfabrication of the stage and sample, we followed the fabrication procedure summarized in Fig. 4.1. First, Al films are deposited on both sides of a silicon wafer followed by photoresist (PR) spin-coating (a). Then, the PR layers and Al films are
patterned by lithography (b) and wet etching (c), respectively. The patterned Al layers serve as masks during ICP-DRIE process. The silicon wafer is etched from the top to make the grooves of grips (d) and then from bottom to release free-standing structures (e). Finally, the PR layers and Al masks are removed (f).

Scanning electron microscope (SEM) images are taken to measure strain and stress of the specimen simultaneously. Matlab based correlation algorithm is used to track arbitrary marks automatically with resolution enhancement up to 1/10 of pixel size. The image tracking marks are created on the uniaxial testing stage and the specimen by FIB milling (Fig. 4.3). The marks on the stage are located on the grip and the force sensing gauge. For the specimens, several marks are created along the vertical surface, for instance near the top, bottom, and neutral planes. These marks allow independent measurement of the strains corresponding to the different planes away from the neutral plane.

4.1.1 Experimental calibration for a force sensor

We previously considered the effect of geometric nonlinearity of the force sensing beams where force-displacement relation becomes

\[ f = k\delta + \bar{k}_3\delta^3 \]  \hspace{1cm} (4.1)

where \( \bar{k} \) and \( \bar{k}_3 \) are linear and nonlinear spring constants (see Chapter 3 for further details). We calibrated the values of \( \bar{k} \) and \( \bar{k}_3 \) experimentally by applying a known force on the beam and measuring its deflection. A weighing scale is used to measure the force. Optical microscope is used to measure the displacement, \( \delta \), of the force sensing beams. Figure 4.2 shows the force-displacement curve and the corresponding best fit curve with \( \bar{k} = 0.322 \text{mN/\mu m} \), and \( \bar{k}_3 = 7.259 \times 10^{-4} \text{mN/\mu m}^3 \). These values are close to the predicted values of \( \bar{k} = 0.325 \text{mN/\mu m} \) and \( \bar{k}_3 = 10.426 \times 10^{-4} \text{mN/\mu m} \). The predicted values are slightly higher due to overestimation of the size of the beams.
In order to test materials with various elastic moduli, the stiffness of the force sensing beams can be modified such that the magnitude of both the elongation of specimens, $\Delta$, (for strain measurement) and deformation of the force sensing beam, $\delta$, (for force measurement) are large enough to be measured from the SEM images. For linear elastic sample, and for small strains, the ratio of $\Delta$ and $\delta$ can be written as (refer Section 3.1.1)

$$\frac{\Delta}{\delta} = \left( \frac{l_{SP}}{E_{SP}A_{SP}} \right) \left( \frac{16NE_{SB}b_{SB}^2h_{SB}}{l_{SB}^3} \right)$$ (4.2)

where $l_{SP}$, $E_{SP}$, and $A_{SP}$ are length, elastic modulus and cross-sectional area of sample for small $\delta$. For a given specimen with its dimension, the geometry of the sensor beams can always be modified to obtain a prescribed value of $\Delta/\delta$. For example, in order to test two materials, one is stiffer than the other by ten times, $b_{SB}$ need to be increased by 2.15 times for the stiffer material to ensure the same $\Delta/\delta$.

### 4.2 Influence of misalignment

We experimentally consider the misalignment errors associated with compliance of a stage and its gripping mechanism, and their influence on the stress non-uniformity in a test sample. For *in situ* tensile test, MEMS based stages and single crystal silicon (SCS) microspecimens are separately fabricated. The crystal orientation of all specimens along the longitudinal direction is $\langle 110 \rangle$. Thus the elastic modulus along the length of the sample is $E_{exa} = 169$ GPa[18, 68, 69].

#### 4.2.1 Compliance of the stage

In order to consider the effect of the stage compliance on the stress state of the sample and compare with the predictions in Chapter 3, shallow grooves (about 30$\mu$m deep) are used for gripping the sample so that the sample is loaded near the top surface of the stage.
This asymmetry of the sample location with respect to mid-height of the stage results in substantial bending on the stage, i.e., $M_{st} \approx rf/2$ where $r$ is the height of the stage and $f$ is the load factor (see Chapter 3). A short SCS sample ($h/l \sim 1$) is used so that the effect of the deformation of the sample on the sample moment, $M_{sp}$, becomes negligible up to fracture strength, i.e., $f^* < 0.1$. For small $f^*$, Eq. 2.13 becomes $e_m^{TM} \approx \pm \frac{6c^*}{h}$ by using Taylor expansion and the measured strains, $\varepsilon_m = \varepsilon_U(1 + e_m)$, can be written as $\varepsilon_m = \varepsilon_U(1 \pm \frac{6c}{h})$ at the top and at the bottom of the sample, respectively. Hence, the misalignment value, $c$, can be quantified from the strain ratio by

$$\frac{d\varepsilon_{bottom}}{d\varepsilon_{top}} \approx \frac{1 - 6c/h}{1 + 6c/h}. \quad (4.3)$$

Figure 4.4(a) shows the average stress ($\sigma = f/A$) as a function of measured strain at the top and bottom of the sample. Due to bending in the sample induced by misalignment error, bottom strain $\varepsilon_{bottom}$ is negative with compressive stress during the initial phase of loading. With increased loading, the stage continues to bend and the sample reverses curvature. The rate of change of the bottom strain with $\sigma$ increases compared to that of top strain. These behaviors are qualitatively consistent with the quantitative predictions in Chapter 3.

In Region A, $\varepsilon_{top} > 0 > \varepsilon_{bottom}$ and $d\varepsilon_{bottom}/d\varepsilon_{top}$ increases from about -1/2, approaching zero, which implies that $h/2 > c > h/6$ (Eq. 4.3), i.e., initially, the contact between the sample and the grip occurs near the top of the sample. With increasing load, the contact moves towards the center of the sample. This is due to the tapered surface of the specimen and the stage fabricated by ICP-DRIE as observed from a SEM image in Fig. 4.5. As the load increases, $\varepsilon_{top}$ and $\varepsilon_{bottom}$ both become positive, and beyond a critical value of the load, $d\varepsilon_{bottom}/d\varepsilon_{top} \approx 2$ (Region B in Fig. 4.4(b)) when $c = \frac{h}{18} = -1.67 \mu m$ by using Eq. 4.3, i.e., the point of contact moves slightly below the neutral axis.

In order to evaluate the elastic modulus of the specimen from the results in Region B, the linear least-square curve-fit is used (strain<1%). The results are $E_{top} = 264$ GPa and
$E_{bottom} = 133$ GPa. By using the average strain, $E_{mean} = \sigma/\varepsilon_{ave} = 177$ GPa (4.7% relative error with respect to $E_{exa}$). This error is due to the asymmetry in the location of the image tracking marks with respect to the neutral axis of the sample. Strains measured from equidistant from the neutral axis would result in a better estimate of the elastic modulus.

### 4.2.2 Gripping mechanism

We now consider the effect of gripping error on the non-uniform stress in the sample. In order to suppress the effect of the compliance of the stage, a new stage in Fig. 4.6 is fabricated where the depth of the grips is half the thickness of the stage so that $M_{st} \approx 0$. The resolution of the force measurement is enhanced by two orders of magnitude with reduction in the width of the force sensing beams from $60\mu m$ to $12\mu m$. These thinner force sensing beams lead to large transverse deformation and hence the nonlinear spring constant, $\bar{k}_3$, is properly considered as discussed in Section 3.1.1 and Section 4.1.1. The specimen has additional free stranding arms that serve as strain measurement gauge with image tracking marks on them (Fig. 4.6(c)-(d)). Thus the sample does not need to be exposed to FIB or the electron beam in the SEM.

The stress-strain and $\varepsilon_{bottom}$ versus $\varepsilon_{top}$ curves for $540\mu m$-long specimen with cross section area $A = 77\mu m^2$ are shown in Fig. 4.7(a)-(b). All dimensions of the specimens are measured within 0.5% error based on SEM images. The apparent moduli of the specimen are $E_{top} = 189\pm 2.6$ GPa (95% confidence interval) with adjusted R-square, $R^2_{adj} = 0.9957$, and $E_{bottom} = 157 \pm 2.0$ GPa (95% confidence interval) with $R^2_{adj} = 0.9984$. $E_{mean} = \sigma/\varepsilon_{ave}$ is 171 GPa with 1.2% relative error with respect to $E_{exa}$. The slope of the $\varepsilon_{bottom}$-$\varepsilon_{top}$ curve is $1.2 \pm 0.01$ (95% confidence interval) with $R^2_{adj} = 0.9933$ by linear fit which indicates improvement in the stress uniformity in the sample compared to the results in Fig. 4.4(b). However, the slope is still higher than one and the stress is non-uniform across the cross section. Since the slope remains constant with increase in load, it implies that the load point does not move along the height of the sample (as was the case in Fig 4.4 during the early stage of loading).
The higher slope of the $\varepsilon_{\text{bottom}} - \varepsilon_{\text{top}}$ curve is due to taper in the grips as shown in Fig 4.5.

In Chapter 3, we showed that the stress-strain response of the sample is highly sensitive to the boundary conditions at the grips for $M_{st} \approx 0$. For a sufficiently long sample, the slope of the $\varepsilon_{\text{bottom}} - \varepsilon_{\text{top}}$ curve can approach to 1 due to the bending of the sample or full engagement between the grip and the sample surfaces (see Section 3.1.2). For the latter case, the rotation of the sample ends is restricted by the grips as schematically shown in Fig. 3.3 in Chapter 3.

Though the experimental results in Fig. 4.7(a)-(b) show significant reduction in the non-uniform stress in the specimen by eliminating the effect of stage compliance, the intrinsic bending stress in the sample is observed due to the gripping error. Even if all the sidewalls of both a sample and stage are perfectly vertical, irregular topography, e.g. scallop formation on sidewalls due to the subsequent Bosch process, still induces asperity contact at the grip and hence transverse misalignment. The degree of the misalignment likely varies from sample to sample and hence it is difficult to control and quantify such misalignment at the micro/nano-scale. Therefore, a self-alignment mechanism such as a hinge is required for pure uniaxial loading and simple interpretation of experimental data as in macroscale counterpart of tensile test.

Two important comments on effect of gripper compliance on force measurement and the stress state of the sample are (1) the specimen, gripper, and force sensing beams are in series and hence even if there is elastic deformation of the grippers due to their compliance, load can be evaluated by measuring deformation of the force sensing beams alone and (2) the stiffness of the grippers is considerably higher compared to those of the sensor beams and the specimen (Fig. 4.8). Figure 4.8(b) shows substantial deformation of the stage and large strain in the specimen compared to the gripper.
4.3 Uniaxial loading

We proposed a novel design for a sample with hinge-like self-aligning mechanisms in Chapter 3. The analysis of the hinged sample showed uniformity of stress across the cross section and along the length even in the presence of gripping error. Here, we test the predictions experimentally.

4.3.1 Self-aligning mechanism

Following Chapter 3, the self-aligning mechanism is schematically shown in Fig. 4.9(a)-(b) which consists of gripped parts, a specimen, and self-aligning beams. The dimensions of different parts are defined by \( W_i, H_i \) and \( L_i \) where \( i = 1, 2, \) and \( 3 \) correspond to the specimen, self-aligning beam 1, and self-aligning beam 2, respectively.

As we discussed in the previous chapter, the first design criterion is that the bending stiffness of the self-aligning beams is much smaller than that of the specimen about desired direction. For instance, beam 1 and beam 2 are designed to be compliant in out-of-plane and in-plane bending directions, respectively. Hence, the bending stiffness ratios between the specimen and the self-aligning beam 1 are

\[
\frac{k_1}{k_2} = \frac{W_1 H_1^3 (2L_2)^3}{W_2 H_2^3 L_1^3} \gg 1 \text{ and } \frac{\hat{k}_1}{\hat{k}_2} = \frac{W_1^3 H_1 (2L_2)^3}{W_2^3 H_2 L_1^3} \ll 1
\]

for out-of-plane and in-plane bending stiffness, respectively. Likewise, the bending stiffness ratios of the specimen to the self-aligning beam 2 are \( k_1/k_3 \ll 1 \) and \( \hat{k}_1/\hat{k}_3 \gg 1 \). Hence, the beams behave as hinges and suppress both in- and out-of-plane misalignment at the grips.

The second criterion is that the maximum stress (average stress + max bending stress) in the self-aligning beams is smaller than the average stress at the gauge section during the uniaxial test. Thus, the gauge section will either yield or fracture before the hinges deviate from linear elasticity. For design of such self-aligning specimens, we consider a
specimen with one hinge for simplicity as shown in Fig. 4.10(a). Loading $f$ is offset by eccentricity $c$ which results in bending moment $M(x)$ on the specimen. Upon loading, the maximum bending stress occurs near the gripped ends. Then the criterion becomes: $f/A_{\text{gauge}} > f/A_{\text{hinge}} + M(x)(h_{\text{hinge}}/2)/I_{\text{hinge}}$ at $x = 0$ and $= l$ where $A_{\text{gauge}}$ is a cross sectional area of the gauge, $A_{\text{hinge}}$ is a cross-sectional area, $I_{\text{hinge}}$ is moment of inertia, and $h_{\text{hinge}}$ is the thickness of the self-aligning beams, respectively. Thus

$$1 > \frac{A_{\text{gauge}}}{A_{\text{hinge}}} \left( 1 + \frac{6c}{h_{\text{hinge}}} \right).$$

(4.4)

In order to ensure prior yielding of gauge to self-aligning beams even with the maximum non-uniform stress, we assume $c = h_{\text{gauge}}/2$. Then Eq. 4.4 becomes

$$1 > I^* \left( 1 + \frac{3\beta}{\beta^2} \right)$$

(4.5)

where $\beta = h_{\text{gauge}}/h_{\text{hinge}}$ and $I^* = I_{\text{gauge}}/I_{\text{hinge}}$. Using Eq. 4.5, $\beta > 3.30, 15.32, 30.33, 60.33, 69.33$ satisfy the second criterion for $I^* = 1, 5, 10, 20, 23$, respectively. This analysis indicates that it is possible to make a generalized self-aligning specimen for mechanical measurement of elastic and plastic material deformations even with maximum non-uniform stress. However, fabrication of the generalized specimen with large $I^*$, for example $I^* = 23$ and $\beta > 69.33$, may be challenging and hence design parameters $I^*$ and $\beta$ of the specimen might need to be altered based on characteristics of test materials to minimize fabrication effort. For example, Fig. 4.10(c)-(d) show analytical prediction of deformed shape and normalized bending stress by uniaxial loading for a plain sample and self-aligning specimens using the analytical model in [46]. These specimens are subjected to 0.01%, 0.05% and 0.2% strains. The dimension of the self-aligning specimens is $l_{\text{hinge}}/l = 1/3$ and $h_{\text{gauge}}/l = 1/50$ with different moment of inertia ($I^* = 5$ and $= 23$).

Note that non-uniform stress error approaches to zero with 0.05% strain for $I^* = 23$ and
with 0.2% strain for $I^* = 5$ while substantial nonuniform stress error is observed for the plain specimen up to 0.2% strain. Hence for mechanical measurement within small elastic strain, large $I^*$ might be sufficient, while for tests beyond 0.2% strain, it is important to consider the second criterion. It is worth noting that in the above analysis we consider the worst scenario by assuming the maximum eccentricity and no constrain on rotation of the sample at the gripped boundaries ($x = 0$ and $l$). Thus the values of $\beta$ are expected to be highly conservative.

### 4.3.2 Self-aligning sample

The self-aligning specimen shown in Fig. 4.9(c)-(d) is fabricated by microfabrication and FIB milling. The gauge section of the sample, 50µm-long with $A = 5\mu m \times 18\mu m$, is never exposed to FIB. We first focus on the performance of beam 1 (hinge 1) in Fig. 4.9(d), i.e., the sample has only one self aligning beam.

We use a self-aligning sample with $I^* = I_{gauge}/I_{hinge} = 1.5$ shown in Fig. 4.11(b1) where $I_{hinge}$ and $I_{gauge}$ are moment of inertia for the beam 1 (hinge 1) and sample (gauge length) (Fig. 4.9(d)). The length of beam 1 is about 160µm. The stress-strain relation and $\varepsilon_{bottom}$ versus $\varepsilon_{top}$ curve upon loading and unloading are shown in Fig. 4.11(a) and (b), respectively. The apparent elastic moduli of the specimen are $E_{top} = 180 \pm 4$ GPa (95% confidence interval) with $R_{adj}^2 = 0.9965$ and $E_{bottom} = 160 \pm 6$ GPa (95% confidence interval) with $R_{adj}^2 = 0.9989)$. $E_{maen}$ is 170 GPa with 0.6% relative error with respect to $E_{exa}$. The strain ratio, $\varepsilon_{bottom}/\varepsilon_{top} = 1.1 \pm 0.02$ (95% confidence interval and $R_{adj}^2 = 0.9959$), is obtained by linear fit. Error in the measured elastic modulus on the top of the sample is $e_m = 6.1\%$, and at the bottom of the sample is 5.6%. The difference in the magnitude of $e_m$ at the top and at the bottom of the sample is further reduced.

Next, we explore the design parameter space by using the same sample but with increased $I^*$ ($I_{gauge}/I_{hinge} = 23$) as shown in Fig. 4.11(b2). The $\varepsilon_{bottom}-\varepsilon_{top}$ curve is shown in Fig. 4.11(b). As expected, the stress uniformity in the sample is further improved for large $I^*$.
since $\varepsilon_{\text{bottom}}/\varepsilon_{\text{top}} = 1.02$.

Finally, Fig.4.11(c) shows the analytical prediction of the $\varepsilon_{\text{bottom}}$-$\varepsilon_{\text{top}}$ curve for $I^{*} = 1.5$ and $= 23$ with load $f$ at the top of the sample (the maximum possible gripping misalignment) [46]. For $I^{*} = 1.5$, $\varepsilon_{\text{bottom}}/\varepsilon_{\text{top}} = 1.06 \pm 0.05$ (95% confidence interval) with $R_{\text{adj}}^{2} = 0.9917$ when the slope is evaluated between AB while $\varepsilon_{\text{bottom}}/\varepsilon_{\text{top}} \approx 1$ (even close to $\varepsilon \sim 0$) for $I^{*} = 23$. Note the transition in $\varepsilon_{\text{bottom}}/\varepsilon_{\text{top}}$ from negative to positive occurs at $\varepsilon_{\text{top}} < 0.05\%$ for both the cases in Fig.4.11(c). Experimentally, the transition from negative to positive slopes for $I^{*} = 23$ occurs at much smaller values of strain that is not detectable, since the misalignment at the grip was less than that considered in the analysis.

We experimentally demonstrate that the self-aligning sample significantly reduces the non-uniform stress. With two hinges, the influence of unaccounted misalignment errors is eliminated or minimized even when the misalignments at the grips induce in- and out-of-plane bending. Thus the hinges allow almost ideal uniaxial loading for the micro/nano scale uniaxial tests. Hence, even when single strain measurement either at the top or bottom of the sample is available, elastic modulus can be measured accurately.

Note that for mechanical testing of curled specimens, magnitude of eccentricity of loading can be much larger than a perfectly flat specimen. For example, consider a curled specimen with an initial radius of curvature $r_{o}$. Then, the eccentricity is a function of axial coordinate of the specimen as $c_{T}(x) = c + r_{o}(1 - \cos(x/r_{o}))$ where $c$ is the eccentricity at the grip. As a result, larger non-uniform stress is induced and hence hinge structures become more important to suppress the non-uniform stress of such curled specimens during mechanical measurement. It is worth noting that even with the hinge structures, data interpretation of uniaxial testing requires careful measurement of the geometry of the specimen (i.e., the coordinates of the centerline) during loading and the residual stress state prior to loading, since the stress state of the specimen is superposition of initial residual stress, bending stress due to off-axis loading by $c_{T}(x)$, and uniaxial stress.
4.4 Conclusion

In uniaxial tension and compression tests, the axis of loading is often misaligned with the sample axis. Misalignment error results in non-uniform stress on the cross section of the sample as well as along its gauge length. More importantly, the degree of misalignment may change in a given experiment with increasing load due to the shift of the location of the load at the grip. The shift is due to the compliance of the sample and/or the loading stage. This results in a nonlinear relation between the measured applied load and strain at a point on the surface of the sample, even though the strains are small and the material is linear elastic. At micro and nano scale, misalignment error can be significant due to limited precision on the grip between the sample and the loading stage compared to the size of the sample. Due to the non-uniformity in stress and strain in the sample, elastic modulus measured from the average stress (force/cross sectional area) and a surface strain can be significantly different from the actual value. We have used this error in elastic modulus as a measure of misalignment error. In order to carry out uniaxial test on small scale samples, we proposed a novel MEMS stage with in-built grips and self aligning mechanism, and a sample with built in hinges. The stage allows in situ tension and compression tests of micro/nano scale dog-bone shape samples. In Chapter 3, we studied the stage and the sample theoretically and computationally to explore the origin of misalignment and the design space for the proposed stage and the sample to minimize misalignment error. Here, we studied the stage and the sample experimentally by carrying out tests on a well characterized material, single crystal silicon. First, we studied the influence of stage compliance on stress non-uniformity in the sample without the hinges. We found, the error in elastic modulus measurement from surface strain may lead to more than 50% error, and a variation in $d\sigma/d\varepsilon$ with increasing load in the linear elastic silicon sample. Next, we minimized the effect of stage compliance by placing the sample (without the hinges) at its mid-height, and studied the effect of gripping misalignment alone. The gripping error induces up to 12% error in apparent elastic modulus.
At the micro/nano scale, the gripping error is unavoidable since any irregular topography, for example taper and asperity, of either the sample and the stage results in such error. The hinged sample, on the other hand, suppresses misalignment error almost entirely and gives an elastic modulus with better than 99% accuracy. The experimental results matched closely with all the predictions made in Chapter 3. Even though the stage and sample were studied in light of elastic materials, they can also be used to study materials beyond elastic limit with uniaxiality of loading.
4.5 Figures

Figure 4.1: The schematic of fabrication flow: (a) Al deposition and photoresist (PR) spin-coating on both sides of a silicon wafer, (b) patterning PR layers by lithography on both sides, (c) patterning the Al films by wet etching, (d) ICP-DRIE etching to make the grooves of grips, (e) to release free-standing structure, and (e) removal of the Al film and PR.
Figure 4.2: The calibration of the force sensor. The solid circles show experimentally measured force-displacement response. The experimental data is fitted by $f = \bar{k}\delta + \bar{k}_3\delta^3$ and linear and nonlinear spring constants are obtained. Also, the spring stiffness of the force sensing beams is predicted by a linear ($\bar{k}\delta$) and a nonlinear ($\bar{k}\delta + \bar{k}_3\delta^3$) approximations (with $\bar{k}$ and $\bar{k}_3$ predicted from geometry and elastic property of single crystal silicon).
Figure 4.3: SEM images of the tensile stage and the specimen: (a) overall view of the stage, (b) a specimen mounted on the grips, (c) image tracking marks on the specimen (dotted circles) and on the stage (solid circle on grip and dash-dot circle on force sensing gauge), and (d) a image of an assembly using the micromanipulator (FIB omniprobe). The specimen is glued to the manipulator by Pt deposition. After the assembly, the specimen is released from the manipulator by ion milling.
Figure 4.4: Experimental results for a 30\(\mu\)m-long specimen: (a) stress-strain response and (b) strain ratio between \(\varepsilon_{\text{bottom}}\) and \(\varepsilon_{\text{top}}\). In (a) \(E_{\text{mean}} = \sigma/\varepsilon_{\text{ave}}\) where \(\varepsilon_{\text{ave}} = (\varepsilon_{\text{top}} + \varepsilon_{\text{bottom}})/2\).
Figure 4.5: A SEM image of tapered sidewall profile due to ICP-DRIE.
Figure 4.6: SEM images of the modified tensile stage: (a) overall view of the stage and the specimen, (b) 90µm-deep groove of a grip, (c) specimen with strain measurement gauges, (d) image tracking marks on the strain measurement gauges (dotted circle) and on the stage (solid circle on grip and dash-dot circle on the force sensing gauge).
Figure 4.7: Experimental and analytical results for a 540μm-long specimen with cross section area $A = 77\mu m^2$. Stress-strain response and strain ratio between $\varepsilon_{\text{bottom}}$ and $\varepsilon_{\text{top}}$ from experimental measurement are shown in (a) and (b).
Figure 4.8: (a) The schematic of the grips and sample. (b) The strain of the sample and stage from the side view (see (a)) by using a finite element analysis as in the previous chapter.
Figure 4.9: The self-aligning specimen: (a)-(b) the schematic for top view and side view, (c) a SEM image of the sample loaded on the stage, and (d) zoom in view of the sample.
Figure 4.10: (a)-(b) Schematics of self-aligning and plain specimens. (c) Transverse deformation ($\chi$) of the specimens due to $f$ with eccentricity $c$ as a function of axial coordinate of the specimens ($x$). Applied loads are corresponding to 0.01%, 0.05% and 0.2% strains. (d) Normalized bending stress by uniaxial loading as a function of $x$. The dimension of the specimen in (c) and (d) is $h_{\text{gauge}}/l = 1/50$ and $l_{\text{hinge}}/l = 1/3$. 

63
Figure 4.11: The experimental and analytical results for a self-aligning sample. The gauge length is 50\(\mu\)m with cross section area 90\(\mu\)m\(^2\). Stress-strain response and strain ratio between \(\varepsilon_{\text{bottom}}\) and \(\varepsilon_{\text{top}}\) from experimental measurement are shown in (a) and (b). The strain ratio by using analytical model in Section 3.2 is shown in (c). (b1) and (b2) are the specimen with \(I^* = 1.5\) and = 23, respectively, where \(I^* = I_1/I\). Note that stress-strain responses upon loading and unloading designated by dotted lines in (a) follow linear relation consistently.
Chapter 5

A novel method for in situ thermo-mechanical testing for micro/nanomaterials

As discussed in Chapter 1 in great details, thermo-mechanical properties of small materials are of great interest to scientists and engineers with rapidly expanding applications of micro/nano technologies even at high temperature. Although in situ thermo-mechanical testing is attractive for studying small scale materials, such in situ characterization of micro/nanoscale samples involves several challenges, e.g., the fabrication and handling of the small samples and high resolution in strain/stress measurements. Also, at high temperature, force measurement for in situ material tests becomes more difficult since traditional force measurement methods (load cell or strain gauge) and a MEMS based method (microfabricated silicon force sensing beams) are often not applicable due to strong coupling between temperature and fundamental mechanisms of force measurement. In this chapter, we present a novel SiC stage which overcomes those challenges and allows in situ uniaxial testing of the micro/nanoscale samples at high temperature with concurrent in situ temperature measurement using a cofabricated temperature sensor.

5.1 A novel SiC MEMS stage

The MEMS SiC stage allows high temperature material testing of independently fabricated samples with a built-in temperature sensor for in situ temperature measurement. The overall size of the stage is small enough to perform in situ tests in SEM and TEM. Three dimensional schematic of the experimental setup is shown in Fig. 5.1. Following Chapter 3 and Chapter 4, the stage has two grooves which serve as grips for a dog bone shape sample and hence
the stage can be used to test independently fabricated samples without limitation on sample
dimensions and materials. Figure 5.1(c)-(d) show a dog-bone specimen before and after
assembly with the stage by micromanipulator. Like the silicon based stage in Chapter 3
and Chapter 4, the specimen is loaded by deforming the stage using a piezoelectric actuator
while a force-deformation relation is analyzed from SEM images. See Chapter 3 and Chapter
4 for further details on the mechanical measurement.

For in situ heating of the sample during mechanical test, the stage is electrically connected
to power supply through copper wires and metal pillars as shown in Fig. 5.1(b) and hence
by applying electrical potential between the two wires, the SiC stage is resistively heated. In
order to measure temperature of a sample during uniaxial tests, the stage has a cofabricated
bimetal type temperature sensor near the grips as shown in Fig. 5.1(a). The detailed
theoretical analysis on the temperature sensor is discussed in the following section. Note
that the SiC stage is thermally and electrically isolated from an SEM sample holder by a
macor insulator due to its small thermal conductivity (1.46 W/m°C) and high resistivity
(>10^{16} \text{cm} \cdot \Omega) (material data sheet from Owens corning) as shown in Fig. 5.1(b).

Silicon carbide is used as a structural material for the stage due to (i) its outstanding
mechanical properties at high temperature, (ii) semiconductive characteristics, and (iii) large
heat conductivity [70, 71]. For example, SiC has high melting temperature (2830\degree C) and
shows small variation in elastic modulus with variation of temperature (elastic modulus de-
creases by 4% at 1000\degree C with respect to the room temperature [72]). This allows in situ force
measurement by measuring deformation of force sensing beams even at high temperatures.
Also, semiconductive characteristic of SiC allows resistive heating of the stage. Finally SiC
has large heat conductive coefficient (340 W/m\degree C, the material data sheet from Cree) so
that heat can be transferred to the test sample efficiently.

It is worth noting that variation of temperature imposes unavoidable challenges in loading
conditions. For example, a stage and sample can have a different coefficient of thermal
expansion which may cause undesired loading/unloading of a sample with variation of tem-
perature. Also, if a sample is glued to loading frames, e.g., metal deposition, effect of thermal stress between the sample and glue should be carefully considered. The presented SiC stage has advantages in such challenging scenarios since (i) the stage has support beams, U-springs, the sample and the sensor beams, all in series and hence any loading, even due to thermal expansion mismatch, on the sample can be measured, and (ii) the sample is assembled with the stage, and hence stress free condition on the sample at the initial stage of loading can be ensured at any given temperature [61].

5.1.1 Temperature sensor: bimetallic type

For in situ temperature measurement, a bimetal type temperature sensor, which consists of two different materials with mismatch in coefficient of thermal expansion (CTE), is used to convert a temperature variation into transverse deformation of the sensor. Three dimensional schematic of such sensor is shown in Fig. 5.2(a). Assuming materials are homogeneous and linear elastic, and CTE and a cross-sectional area are constant during uniform heating, the curvature of radius \( r \) for temperature change \( \Delta T \) can be written as [73]

\[
\frac{1}{r} = \frac{6w_1w_2E_1E_2t_1t_2(t_1 + t_2)(c_1 - c_2)\Delta T}{(w_1E_1t_1^2)^2 + (w_2E_2t_2^2)^2 + 2w_1w_2E_1E_2t_1t_2(2t_1^2 + 3t_1t_2 + 2t_2^2)}
\] (5.1)

where \( w_i, t_i, l_i, c_i, E_i \) are width, thickness, length, coefficient of thermal expansion, and Young’s modulus of material 1 \((i = 1)\) and material 2 \((i = 2)\), respectively. The total transverse deformation, \( \delta_T \), of the temperature sensor (see the sideview in Fig. 5.2(a)) is

\[
\delta_T = r(1 - \cos \theta) + l_2 \sin \theta
\] (5.2)

where \( \theta = l_1/r \). Note that the magnitude of \( \delta_T \) is linearly dependent on \( l_2 \) for small \( \theta \) and hence the resolution of the sensor can be enhanced with increase of \( l_2 \).

We consider a temperature sensor consisting of two materials for high temperature ap-
applications, platinum (material 1) and silicon carbide (material 2) (see Fig. 5.2(a)). The dimensions of the sensor are $w_1 = 256 \mu m$, $l_1 = 200 \mu m$, $l_2 = 1120 \mu m$, $t_1 = 10 \mu m$, and $t_2 = 35 \mu m$. The material properties are $E_1 = 168$ GPa and $c_1 = 8.8 \times 10^{-6} ^\circ C$ for platinum (material 1) [74] and $E_2 = 448$ GPa and $c_{SiC} = 4.3 \times 10^{-6} ^\circ C$ for silicon carbide (material 2) [75, 76]. Transverse displacement of the temperature sensor is shown in Fig. 5.2(a) as a function of temperature. We also consider the temperature sensor with the same dimensions and materials by using a finite element analysis which matches with the analytical prediction as the slopes of analytical and FEA predictions are 11.12nm/$^\circ C$ and 10.90nm/$^\circ C$, respectively, in Fig. 5.2(a).

To further increase the resolution of in situ temperature measurement, we propose a novel design of the temperature sensor where bi-metal temperature sensors are in series as shown in Fig. 5.2(b). Hence, for small $\theta$, the total displacement of $N$ sensors in series is $\sim N\delta T$ by superposition of deformation of each sensor. In order to validate this simple analysis, we simulate a temperature sensor with $N = 4$ as shown in Fig. 5.2(b) where the slopes of $(\delta, \Delta T)$ at A, B, C, and D are 10.56nm/$^\circ C$, 21.55nm/$^\circ C$, 32.55nm/$^\circ C$, and 43.52nm/$^\circ C$.

5.1.2 Analytical model

Here we consider electrical power of the SiC stage during Joule heating. To study the effect of geometry of the stage on the electrical power analytically, we first simplify the SiC stage shown in Fig. 5.3(a) by a lumped component model in Fig. 5.3(b). The resistors in Fig. 5.3(b) represent resistance of the circuit components on the SiC stage between designated locations by A-F and the resistance of the resistors is obtained from geometry of the stage and known resistivity of the SiC wafer ($\rho^{-1} = 0.07 \text{cm} \cdot \Omega$, material data sheet from Cree). The contact between the stage and macor insulator frame is ignored in the analysis due to large resistivity of the macor.

From Fig. 5.3(b) and by using Kirchhoff laws, the lumped model can be written as the
following linear system

\[
\begin{align*}
    i_T &= i_1 + i_1^* = i_5 + i_5^* \\
    i_1 &= i_2 + i_3 \\
    i_4 &= i_3 + i_3^* = i_6 + i_6^* \\
    i_5 &= i_6 + i_2 \\
    V &= i_1 R_1 + i_2 R_{DE} + i_5 R_{EF} \\
    0 &= i_3 R_{DG} + i_4 R_{GH} + i_6 R_{HE} - i_2 R_{ED} \\
    i_i &= i_i^*
\end{align*}
\]

(5.3)

where \( i = 1, 2, 3, 5, \) and 6 due to symmetric geometry of the stage about A-F and \( R_1 = R_{AB} + R_{BC} + R_{CD} \). From Eq. 5.3, the normalized electrical powers at the sample, between D-E and D’-E’, and at two U-beams are

\[
\begin{align*}
    \bar{W}_{GH} &= \frac{2R_{DE}^2 R_{GH}}{C_1(R_{EF}C_2 + R_tC_1 + R_{DE}C_3)} \\
    \bar{W}_{DE} &= \frac{2R_{DE}C_2}{C_1(R_{EF}C_2 + R_tC_1 + R_{DE}C_3)} \\
    \bar{W}_{BC} &= \frac{2R_{BC}C_1}{R_{EF}C_2 + R_tC_1 + R_{DE}C_3}
\end{align*}
\]

(5.4)

where \( C_1 = R_{DE} + R_{DG} + 2R_{GH} + R_{HE}, C_2 = R_{DG} + 2R_{GH} + R_{HE}, \) and \( C_3 = R_{DG} + 2R_{GH} + R_{EF} + R_{RHE}. \) Figure 5.3(c)-(e) show normalized power at the U-beams, frames, and sample, respectively, as a function of sample resistance. The U-beams are connected to other components in series and have large resistance due to their large length to cross-section ratio and therefore the normalized power at the U-beams is greater than 93.4% (see Fig. 5.3(c)) within \( 0 < R_{GH} < 8000 \Omega. \) Therefore, the U-beams are a major heat source during Joule heating. Note that \( R_{DG} \gg R_{DE} \) and \( R_{HE} \gg R_{DE} \) due to the geometry of these components, i.e., \( l_{DG}/A_{DG} \gg l_{HE}/A_{HE} \gg l_{DE}/A_{DE}. \) Since DG and HE are in parallel with DE (see Fig. 5.3(a)-(b)), the current through the sample (\( i_4 \) in Fig. 5.3(b)) is much smaller than that through the frame of the SiC stage (\( i_2 \) and \( i_2^* \)) and hence \( \bar{W}_{GH} \ll \bar{W}_{DE} \) as shown in Fig.
5.3(d) and (e). For example, consider electrical power of a 200µm-long SCS sample with cross section area 25µm$^2$ and $\rho^{-1} = 5.5\text{cm} \cdot \Omega$ which results in $\bar{W}_{GH} = \sim 10^{-5}\%$ from Eq. 5.4. This result indicates that electrical power consumed by the sample can be ignored. Hence sample temperature is insensitive to the sample resistance and potential material damages of micro/nanomaterials due to high current directly through the sample, e.g., electromigration, can be prevented.

For the geometry design of the SiC stage, consider a sample with $R = 42\Omega$ which corresponds to the maximum normalized power at the sample (0.023%) in Fig. 5.3(e). In order to test such samples and further reduce the effect of sample resistance on sample temperature, geometry of the stage can be altered such that $d\bar{W}_{GH}/dR_{GH}$ is away from the resistance of the sample from Eq. 5.4. For example, decrease of $R_{DE}$ by increase of the cross section at DE results in substantial reduction in the electrical power consumption at the sample as shown in Fig. 5.3(f).

5.1.3 Numerical model

In the previous section, we considered the electrical power at the different components of the SiC stage during Joule heating by using the lumped model, but the model only predicts electrical response of the SiC stage. To obtain the temperature profile of the stage and sample, we carry out a finite element (FE) analysis on a one-to-one scale three dimensional solid model (see Fig. 5.1(b)) with multiphysics package, COMSOL.

For conductive heating, we assume that the resistivity of heating element, the SiC stage, is linearly dependent on temperature ($\rho = \rho_o(1+\alpha(T-T_o))$ where $\alpha$ is temperature coefficient, $\rho$ and $\rho_o$ are conductivity of the material at measured temperature $T$ and reference temperature $T_o$, respectively [73]. For thermal heat transfer analysis, we consider thermal radiation due to high temperature and ignore thermal convection due to vacuum environment during in situ testing. We assume that ambient temperature is constant (25°C) due to small size of the SiC stage compared to an SEM chamber. For stress-strain analysis, all materials
are assumed to be homogeneous, isotropic, and elastic. During the multi-physics analysis, continuous boundary conditions are used for all surface contacts between different parts. Here, we only consider steady-state solutions in order to simulate a quasi-static condition during experimental measurement.

Electrical, thermal, and mechanical responses of the SiC are shown in Fig. 5.4(a)-(c), respectively, for input voltage 50V where $\alpha_{\text{SiC}} = 1.5 \times 10^{-2}/\degree\text{C}$ [77]. Electrical potential field in Fig. 5.4(a) shows the largest potential drop at U-beams as predicted by the lumped model. The corresponding temperature by Joule heating is shown in Fig. 5.4(b) where the maximum temperature occurs at the U-beams. Note that the macro frame insulates the stage electrically and thermally in Fig. 5.4(a)-(b). Thermal isolation is important as thermal damage of imaging equipment or other parts due to high temperature during in situ thermo-mechanical study is potentially a critical issue. In Fig. 5.4(c), the mechanical response of the temperature sensor due to variation of temperature is shown where the materials and geometry of the temperature sensor are the same as in Section 5.1.1.

Next, we numerically consider variation of temperature across the sample for a 200$\mu$m-long SCS sample with $\rho^{-1} = 5.5\text{cm} \cdot \Omega$. Figure 5.5(a) shows the temperature profile of the sample across its length for input voltage 50V. Note that temperature variation across the sample is $379.3 \pm 3.64\degree\text{C}$ and the normalized temperature varies between 100% and 97.32% (with 2.7% variation with respect to maximum temperature). In order to study the effect of sample resistance on the sample temperature, we perform parametric sweep in resistivity of sample by using FEA and confirm that sample temperature is insensitive to sample resistance due to small electrical power through the sample which agrees with the conclusion from the lumped model. Finally, temperature profile of the sample with input voltage is shown in Fig. 5.5(b) where difference in the temperature between the both ends of the sample is consistently small (within 3% variation with respect to maximum temperature) up to 120 V.

Our numerical simulation shows uniform temperature profile across the sample. This
is important to study size dependent thermo-mechanical properties as a large temperature gradient along the sample may result in local plastic deformation of material, even with uniaxial loading, at a point where the temperature is higher. Also, a large temperature gradient can lead to nonuniform stresses due to variation in thermal expansion across the sample.

### 5.2 In situ uniaxial measurement at high temperature

In the previous section we theoretically considered electrical, thermal, and mechanical responses of the SiC stage. Here in order to validate our theoretical predictions, we fabricate the SiC stage and experimentally carry out high temperature uniaxial testing of SCS microsamples in SEM.

The fabrication of the SiC stage is shown in Fig. 5.6. From the backside of a 256µm-thick 6H SiC wafer, we create four through holes which serve as alignment marks (21Amp and 40 passes) and locally thin the wafer to reduce the height of the temperature sensing beam (21Amp and 6 passes) by using laser milling (Potomac). The laser medium is neodymium-doped yttrium aluminum garnet (Nd:YAG) with wavelength 1064nm and the spot size is about 30µm (CEO SS-010R). Next we flip and align the wafer by using the alignment marks. From the top side, the temperature sensing beam is created (19Amp and 10 passes) and other inner structures of the SiC stage are fabricated (21Amp and 50 passes). Then the outer frame of the stage is cut (21Amp and 45 passes). During the laser milling, debris (silicon oxide) deposition occurs on the SiC stage which is removed by HF wet etching as shown in Fig. 5.6(g)-(h). The groove of the grips is created by focused ion beam milling. Finally, platinum is deposited on the sidewall of the temperature sensing beam to make a bimorph temperature sensor using FIB deposition. Figure 5.7(a) shows an SEM image of the SiC stage.
5.2.1 **In situ temperature measurement**

Here we experimentally calibrate the temperature sensors on the SiC stage. During in situ calibration of temperature sensors, the SiC stage is placed on macor crucible and uniformly heated by using a 1000°C ESEM heater (see Fig. 5.8(a)). We precalibrated temperature at crucible surface as a function of heater temperature where the relation is nonlinear due to thermal radiation at high temperature. In order to quasi-statically correlate deformation of bimaterial sensors with variation of temperature, we incrementally increase the heater temperature up to about 600°C with 10min time intervals. With increase in temperature, a series of SEM images is taken and analyzed to measure displacement of the temperature sensor as shown in Fig. 5.8(b). $\Delta d - \Delta T$ curve in Fig. 5.8(b) shows a linear relation as predicted by the theoretical model in Section 5.1.1. By using linear least squares fit, the slope of $\Delta d - \Delta T$ is $15.50 \pm 0.91\text{nm/}^\circ\text{C}$ (95% confidence interval with $R_{adj}^2 = 98.81\%$).

The main source for the discrepancy between the numerical calibration ($10.90\text{nm/}^\circ\text{C}$) and experimental calibration ($15.50\text{nm/}^\circ\text{C}$) on the temperature sensor is the limited accuracy in laser milling and nonuniform topography of platinum deposition during microfabrication of the sensor. Note that $\Delta d - \Delta T$ responses for increase and decrease in temperature overlap each other indicating repeatability of bimaterial sensors.

It is worth noting that for in situ measurement where chamber pressure is typically on the order of $10^{-5} \sim 10^{-6}\text{torr}$, the effect of thermal oxide on the deformation of the temperature sensor can be ignored. For example, consider a temperature sensing beam with 20$\mu\text{m}$ in width. After 50hr of dry thermal oxidation of silicon carbide at 1100°C even in relatively high pressure environment (7.5torr) compared to the in situ environment, thickness of an oxide layer is about 10nm and hence the ratio of moment of inertia ($I_{\text{SiO}_2}/I_{\text{SiC}}$) at the cross section is in the order of $10^{-10}$[78].
5.2.2 In situ mechanical test of SCS samples at high temperature

In order to characterize the SiC stage, we measure elastic properties of SCS samples, a well characterized material at different temperatures [79, 80]. To independently measure sample temperature during Joule heating, we fabricate a sample with a temperature sensor as shown in Fig. 5.7(b)-(c). The SCS sample is fabricated by ICP-DRIE (see [47] for detailed fabrication procedure) and then Pt is deposited by using FIB deposition as shown in Fig. 5.7(c). During fabrication, the sample gauge is never exposed to ion beam to avoid effect of artificial defects such as implantation of gallium ions, formation of an amorphous layer, and surface roughening which may alter material responses [38]. The temperature sensor is precalibrated by the same calibration procedure discussed in the previous section. The orientation of all specimens along the longitudinal direction is <110>.

Dimension of the sample (see Fig. 5.9(a)) is $L = 226.6 \mu m$ (total gauge length) and $l = 131.7 \mu m$ (gauge length between the strain measurement arms) with uniform cross sectional area $20.68 \mu m^2$ along the gauge. To prevent thermal oxidation of a SCS sample during a mechanical test, all in situ SEM experiments are conducted in an environmental SEM chamber in argon environment. Note that although the SiC stage is not affected by oxidation during in situ test in SEM in normal dry mode due to limited oxygen in the chamber and subsequently slow oxidation rate as discussed earlier, we experimentally found that the stage cannot be used when water vapor is present as imaging media due to much higher imaging pressure (1.5-2.0torr) during ESEM and much faster oxidation rate for SiC with water vapor at high temperature [81].

As uniaxiality of loading is one of the key requirements for uniaxial tests, we first consider nonuniform stress state of the sample at the gauge due to gripping error, i.e., the asymmetric gripping of the sample with respect to sample neutral axis by eccentricity $c$. Following
Chapter 2, normalized nonuniform stress for a sample with height $h$ can be written as

$$
\epsilon_{m}^{TM} = \frac{\sigma_{B}}{\sigma_{U}} = \pm \frac{6c}{h} \frac{\cosh \left( \sqrt{f^*} \left(2x^* - 1\right)/2 \right)}{\cosh \left( \sqrt{f^*}/2 \right)} \quad (5.5)
$$

where $\sigma_{U}, \sigma_{B}$ are the uniaxial and bending stress and $f^*, x^*$ are normalized loading and normalized distance along the undeformed sample. For the given sample dimension, the normalized nonuniform stress for the maximum eccentricity of loading ($c = h/2$) is shown in Fig. 5.9. Due to the large ratio of height to length for the sample (of order $10^{-2}$), uniaxiality of loading on the gauge part of the sample is ensured even at low strain. In order to experimentally verify the uniaxiality of loading, we first test a single crystal silicon sample at room temperature. The stress-strain response of the sample due to loading/unloading is shown in Fig. 5.10(a). From the linear elastic stress-strain curves, we recover the elastic modulus of silicon along $<110>$ crystal orientation, $E = 170.6 \pm 2.7$ GPa (95% confidence interval) by least-square-linear fit (within 1.0% error with respect to known elastic modulus, 169GPa [18, 69, 82]).

Though we measured elastic response of a SCS sample without influence of nonuniform stress within small elastic strain regime due to the large $h/l$ ratio, considerable nonuniform stress is still expected near the both ends of the sample (see Fig. 5.9). This may result in local stress and strain gradients, premature apparent yielding, and nonuniform strain hardening with large plastic deformation. Hence, active self-aligning mechanism, for example, a hinge mechanism [46, 47] may be essential for improved accuracy of mechanical measurement.

In Section 5.2.2, we numerically showed that the macor substrate efficiently behave as thermal barriers between the SiC stage and other components of the testing apparatus. Here, we experimentally measure temperature at the piezoactuator ($T_p$), one of the most temperature sensitive parts near the heat source, with increase in temperature on the SiC stage ($\Delta T_{SiC}$). The temperature at the actuator is measured by thermo-couple wires with two different conditions: (i) passive cooling and (ii) water cooling. As predicted by numerical
experiment, the experimental results in Fig. 5.11(a) show that the macor substrate thermally isolate the heating element from the piezoactuator. This is evident from the large temperature difference between the SiC stage and actuator for the both passive and water cooling cases. However, the results also indicate that an active cooling system, i.e. water cooling system in Fig. 5.7(d), may be required to achieve higher temperature even when a heating element is small and consumes small power. For example, in Fig. 5.11(a) $T_p=34.2^\circ$C for $\Delta T_{\text{SiC}}=139.5^\circ$C with water cooling while $T_p=45.1^\circ$C $\Delta T_{\text{SiC}}=128.8^\circ$C with passive cooling.

Next, we measure stress-strain responses of the SCS sample with increase in temperature by Joule heating. The corresponding temperature from temperature sensors on the stage and sample is shown in Fig. 5.11(b). As theoretically predicted in Section , the slope of temperature with applied voltage for the stage and sample decreases at higher temperature when radiation causes significant heat loss. However, we find considerable discrepancy between FEA (see Fig. 5.5(b)) and experimental results in temperature difference at the stage and sample. This discrepancy is due to the assumption of perfect surface-to-surface contact in FEA while actual surface contact is influenced by asperity on both sample and grippers. Since it is difficult to experimentally characterize the exact condition for the surface-surface contact at the grips, a temperature sensor on the sample is essential for the accurate temperature measurement.

Finally, we obtain stress-strain responses of the sample at different temperatures with the water cooled stage. Figure 5.10(a)-(f) show the stress-strain responses of the SCS microsample based on a series of SEM images at the same magnification. Elastic moduli are obtained by least-square-linear fit at room to 403$^\circ$C with 95% confidence intervals. The reduced values match well with those reported in the literature [18, 69, 79, 80, 82]. Note that the confidence interval at 25$^\circ$C is much smaller than that at 341$^\circ$C since in the former case a larger number of data are available and stress-strain responses are corresponding to larger load which induces larger deformation of the force sensing beams as well as sample. Strategies to study such small deformation with high enough resolution are to (i) increase
magnification and resolution of SEM images, (ii) lengthen sample length, and (iii) reduce stiffness of the force sensing beams. Further discussion on modification of sample length and stiffness of the force sensing beams for the resolution enhancement can be found in [47].

5.3 Conclusion

We present the SiC based MEMS stage to test micro/nanoscale materials under uniaxial tension at room to elevated temperatures in situ in SEM. The stage serves as the heater, and has built in force and temperature sensor, in addition to self aligning mechanisms. The stage is operated by a piezoactuator. A macor substrate separates the two for thermal isolation. In addition, a water cooling system prevents the actuator from overheating. First, we characterized the stage theoretically where we found that Joule heating of the stage induces uniform temperature of the sample (within 3% variation in the temperature) and sample temperature is insensitive to sample resistance. Experimentally, we fabricated the SiC stage by laser and focused ion milling, and a single crystal silicon sample with a built-in sensor. Both the temperature sensors, on the stage and the on the sample, were precalibrated independently. Uniaxial tensile tests were carried out on the Si sample at room to 403°C in SEM. We recovered the known elastic modulus of the SCS sample at room temperature within 1% error and reduced moduli at elevated temperatures up to 403°C. The moduli values match well with those published in the literature. Our theoretical and experimental demonstrations show that the SiC allows to test micro/nanomaterials in analytical chambers with temperature variation/measurement.
5.4 Figures

Figure 5.1: Three dimensional schematics of the SiC stage: (a) overall view of the SiC stage (b) experimental setup for in situ uniaxial tests, (c)-(d) are enlargement of C in (b) which show a sample before and after assembly with the stage, respectively. In (a), zoom-in view of a bi-metal type temperature sensor is shown. In (b), the stage is thermally isolated from an SEM holder sample holder by a macor heat insulator. The metal pillars are connected to copper wires so that electrical potential can directly be applied to the stage for resistive heating.
Figure 5.2: (a) The schematic and theoretical prediction of the SiC-Pt temperature sensor with temperature. (b1) the 3-dimensional schematic of bimorph sensors in series for the resolution enhancement, (b2) an FEA result for mechanical displacement of the sensor at 700°C, and (b) deformation-temperature at A, B, C, and D. In (b2), red, yellow, green, and blue indicate small to large displacement of the sensor, respectively.
Figure 5.3: (a) The schematic of the SiC stage for electrical connection through the stage and (b) corresponding lumped model. Electrical power cross (c) U-beams, (d) the frames of the SiC stage, and (e) the sample. In (f), the electrical power at the sample as a function of $R_{DE}$ is shown. The indicated data point in (f) corresponds to the maximum electrical power in (e).
Figure 5.4: A finite element analysis for the SiC stage with metal pillars and a macor frame: (a) electrical (b) thermal, and (c) mechanical responses of the system at 50 V.
Figure 5.5: FEA analysis (a) Variation of temperature across the sample length at 50V (between A and B) and (b) temperature at the sensor at C and at the ends (at A and B) of the sample as a function of input voltage (V).
Figure 5.6: Fabrication flow of a SiC stage for high temperature in situ uniaxial tests. From a 256µm-thick SiC wafer in (a), trench and through holes are created by using laser milling from the back side of the wafer to reduce height of a temperature sensing beam and to create alignment marks for the front side fabrication (see (b) and (f)). In (c), all inner structures, such as supporting beams, force sensing beams, and U-beams, of the SiC are fabricated by laser milling from the front side of the wafer. In order to align the backside trench with the temperature sensing beam on the front side, the alignment marks in (f) are used. During laser milling, SiO$_2$ deposition occurs (see (c) and (g)) which is cleaned by HF wet etching as shown in (d) and (h). The grooves of grips for sample gripping are etched and Pt is deposited to make a bimetal temperature sensor in (e) by using focused ion beam (FIB) etching and deposition, respectively. Finally, the outer frame of the SiC stage is cut by laser milling.
Figure 5.7: The images of the SiC stage, SCS sample, and linear stage: SEM images of (a) overall view of the SiC stage, (b) the SCS sample and (c) zoom-in view of a built-in bimaterial sensor with Pt-Si junctions, and (d) the image of the SiC stage on a linear stage where I, II, III, and IV designate the SiC stage, piezoactuator, water cooling block, and macor frames, respectively.
Figure 5.8: In situ calibration of a temperature sensor: (a) crucible temperature with increase in ESEM heater temperature and (b) displacement of a bimaterial sensor on the SiC stage as a function of temperature. We melt a piece of tin (b1) near one of two grips and glass (b2) near U-beams.
Figure 5.9: Uniaxiality of loading during in situ mechanical testing.
Figure 5.10: Stress-strain responses of the SCS sample at different temperatures (room temperature to 403°C).
Figure 5.11: (a) Temperature at the piezoactuator with and without water cooling as a function of temperature at the SiC stage and (b) temperature at the stage (circle) and sample (square) with applied voltage.
Chapter 6

Size and temperature dependent BDT behavior in SCS

Size dependent material properties of brittle semiconductive materials such as Si and SiC become important as they are the most commonly used materials for micro/nano scale devices. In many cases, the small scale systems are required to operate even at high temperature as outlined in Chapter 1. Hence accurate characterization of thermo-mechanical properties and understanding of fundamental mechanism for material deformation are essential since plastic deformation can lead to substantial change in electrical and mechanical responses of materials.

One of common thermo-mechanical behaviors in materials except FCC metals is the brittle-to-ductile transition (BDT). This thermally and mechanically coupled phenomenon is conventionally defined by the rapid change in the fracture behavior from brittle to ductile failure [83] due to large increase in dislocation density with temperature [84]. The transition temperature is determined by temperature dependence of dislocation mobility in different materials [84]. Also it is well known that the range of temperature over which transition occurs depends on initial dislocation density. For example, steel with high bulk dislocation density shows gradual transition over tens of degrees Celsius below room temperature [83] while transition occurs within a few degrees Celsius for single crystal silicon at 550°C [52]. Although Charpy test was proposed to study BDT behavior in metals, a bulk silicon sample with a predefined notch has been generally tested using a bending method at a much lower strain rate than Charpy test due to its intrinsically limited plasticity [52].

Several experimental and computational studies suggest BDT temperature reduction with sample size as discussed in Chapter 1. However, size dependent BDT is not conclusive
in the literature as there are controversial results as well. One of the main reasons for limited experimental data and controversies in the literature is due to the lack of a robust and rigorous in situ method for thermo-mechanical measurement. For unambiguous experimental investigation of size dependent BDT behavior in SCS, it is essential to control not only sample size, but also sample temperature as BDT is intrinsically sensitive to temperature. Here we address those issues using the novel thermo-mechanical testing method (refer Chapter 5) with simultaneous control of two key parameters, sample size and temperature.

In the present chapter, we experimentally study thermo-mechanical behavior of single crystal silicon at the micro/nano scale with variation of sample size and temperature. We hypothesize reduction of BDT temperature due to strong effect of surface with decrease in sample size. In order to validate our hypothesis, we carry out in situ thermo-mechanical experiment and observe significant reduction in brittle-to-ductile transition temperature.

### 6.1 Experimental methods

In situ material testing offers an attractive feature in studying of micro/nano materials as it provides direct structure-property relationship due to ultra high resolution of Electron Microscopy observations [31, 32, 34, 35]. However, in situ thermo-mechanical characterization of micro/nanoscale samples involves several challenges as we considered in the previous chapter. In order to overcome those challenges and to carry out in situ thermo-mechanical testing of single crystal silicon at micro and nano scale, we use a novel SiC based MEMS apparatus which allows to test the independently fabricated micro/nanoscale samples with concurrent control of sample size and temperature. Refer Chapter 5 for further details.

### 6.1.1 A microdevice for thermo-mechanical measurement

A novel SiC MEMS stage for in situ thermo-mechanical testing is shown in Figure 6.1(a). The overall structure of the SiC stage and the procedure of in situ thermo-mechanical mea-
Although the stage offers a uniaxial testing mode as in Chapter 5, we utilize bending test to study size and temperature dependent plasticity in SCS samples. Bending limits the high stress region to a small volume, thus minimizing the likelihood of fracture due to surface flaws. Thus, bending allows to increase sample stress to explore the possible onset of plasticity. For example, consider a simple cantilever beam loaded by a load $F$ as shown in Fig. 6.1(e). For simplicity, we assume the cantilever beam is linear elastic, isotropic, and homogeneous, and material deformation is small. It is easy to show that the stress state due to $F$ is $\sigma(x, y) = F(L - x)y/I$ in the cantilever beam[85] where $x$ and $y$ are coordinates of the beam in longitudinal and transverse directions, $L$ and $H$ are length and height of the beam, respectively, and $I$ is a moment of inertia of the given cross section (see Fig. 6.1(e)). In order to study the onset of plasticity at $\sigma_{\text{yield}}$, we may apply $F$ during bending test such that a material sample yields in a small volume in the vicinity of $x = 0$ and $y = \pm H/2$, while bending stress elsewhere is smaller than $\sigma_{\text{yield}}$. On the other hand, with uniaxial loading, the entire gauge length of the sample is subjected to $\sigma_{\text{yield}}$ to induce material yield. As a result, the likelihood of brittle fracture without any plastic deformation during uniaxial test near BDT temperature can be much higher as we experimentally observed.

### 6.1.2 SCS bending samples

The SCS bending sample shown in Fig. 6.1(b) is fabricated by lithography based microfabrication technology. For further reduction of sample size down to submicron size and minimization of surface roughness with high precision, we use focused ion beam (FIB) milling followed by Freon (CF$_4$) reactive ion etching (RIE) with etching depth $>100$nm. This additional RIE etching is to eliminate effect of gallium ion bombardment on material deformation of SCS as the implantation depth is 40-56nm for silicon [86]. High precision microfabrication by FIB also ensures the identical dimension of the eight bending arms for symmetric bending deformation of the sample. The crystal orientation of all bending arms
along the longitudinal direction is $<110>$ with the elastic modulus 169GPa [47].

It is worth noting that although AFM based bending test is frequently used to characterize small scale materials [18, 19] due to its simplicity, there may be stick-slip between an AFM tip and bending arm during loading of a sample which makes data interpretation difficult. On the other hand, all bending arms in Fig. 6.1(c) have well defined boundary conditions. Hence, the stress state of the bending sample can be evaluated from a force-displacement relation. For example, we find a good agreement between experimental and predicted force displacement relation at room temperature. The difference in the slope of the $f - \delta$ in Fig. 6.1(d) is within 1% for $h = 1.5\mu m$. Further details on the analytical $f - \delta$ relation can be found in Chapter 7.

### 6.2 Experimental Results and Discussions

Here we experimentally explore size and temperature dependent BDT behavior by testing the SCS bending widths varying from 720nm to 8.7µm. For each temperature, we used Joule heating of the SiC stage and maintained the temperature for 20 minutes before any mechanical loading of the sample was applied. All experiments were carried out in vacuum environment ($<10^{-6}$torr) to eliminate the effect of oxidation on the plastic deformation of SCS at high temperature.

#### 6.2.1 Thermomechanical response of SCS bending samples

The experimental results for $h = 720nm$, $= 1.5\mu m$, and $= 8.7\mu m$ are shown in Fig. 6.2. In order to evaluate the stress state of the sample during in situ experiment, we obtained the theoretical $f - \delta$ relation (the dotted lines) and the corresponding maximum bending stress ($\sigma_{\text{max}}$) in the sample using linear elastic finite element analysis (Fig. 6.2). Note that the slope of the $f - \delta$ decreases with temperature due to reduction of elastic modulus of SCS with temperature [18, 60, 61, 69, 82].
Experimental $f - \delta$ response for $h = 1.5\mu m$ (Fig. 6.2(a)) shows linear elastic behavior at $25^\circ C$, $184^\circ C$, and $293^\circ C$. First yielding occurs at $340^\circ C$ with $3.1\mu m$ plastic displacement after removal of any mechanical loading on the sample. In order to experimentally confirm the BDT behavior (sudden change in characteristic of material failure from brittle to ductile with temperature), we tested another sample with $h = 1.5\mu m$ again at $293^\circ C$, when the sample failed in fracture without any plastic deformation. Thus, for $h=1.5$ um, BDT occurs at a temperature between $293^\circ C$ and $340^\circ C$.

To study the role of sample size on BDT, we used a smaller sample with $h = 720nm$. From the $f - \delta$ response in Fig 6.2(b), we observed linear elastic behavior at $25^\circ C$ and $184^\circ C$. At $293^\circ C$ the sample deformed plastically with $1.8\mu m$ permanent deformation after unload. Thus, BDT temperature decreased with sample size. At higher temperature ($340^\circ C$), 720nm sample showed plastic deformation as expected ($2.4\mu m$ permanent deformation after the loading/unloading cycle). When sample size is larger, i.e., $h = 8.7\mu m$, $f - \delta$ response shows linear elastic behavior at $184^\circ C$, $293^\circ C$, and $340^\circ C$ during loading and unloading of the bending sample. At $375^\circ C$, we measured $250nm$ and $1.7\mu m$ permanent deformation after the first and second loading/unloading cycles, respectively, i.e., BDT temperature is between $340^\circ C$ and $375^\circ C$. Thus, BDT temperature increases for larger sample size. Compared to bulk ($823K$ [52]), the temperature reduces by 21%, 25.5% and 31% for samples with $h = 8.7, 1.5$ and $0.72\mu m$ respectively.

Figure 6.3 shows the SEM images of plastic deformation in the SCS bending samples. In Fig. 6.3(a), the initial configuration of the bending sample before any mechanical loading is shown (the zoom-in view of Area A is shown in Fig. 6.3(b)). Figure 6.3(c) shows significant change in the sample configuration due to plastic deformation after complete removal of any mechanical loading. Figure 6.3(d) shows the zoom-in view of Area B in Fig. 6.3(b) after ductile fracture of the SCS sample. Note that the permanent curvature with respect to the reference lines in Fig. 6.3(b) and (d) clearly indicates substantial plastic deformation of the bending arms.
6.2.2 Mechanism for size and temperature dependent BDT

Our in situ experimental study indicates that brittle-to-ductile transition of single crystal silicon depends on sample size and temperature as the BDT temperature decreases with sample size. Here we propose a mechanism for the observed size and temperature dependent BDT in SCS. The proposed mechanism emphasizes the onset of plasticity that is controlled by surface dislocation nucleation due to (i) large surface-to-volume ratio, (ii) reduced dislocation nucleation energy at the surface, and (iii) low bulk dislocation density.

With reduction of sample size, surface effects become increasingly important as the surface-to-volume ratio is inversely proportional to the characteristic length scale of the sample size. Hence for small samples, dislocations are strongly influenced by nearby surfaces and interfaces. Dislocations move towards free surfaces and escape to minimize the stress and strain energy of the crystal, i.e., they are subjected to attractive image forces from the surface. The image force is inversely proportional to the distance from the free surface [87]. Hence self-energy or the strain energy of the crystal induced by a single dislocation near free surfaces [88] can be substantially lower than the energy due to a dislocation in the bulk. Thus introduction of a dislocation to a small sample is likely to be less energy expensive than that in a large crystal. For example, Shuch et al experimentally found that using nanoindentation the energy to nucleate a surface dislocation in single crystal platinum is 0.28eV, whereas the corresponding bulk value is 1.3eV [89]. Several MD simulation results of small scale SCS samples show that the surface serves as a source of dislocations in small samples[55–57]. Kang and Cai predicted that SCS can plastically deform at room temperature by dislocation initiated from surface[55] when sample size is less than 4nm.

In addition, the number of preexisting defects including dislocations in a sample is proportional to the volume of the sample, and thus rapidly reduces with sample size. For semiconductive materials like SCS with extremely low defect density, nano or even micro samples can have only a few or even no preexisting dislocations [87]. Hence the onset of
plasticity is likely controlled by surface dislocation nucleation rather than dislocation multiplication from preexisting dislocations.

Consider a material sample with perfect crystal structure. The sample is in thermodynamic equilibrium and the probability density of an atom at a specific position in the crystal structure is given by Boltzmann distribution at temperature $T$ [90]. With increase in thermal energy in the crystal, thermal vibration of the atom becomes larger and hence the probability of having an atom with higher energy state increases in the system. When the thermal energy is sufficiently large, there is the possibility of having an atom jump out of the lattice and nucleate a dislocation. Let the activation energy of dislocation nucleation be $U$. Then the rate at which dislocation nucleates per unit volume at absolute temperature $T$ is given by $\dot{n} = f \exp(-U/kT)$, where $k$ is the Boltzmann constant and $f$ is a constant [90]. Thus, for SCS, one expects that as temperature approaches a critical temperature, there will be an abundance of dislocations which will facilitate plasticity. Below this temperature, not only dislocation nucleation rate is low, but also dislocation glide of existing dislocations is limited due to high Peierls stress. BDT is believed to be due to an abundance of dislocation at a critical temperature, and a reduction of Peierls stress against dislocation motion. An external stress on the material facilitates the nucleation of dislocation, i.e., reduces the energy barrier for nucleation. Let $U - F^*$ be the remaining barrier due to a given applied stress on the sample. Then the nucleation rate changes to $\dot{n} = f \exp(-(U - F^*)/kT)$.

In order to explore the size effect on BDT temperature, consider surface and volume dislocation nucleation in a cylindrical single crystal silicon sample shown in Fig. 6.4(a) at a given temperature $T$. The length and diameter of the sample are $l$ and $d (= 2R)$, respectively, and $r$ is a radial coordinate of the sample. Suppose the surface effects become fully dominant within small interval $R > r > R - \alpha a$ where $a$ is the length of the crystalline unit cell and $\alpha$ is a constant representing a number of unit cells. Since dislocation nucleation from surface requires less energy as discussed earlier, we introduce the dislocation nucleation rate from the surface with activation energy $U_S$. Hence dislocation nucleation rate in volume ($\dot{n}_V$) and
at surface ($\dot{n}_S$) can be written as follows

$$
\begin{align*}
\dot{n}_V &= f_V \exp\left(-\frac{U_V}{kT}\right) \left[s^{-1}m^{-3}\right], R - \alpha a > r > 0, \\
\dot{n}_S &= f_S \exp\left(-\frac{U_S}{kT}\right) \left[s^{-1}m^{-3}\right], R > r > R - \alpha a
\end{align*}
$$

(6.1)

where $f_V$ and $f_S$ are constants, and $U_V > U_S$ are activation energies from volume ($V$) and surface ($S$), respectively. The total dislocation nucleation rate in a given volume $V_T = \pi R^2 l$ becomes $\dot{N}_{TOT} = V_V \dot{n}_V + V_S \dot{n}_S$ where $V_V = (R - \alpha a)^2 \pi l$ and $V_S = V_T - V_V$ (see Fig. 6.4(a)). The corresponding dislocation nucleation rate per volume ($= \dot{N}_{TOT}/V_T$) is

$$
\dot{n}_{TOT} = \left(1 - \frac{\alpha a}{R}\right)^2 \dot{n}_V + \left(2 \frac{\alpha a}{R} - \left(\frac{\alpha a}{R}\right)^2\right) \dot{n}_S.
$$

(6.2)

For bulk materials ($R \gg \alpha a$), Eq. 6.2 gives $\dot{n}_{TOT} \to \dot{n}_V$ due to weak surface effects while $\dot{n}_{TOT}$ converges to $\dot{n}_S$ with $R \to \alpha a$ as the surface effects become dominant.

Let $\dot{n}_{BDT}$ be the nucleation rate at BDT in bulk SCS at $T_{BDT}$. If $\dot{n}_{BDT}$ is the necessary and sufficient condition for BDT at all size scales, then one expects that BDT temperature decreases with size. The qualitative nature of this decrease can be revealed by equating $\dot{n}_{TOT}$ at $T$ in Eq. 6.2 to $\dot{n}_{BDT}$ for bulk, i.e., equating $f_V \exp(-U_V/kT_{BDT})$ to the right side of Eq. 6.2. This gives

$$
\frac{1}{\exp(-\beta)} \left((1 - a^*)^2 \exp(-\beta/T^*) + (2 - a^*) a^* f^* \exp(-U^* \beta/T^*)\right) = 1
$$

(6.3)

where $a^* = \alpha a / R$, $f^* = f_S / f_V$, $U^* = U_S / U_V$, $T^* = T/T_{BDT}$, and $\beta = U_V / kT_{BDT}$. Using Eq. 6.3 and a well known value for BDT in bulk scale SCS ($\beta = 23.71$ [52]), BDT trend is shown in Fig. 6.4(b)-(c) as a function of sample size. As experimentally observed in Fig. 6.2, BDT temperature gradually decreases with sample size due to stronger surface effect. Our proposed model captures the qualitative behavior of the observed size and temperature dependent BDT as shown Fig. 6.4. At this point, further characterization of $U_S$ and $f_S$ is required for quantitative prediction of size dependent BDT behavior for SCS.
6.3 Conclusion

In this chapter we carried out in situ thermo-mechanical tests, which allow quantitative in situ measurement with concurrent control of sample size and temperature for silicon crystal silicon. Our experimental investigation revealed that the onset of plasticity in single crystal silicon depends on not only temperature, but also sample size as we observed up to 31.2% reduction in the BDT temperature with respect to bulk silicon. This size dependent BDT behavior was explained with plasticity that is controlled by surface dislocation nucleation. It is worth noting that the yield strength of the SCS samples in Fig. 6.2(a)-(c) seems size dependent as the maximum bending stress increases with decrease in sample size. In the following chapter we will consider the size dependent yield behavior of SCS in detail.
6.4 Figures

Figure 6.1: Experimental method for in situ thermo-mechanical test: (a) an SEM image of the MEMS stage for in situ test, (b) zoom-in view (Area C) of the silicon bending sample on the MEMS stage, (c) the schematic of the bending sample, (d) a force($f$)-displacement($\delta$) relation, (e) a schematic of a cantilever beam loaded by $F$, and (f1)-(f2) zoom-in view of area A in (b). During in situ test, the sample displacement($\delta$) and applied force($f$) are measured by analyzing high resolution SEM images as in (d). Displacement measurement is done without exposing the bending arms to electron beams during in situ test by measuring $\delta$ away from the bending arms (Area A in (b)).
Figure 6.2: Force-displacement responses of single crystal silicon samples with variation of sample size and temperature: (a) $h = 8.7\,\mu m$, (b) $h = 1.5\,\mu m$, and (c) $h = 0.72\,\mu m$. As shown in (d) for all sample sizes, the plasticity is observed at lower temperature with reduction of sample size.
Figure 6.3: The SEM images of the SCS bending sample: (a) initial configuration without any mechanical loading, (b) zoom-in view of Area A, (c) plastic deformation of the bending arm after complete unloading of the sample, and (d) zoom-in view of Area B after material failure of the sample. Note that permanent curvature after failure in (d) indicates considerable plastic deformation with respect to the reference lines in (b) and (d), respectively.
Figure 6.4: Mechanism for size and temperature dependent brittle-to-ductile transition (BDT) in silicon. (a) the schematic of the dislocation nucleation from the sources in the volume and at the surface. (b)-(c) theoretical predictions of the temperature for BDT in the sample as a function of the radius of the cylinder and the ratios of activation energies for dislocation nucleation from surface and bulk, and the ratio of the constants $f_S/f_V$. 
Chapter 7

Stress Gradient Plasticity in SCS

In Chapter 6, we considered size and temperature dependent plasticity in SCS. Using the in situ thermo-mechanical testing method introduced in Chapter 5, we unambiguously showed BDT temperature reduction with sample size. Also in situ experimental results in Chapter 6 revealed size dependent yield strength in SCS. In order to interpret the observed size dependent BDT behavior, we proposed the mechanism by the surface dominant dislocation nucleation due to the stronger surface effects with reduction of sample size. However, size dependent flow strength in SCS incorporated with its unique characteristics due to covalent atomic bonds and sample size dependent stress gradient during bending test has not been considered.

In this chapter, we explore the size dependent yield strength for single crystal silicon. For theoretical study, we employ a continuum model to explore the effect of sample size and stress gradient on the onset of plasticity. Using the model, we show that stress concentration due to the dislocation pile-up is strongly influenced by stress gradient within the sample. Also, the model predicts size dependent behavior occurs even at larger sample size for single crystal silicon due to large Peierls stress. To measure yield strength of SCS bending samples, we use an elastic-plastic force-displacement relation. The analysis on the yield strength of SCS bending samples validates that yield behavior of SCS depends on sample size as predicted by our proposed model.
7.1 Effect of Stress Gradient and Size on Plasticity in SCS

In order to study the role of sample size on yield behavior of single crystal silicon (SCS), we utilize an isotropic elastic continuum model. With the model, we explore interaction between dislocations under influence of stress gradient due to bending and effect of their interaction on the SCS strength at micro/nano scale.

7.1.1 Stress gradient plasticity in SCS

During the following analysis, we only consider edge dislocations for the simplicity. Furthermore, we assume that the onset of plasticity is associated with dislocation nucleation rather than dislocation multiplication among preexisting dislocations due to the following reasons. First, the number \(N_{\text{pre}}\) of preexisting dislocations for a given dislocation density is proportional to material volume \(r^3\) where \(r\) is the characteristic length of the sample and hence \(N_{\text{pre}}\) rapidly decreases with \(r\). Second, SCS has extremely low dislocation density compared to metals\[87\]. Thus, a SCS sample may have only a few or no dislocations at nanoscale or even at microscale. Third, dislocation escape\[9\] and starvation\[8\] have been observed in small scale samples due to strong surface effects which may prevent plasticity by dislocation multiplication even with the considerable number of preexisting dislocations. To study the influence of stress gradient on plasticity in SCS, we will apply pure bending moment \(M\) to a SCS sample.

In addition, we assume that dislocation nucleates from surface, not in volume, at the onset of plasticity because (i) the maximum stress occurs at the surface due to stress gradient in a sample and (ii) surface dislocation nucleation activation energy seems lower than bulk dislocation nucleation activation energy as suggested by several experimental and computational studies for SCS and metals\[57, 89, 91\] (see Chapter 6 for further details). Note that even without the second argument, dislocation nucleation during the initial stage of material...
yield will occur at the surface as the maximum stress always appears at the surface in pure bending. We will discuss this issue later.

Now consider a SCS sample with crystal orientation $<110>$ as shown in Fig. 7.1(a). The schematic in Fig. 7.1(a) shows the surface dislocation nucleation due to the pure bending moment $M$ where $d$ and $L$ are the thickness and the total length of the sample, respectively, and $l$ is the length of slip planes along slip directions for SCS (SCS has $<111>$ slip planes). For the SCS bending sample with the given crystalline orientation and bending direction in Fig. 7.1, it can be shown that slip occurs either on $(111)<0\bar{1}1>$, $(111)<\bar{1}01>$, $(1\bar{1}1)<101>$ or $(1\bar{1}1)<011>$ where they have the same Schmid Factor (1/$\sqrt{6}$). Possible slip directions for the SCS sample are represented by different slip directions in Fig. 7.1(a). In our analysis we consider only one slip direction as we assume there is no interaction between slip planes due to the following reason. We assume that the width $w$ (out of plane direction) and length $L$ of the sample are much larger than $d$. The zoom-in view of Area C in Fig. 7.1(a) is shown in Fig. 7.1(b) where $s$ is a coordinate along the slip direction from surface dislocation nucleation source at A, and $\Delta L$ is a mean average distance between activated slip planes. As the main focus of the present chapter is on the onset of plasticity where there may be only a few activated slip systems, we assume $L \gg \Delta L$ such that there is no interaction between slip planes in the SCS sample with $L \gg d$.

With the pure bending moment $M$, stress state at any cross section of the sample (D-E in Fig. 7.1(b) and (c)) is given by [85]

$$\sigma(\kappa) = \frac{M\kappa}{I}$$

where $\kappa$ is the vertical coordinate of the sample from its neutral axis and $I$ is a moment of inertia for the sample cross section (see Chapter 2 for details). Now, the resolved shear
stress due to $M$ along the slip directions (A-B in Fig. 7.1(b)) is

$$
\tau_{res}(\kappa) = \pm \frac{1}{\sqrt{6}} \sigma(\kappa) = \pm \frac{1}{\sqrt{6}} \frac{M\kappa}{I}
$$

(7.1)

using the Schmid factor[92]. Note that we have the maximum shear stress at $\kappa = \pm d/2$ from Eq. 7.1 and hence material yield occurs at the surface as mentioned earlier.

Consider the SCS sample that is under the shear stress $\tau_{res}(s)$ as shown in Fig. 7.1(d) along the slip direction A-B in $s$ coordinate where $\tau_s$ is the magnitude of the maximum shear stress at the surface due to bending moment $M$ ($\tau_{res}(s) = \tau_s$ at $s = 0$). $\tau_{res}(s)$ can be obtained using Eq. 7.1 for a given $M$. Let $\tau_N$ be a resolved shear stress along the slip direction A-B which corresponds to the onset of surface dislocation nucleation without effect of stress gradient, i.e., surface dislocation nucleation under uniaxial stress. For sufficiently large $M$ such that $\tau_s > \tau_N$, surface dislocation nucleation may occur from Source A. Since we assumed that the sample has perfect crystalline structure and there is no interaction between dislocations on different slip planes, dislocation movement is hindered only by Peierls stress ($\tau_{peierls}$) in the following analysis.

Suppose that the leading dislocation moves away from Source A by distance $-l_{cr}/2$ and remains stationary due to the interaction between the dislocations in the pile-up and Peierls stress at equilibrium (see Fig. 7.1(d)). The stress ($\tau_{Lead}$) at the leading dislocation (at $s = -l_{cr}/2$) is magnified by the dislocation pile-up within $0 > s > -l_{cr}/2$. Hence at $s = -l_{cr}/2$ (or at $\kappa = \kappa_{cr}$ in $\kappa$ coordinate) the stress at the leading dislocation is balanced with Peierls stress, i.e., $\tau_{peierls} = \tau_{Lead}$, where the local shear stress ($\tau_{res}(s) = \tau_{cr}$ at $s = -l_{cr}/2$) can be substantially smaller than $\tau_{peierls}$ as schematically shown in Fig. 7.1(d). Note that surface dislocation nucleation process is intrinsically under strong influence of surface, for example, mirror force on an edge dislocation is inversely proportional to the distance between the dislocation and the surface. In order to take such strong influence of the surface into account during the surface dislocation nucleation, we introduce mirror
dislocations within \( l_{cr}/2 > s > 0 \) as shown in Fig. 7.1(d) where the burgers vector has an opposite sign of those dislocations in \( 0 > s > -l_{cr}/2 \).

Note that the role of Peierls stress becomes increasingly relevant in plastic deformation of SCS under influence of stress gradient. For example, FCC metals have low Peierls stress in the range of \( 10^{-6}G^{-1}10^{-5}G \) while the theoretical strength of the FCC metals is of order \( 10^{-1}G \) where \( G \) is a shear modulus[87]. On the other hand, single crystal silicon has large Peierls stress due to strong covalent bond, e.g., Kang and Cai predicted \( \tau_{th} = 5.2\text{GPa} \) (theoretical resolved stress) and \( \tau_{peierls} = 2.23\text{GPa} \) (Peierls stress) using computational simulation[55]. Hence, unlike FCC metals, large Peierls stress in SCS is expected to behave as major obstacles for the dislocation motion during nucleation dominant plasticity. In the following section we will consider the stress at the leading dislocation with the influence of the dislocation pile-up and free surface using a continuum model in great details.

### 7.1.2 Dislocation density with stress gradient

Here an isotropic elastic continuum model is used to study the equilibrium dislocation pileup density under an applied shear stress[93, 94]. Consider a SCS sample shown in Fig. 7.1 where the identical edge dislocations are on the same slip plane A-B. To find the equilibrium state of dislocations within the plane, we will consider the net force balance due to an applied external stress and interaction of the dislocations. A dislocation under an applied stress field \( \tau_{res}(s) \) experiences a force that is the product of its Burgers vector \( (b) \) and an appropriate stress component, i.e., \( \tau_{res}(s)b \) for edge dislocation at \( s[93] \). If we introduce another identical dislocation to the slip plane at \( s' \), it can be shown that the repulsive interaction force between two dislocations is \( f_{\text{int}} = -Gb/2\pi(1-\nu)(s-s') \) where \( G \) is shear modulus and \( \nu \) is Poisson’s ratio [87]. At the equilibrium, the force acting on a dislocation at \( s \) is balanced with the interaction forces with all neighboring dislocations where many dislocations can be homogenized with a continuous dislocation density function \( n(s) \) as shown by Bilby and Eshelby[93]. Hence the relation between a given shear stress \( \tau_{res}(s) \) and the corresponding
continuous dislocation density \( n(s) \) (see Fig. 7.1(c)) at the equilibrium is given by

\[
\tau_{\text{res}}(s)b + \frac{Gb^2}{2\pi(1-\nu)} \int_{-l_{cr}/2}^{l_{cr}/2} \frac{n(s')}{s-s'} ds' = 0
\]  

(7.2)

where \( s \) is a coordinate along the slip direction and \( s' \) is a dummy variable. Following Hirth[91], we introduce \( \eta = s/(l_{cr}/2) \) and \( f(\eta) = n(l_{cr}\eta/2) \) and then Eq. 7.2 can be simplified as

\[
\frac{2(1-\nu)}{Gb} \tau_{\text{res}}(\eta) + \frac{1}{\pi} \int_{-1}^{1} \frac{f(\eta')}{\eta - \eta'} d\eta' = 0
\]  

(7.3)

where \( 1 > \eta > -1 \).

The given shear stress within \( 0 > s > -l_{cr}/2 \) can be written as \( \tau_{\text{res}}(s) = (\tau_s - \tau_{cr})s/(l_{cr}/2) + \tau_s \) to satisfy \( \tau_{\text{res}}(0) = \tau_s \) and \( \tau_{\text{res}}(-l_{cr}/2) = \tau_{cr} \). Note that \( \tau_{\text{res}}(s) = 0 \) at \( s = -l/2 \) and hence we have \( \tau_{cr}/\tau_s = 1 - l_{cr}/l \). As our focus is on the onset of plasticity, i.e., \( l \gg l_{cr} \), we assume that stress field \( (\tau_{\text{res}}(s)) \) due to bending moment is linear even after dislocation nucleations. Now to take the effect of free surface into account, we introduce the mirror stress field about the free surface (see Fig. 7.1(d)) and thus the resolved shear stress within \( l_{cr}/2 > s > -l_{cr}/2 \) becomes \( \tau_{\text{res}}(s) = \tau_{cr} + (\tau_s - \tau_{cr})(1 - |s/(l_{cr}/2)|) \) or using \( \eta = s/(l_{cr}/2) \)

\[
\tau_{\text{res}}(\eta) = \tau_{cr} + (\tau_s - \tau_{cr})g(\eta)
\]  

(7.4)

where \( g(\eta) = 1 - |\eta| \). The shear stress gradient is \( d\tau_{\text{res}}(\eta)/d\eta = \pm(\tau_s - \tau_{cr}) \) from Eq. 7.4. Note that \( g(\eta) = 1 - |\eta| \) is an even function and hence Eq. 7.4 can be written as

\[
\tau_{\text{res}}(\eta) = \tau_{cr} + (\tau_s - \tau_{cr}) \sum_{i=0}^{k} a_i \eta^i
\]  

(7.5)

where \( i = 0, 2, 4, .., k \to \infty \), and \( a_i \) are coefficients. For arbitrary polynomials such as Eq. 7.5, it has been shown that the solution for \( f(\eta) \) in Eq. 7.3 is given by [93]

\[
f(\eta) = \frac{2(1-\nu)}{\pi Gb^2} \int_{-1}^{1} \left( \frac{1-\eta'^2}{1-\eta^2} \right)^{1/2} \tau_{\text{res}}(\eta') \frac{d\eta'}{\eta - \eta'}
\]  

(7.6)
Although Eq. 7.6 can be solved for Eq. 7.5 with arbitrarily large $k$ (for example Fig. 7.2(a)-(b) shows the cases for $k = 100$ using a numerical method), we approximate $g(\eta)$ by a second order polynomial, i.e., $g(\eta) \approx a_0 \eta^0 + a_2 \eta^2$, where $1 > \eta > -1$. Hence Eq. 7.5 becomes

$$
\tau_{res}(\eta) \approx \tau_{cr} + (\tau_s - \tau_{cr})(a_0 \eta^0 + a_2 \eta^2).
$$

We will show later that the quadratic polynomial is a good approximation for the given shear stress $\tau_{res}(\eta)$ in Eq. 7.4. Equation 7.3 can be solved for the given stress field $\tau_{res}(\eta)$ in Eq. 7.7 by using Tchebyscheff polynomials $[91]$ and the corresponding dislocation density $n(s)$ is

$$
n(s) = \frac{2(1-\nu)}{Gb\sqrt{1-(\frac{s}{l_{cr}/2})^2}} \left[ Ta_2 \left( \frac{s}{l_{cr}/2} \right)^3 + \left( \tau_{cr} + Ta_0 - \frac{1}{2} Ta_2 \right) \left( \frac{s}{l_{cr}/2} \right) \right]
$$

where $T = \tau_s - \tau_{cr}$.

Figure 7.2 shows the dislocation density within $1 > s/(l_{cr}/2) = \eta > -1$ using Eq. 7.8 for $\tau_{cr}/\tau_s = 0$ and $= 1$ where $a_0 = 0.81159$ and $a_2 = -0.93291$ are obtained by the least squares fitting of $g(\eta) = 1 - |\eta|$ by a polynomial function $a_0 \eta^0 + a_2 \eta^2$. Note that dislocation density $n(\eta)$ is negative in $0 > \eta > -1$, but positive in $1 > \eta > 0$ due to the opposite sign of burgers vector. We find a good agreement between the cases of $k = 2$ and $k = 100$ in Fig. 7.2(a)-(b) and thus from now on, Eq. 7.8 will be used to explore the role of dislocation interaction in the following analysis.

### 7.1.3 Effect of stress gradient on stress concentration of dislocation pile-up

To explore size dependent plasticity in SCS, we consider stress at the leading dislocation with variation of nondimensionalized shear stress gradient $(d\tau_{res}(\eta)/d\eta = \pm(\tau_s - \tau_{cr}))$ and nondimensionalized sample size $h^* = h/b$. Bilby and Eshelby[93] showed that the stress at
the head of dislocation pile-up \((s = -l_{cr}/2)\) can be obtained by

\[
\tau_{Lead} = -\frac{1}{2} \frac{Gb\pi}{2(1-\nu)} \lim_{s \to -l_{cr}/2} (s + l_{cr}/2)n^2(s)
\]  

(7.9)

for a given dislocation density \(n(s)\). From Eq. 7.8 and Eq. 7.9, the stress at the leading dislocation \((s = -l_{cr}/2)\) becomes

\[
\tau_{Lead} = \frac{\pi(1-\nu)\tau^2_{peierls} l_{cr}}{16Gb} (\alpha + \beta \tau_{cr}/\tau_s)^2
\]  

(7.10)

where \(\alpha = 2a_0 + a_2\) and \(\beta = 2 - 2a_0 - a_2\). Figure 7.3(a) shows nondimensional shear stress at the leading dislocation \((\tau^*_{Lead} = \tau_{Lead}/Q)\) due to dislocation pile up with variation of stress gradient \((1 \geq \tau_{cr}/\tau_s \geq 0)\) where \(Q = (1-\nu)\tau^2_{cr} l_{cr}/Gb\). The result indicates that for larger stress gradient, stress concentration becomes smaller at the leading dislocation. For example, \(\tau^*_{Lead} = 0.7854\) for uniform stress \((\tau_{cr}/\tau_s = 1)\) while \(\tau^*_{Lead} = 0.09356\) for the maximum gradient \((\tau_{cr}/\tau_s = 0)\) in pure bending. The stress concentration with uniform stress is 8.4 times larger than that for the maximum gradient and hence stress gradient is intrinsically associated with material strength.

At equilibrium \((\tau_{Lead} = \tau_{peierls})\), Eq. 7.10 can be written as

\[
\tau_s = \sqrt{4Gb\tau_{peierls}} \frac{2}{\pi(1-\nu)l_{cr}} \frac{\alpha + \beta (1-l_{cr}/l)}{\alpha + \beta (1-\nu)}.
\]  

(7.11)

Equation 7.11 gives the magnitude of surface shear stress \((\tau_s > \tau_N)\) to satisfy equilibrium state due to the balance between Peierls stress and stress at the leading dislocation in dislocation pile-up for given parameters (depth of plastic deformation zone \((l_{cr})\) and material properties \((\nu, b, \text{and} \ G)\)). For the validation of the derivation, we consider a uniform shear stress case, i.e., \(\tau_{cr}/\tau_s = 1\). For \(\tau_{cr}/\tau_s = 1\), \(\alpha + \beta = 2\), Eq. 7.11 becomes

\[
\tau_s = \sqrt{4Gb\tau_{peierls}} \frac{2}{\pi(1-\nu)l_{cr}}
\]
which matches with the exact solution for uniform stress [94].

Using Eq. 7.11, Fig. 7.3(b) shows the nondimensional surface stress ($\tau_s^* = \tau_s/\tau_N$) as a function of the nondimensional sample size ($l^* = l/b$) with variation of nondimensional Peierls stress ($\tau_{peierls}^* = \tau_{peierls}/\tau_N$) for a given small depth ($l_{cr}^* = l_{cr}/l = 0.1$) of plastic deformation zone where $\tau_N^* = \tau_N/G = 1/40$ and $\nu = 0.28$. The result indicates that size dependent material strength is dependent on Peierls stress. For example, the values of $\tau_{peierls}^*$ and $l^*$ for $\tau_s^* = 1.5$ are summarized in Table 7.1 which indicates that size dependence occurs at larger sample size with increase in $\tau_{peierls}^*$. It is worth mentioning that in case of small Peierls stress ($\tau_{peierls}/\tau_N$) like FCC metals, the depth of plastic deformation zone will converge to $l$ and hence the yield strength is inversely proportional to square root of sample size, well known for geometrically necessary dislocation [22, 95].

Table 7.1: The summary for the values of $\tau_{peierls}^* = \tau_{peierls}/\tau_N$ and $l^* = l/b$ for $\tau_s^* = \tau_s/\tau_N = 1.5$

<table>
<thead>
<tr>
<th>$l^* = l/b$</th>
<th>0.01</th>
<th>0.2</th>
<th>0.4</th>
<th>0.6</th>
<th>0.8</th>
<th>1.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\tau_{peierls}^*$</td>
<td>19</td>
<td>377</td>
<td>751</td>
<td>1112</td>
<td>1501</td>
<td>1890</td>
</tr>
</tbody>
</table>

As mentioned in Chapter 1, Östlund et al [54] observed ductility in SCS samples with $<300\sim400$ nm in diameter at room temperature. Unlike micropillar compression tests, bending tests of silicon beam with a thickness 255nm [18], and silicon nanowire with diameter between 200nm and 300nm [19] did not show any plastic deformation at room temperature. Our analysis may offer a reasonable explanation for this discrepancy in the literature.

Upon mechanical load on an SCS sample, silicon atoms either rearrange their microstructure by dislocation nucleation or fail by propagation of a crack. At room temperature, bulk silicon always fails in brittle manner due to much smaller fracture strength than yield strength. With reduction of sample size, fracture strength increases significantly due to reduction of flaw size, but surface dislocation nucleation with smaller activation energy becomes relevant as discussed in Chapter 6. Hence yielding may be energetically favorable
during compression test as observed in [54]. However, in a bending test, a small scale sample may also yield, but the yield stress increases due to stress gradient. Thus the sample may fail by fracture before yielding, as observed in bending test [18, 19].

7.2 In situ thermo-mechanical experiment

For experimental validation of the size dependent yield strength in SCS, we use the in situ experimental data from Chapter 6. In order to compare yield strength for SCS samples with different $h$, we introduce a model which gives a force-displacement relation for SCS bending samples within and beyond linear elastic deformation regime.

7.2.1 Force-displacement relation: elastic deformation

First we consider the force-displacement relation of the bending sample within linear elastic deformation regime. The both ends (B and C in Fig. 7.4(a)) of the sample are clamped by gripping mechanism and the sample is subjected to a load $f$. The ends are free to translate axially. To load the sample, the upper loading frame (B) moves away from the fixed loading frame (C) in loading direction $f$. The applied load $f$ is measured by a force sensor. The corresponding deformation ($\delta$) of the bending sample is measured by Electron Microscopy images. During the derivation of the model, we assume small deformation, homogeneous and linear elastic material, the small slope of the deformed sample with repeat to unity, and perfect structural symmetry of a bending sample.

Due to the assumption of the perfect structural symmetry of the bending sample, we consider the free body diagram shown in Fig. 7.4(b). Here, the curve $a'-b'-c'-d'$ presents the shape of the neutral axis of the sample upon loading from the initial configuration of the bending sample $a-b-c-d$ where the resultant deformation of the sample $(d-d')$ becomes $\delta/2$ due to symmetry of the bending sample. We assume that the curvature of the deformed sample at any cross section depends only on the magnitude of the bending moment. Then
the moment-curvature relation can be written as

\[ M(x) = \frac{EI}{\rho(x)} \approx EI \frac{d^2y(x)}{dx^2} \]  \hspace{1cm} (7.12)

where \( \rho(x) \) is a radius of curvature due to bending, \( I \) is moment of inertia, and \( x \) and \( y \) are coordinates of the system as shown in Fig. 7.4(b). We confirmed that the small deformation assumption in Eq. 7.12 gives a force-displacement relation within 1% error with respect to the FEM results for the given sample deformation range of interest in Chapter 6. Hence we will use Eq. 7.12 in the following analysis. For the free body diagram in Fig. 7.4(b), we have

\[
\begin{align*}
M_a - (f/2)x &\approx EI_1 \frac{d^2y_{ab}}{dx^2}(x) \\
M_a - (f/2)x &\approx EI_2 \frac{d^2y_{bc}}{dx^2}(x)
\end{align*}
\]  \hspace{1cm} (7.13)

where \( M_a \) is a bending moment acting at \( a \) (see Fig. 7.4(b)). By using the boundary conditions \( y_{ab}(0) = 0, y_{ab}'(0) = 0, y_{ab}(l_{ab}) = y_{bc}(l_{ab}), \) and \( y_{ab}'(l_{ab}) = y_{bc}'(l_{ab}), \) Eq. 7.13 can be solved as

\[
\begin{align*}
y_{ab}(x) &= -\frac{x^2(-6M_a + fx)}{12EI_1} \\
y_{bc}(x) &= \frac{(I_1 - I_2)(l_{ab}(f(l_{ab}^2 - 3l_{bc}^2) - 6(l_{ab} - 2l_{bc})M_a) + 3l_{bc}(l_{bc} - 4M_a)x) + 6l_{bc}M_a x^2 - f l_1 x^3}{12EI_1 I_2}
\end{align*}
\]  \hspace{1cm} (7.14)

where \( I_1 \) and \( I_2 \) are moments of inertia at bending arms \( (l_{ab} \) and \( l_{cd} \)) and \( l_{bc}, \) respectively, as shown in Fig. 7.4(b) and (d). With \( y_{bc}''(l_{ab} + l_{bc}/2) = 0 \) due to the geometrical symmetry of the bending sample, \( M_a = f(2l_{ab} + l_{bc})/4 = fL/8. \) By substituting \( M_a \) to Eq. 7.14, \( y_{bc}(x) \) can be written as

\[ y_{bc}(x) = f EI_1 \left[ \left( \frac{l_{ab}^3}{6} - \frac{3l_{ab}^2 l_{bc}}{8} + \frac{l_{ab} l_{bc} x}{2} \right) \left( 1 - \frac{I_1}{I_2} \right) + \frac{I_1}{I_2} \left( \frac{l}{8} - \frac{x}{12} \right) x^2 \right]. \]  \hspace{1cm} (7.15)

For a uniform cross section case \( (I_1 = I_2), \) Eq. 7.15 gives a force-displacement relation for a simple beam case for the considered boundary conditions as expected. Note that for a case
of $I_1/I_2 = h_2^3/h_2^3 \ll 1$, Eq. 7.15 can be further simplified as

$$y_{bc}(x) \approx \frac{fl_{ab}}{2EI_1} \left( \frac{l_{ab}^2}{3} - \frac{3l_{ab}lb_{bc}}{4} + lb_{bc}x \right).$$

Finally, for the elastic deformation of the bending sample, $\delta_{EI}$ can be obtained by $\delta_{EI} = 4y_{bc}(x)$ at $x = l/2$.

### 7.2.2 Force-displacement relation: elastic-plastic deformation

Here we consider a stress-deformation of the bending sample beyond elastic deformation. As mentioned earlier, plasticity in SCS is expected by dislocation nucleation process rather than multiplication of preexisting dislocations and hence we assume that there is no work hardening during the early stage of SCS sample yield. The maximum bending stress occurs at $a$ (see Fig. 7.4(b)). Hence at the onset of plasticity of the sample ($\sigma_{\text{max}} = \sigma_y$) we have[85]

$$\sigma_y = \frac{M_{\text{max}}c}{I_1} = \frac{3}{16} \frac{fL}{b_w c_1^2}$$

at $x = 0$ and $y = c_1$ where $b_w$ is uniform width of the bending sample, $\sigma_y$ is yield strength, and $L$ is the total length of the bending sample (see 7.4(b)). Hence the critical force, $f_{cr}$, associated with the onset of the plastic deformation at $a$ is

$$f_{cr} = \frac{16b_w c_1^2}{3L} \sigma_y.$$ 

Now consider material yield at arbitrary cross section $p$ between $a$ and $b$ in Fig. 7.4(b). Suppose the applied force is sufficiently large such that magnitude of bending stress at $\kappa = \pm \kappa_{cr}$ (see Fig. 7.4(c)) due to $M(x)$ is equal to $\sigma_y$, i.e., $\sigma(\kappa_{cr}) = \sigma_y$. As we assumed no work hardening, the bending stress state at $p$ in Fig. 7.4(b) alters after material yield as schematically shown in Fig. 7.4(c). Due to the conservation of bending moment $M(x)$, the
elastic and elastic-plastic stress state at \( p \) satisfies

\[
M(x) = f/2(L/4 - f x) \tag{7.16}
\]

\[
= 2 \left( \int_0^{\kappa_{cr}} \kappa^2 \frac{\sigma y}{\kappa_{cr}} b_w d\kappa + \int_{\kappa_{cr}}^{\kappa_1} \sigma y \kappa b_w d\kappa \right)
\]

\[
= \sigma y b_w \left( c_1^2 - \kappa_{cr}^2/3 \right).
\]

From Eq. 7.16, \( \kappa_{cr} \) can be written as

\[
\kappa_{cr} = \sqrt{3 \left( c_1^2 - \frac{f}{\sigma y b_w} \left( L - \frac{x}{2} \right) \right)}.
\]

Let \( \kappa_{cr} = c_1 \) then \( x_{cr} = L/4 - 4\sigma y b_w c_1^2/3f \) and thus plastic deformation zone can be defined as \( c_1 > \kappa > \kappa_{cr} \) and \( x_{cr} > x > 0 \).

From \( \sigma(x, \kappa) = M(x) \kappa/I \) and Eq. 7.12, \( \sigma(x, \kappa) = E\kappa d^2 y/dx^2 \) which gives \( \sigma_y = E\kappa_{cr} y'' \) or \( \kappa_{cr} = \sigma_y/E y'' \) at \( \kappa = \kappa_{cr} \) and \( \sigma = \sigma_y \). Equation 7.16 can be written as

\[
M(x) = f/2(L/4 - f x)
\]

\[
= \sigma y b_w \left( c_1^2 - \frac{1}{3} (\sigma_y/E y'')^2 \right)
\]

and hence we have

\[
\begin{cases}
\frac{d^2 y_{axcr}}{dx^2} = \beta / \sqrt{3 - \frac{3}{\alpha} \left( \frac{f L}{8} - \frac{f x}{2} \right)} \text{ where } x_{cr} > x > 0 \\
\frac{d^2 y_{axcrb}}{dx^2} = \frac{1}{E_1} \left( \frac{f L}{8} - \frac{f x}{2} \right) \text{ where } l_{ab} > x > x_{cr} \\
\frac{d^2 y_{axc}}{dx^2} = \frac{1}{E_2} \left( \frac{f L}{8} - \frac{f x}{2} \right) \text{ where } l/2 > x > l_{ab}
\end{cases}
\tag{7.17}
\]

where \( \alpha = \sigma y b_w c_1^2 \) and \( \beta = \sigma_y/E c_1 \). With boundary conditions \( y_{axcr}(x) = 0 \), \( dy_{axcr}(x)/dx = 0 \) at \( x = 0 \), \( y_{axcr}(x) = y_{xcrb}(x) \), \( dy_{axcr}(x)/dx = dy_{xcr}(x)/dx \) at \( x = x_{cr} \), and \( y_{xcrb}(x) = y_{bc}(x) \), \( dy_{xcrb}(x)/dx = dy_{bc}(x)/dx \) at \( x = l_{ab} \) and using a condition from symmetric geometry
(d^2y_{bc}(x)/dx = 0 at x = l/2), Eq. 7.17 can be solved and \( \delta_{PL} \) can be written as

\[
\delta_{PL} = \frac{\lambda(8c^3 - h_2^3) l_{bc}^3 \sigma_y}{3cEh_2^3l} - \frac{\zeta l^2 \sigma_y}{3cE\lambda} - \frac{(-5 + 3\zeta)l^2 \sigma_y}{3cE\lambda^2}
\] (7.18)

where \( \zeta = \sqrt{3 - 2\lambda} \) and \( \lambda = f/f_{cr} \). In the following section, we will use Eq. 7.18 to compute yield strength of SCS samples.

### 7.2.3 Yield Strength Measurement

Here we use the in situ experimental data for SCS samples with \( h = 8.7 \mu m, = 1.5 \mu m, \) and \( = 720nm \) from Chapter 6 to validate size dependent yield strength predicted by our analytical model. To obtain yield strength of SCS sample, we use least squares fitting between the experimental data in Fig. 6.2 and Eq. 7.18. For example, the best fit for the sample with \( h = 720nm \) at 340°C is shown in Fig. 7.5(a). The yield strength for other two sample sizes is also obtained by the same procedure as the results are shown in Fig. 7.5(b). The yield strengths are \( \sigma_y = 0.626GPa, = 1.2GPa, \) and \( = 1.737GPa \) for \( h = 8.7 \mu m, = 1.5 \mu m, \) and \( = 720nm, \) respectively. The experimentally data indicate that the flow stress of SCS depends on sample size and hence validate our model prediction. Note that the yield strength for \( h = 8.7 \mu m \) is measured at 375°C as no plasticity was measured at 340°C due to size dependent BDT (see Chapter 6 for details).

### 7.3 Conclusion

In the present chapter, we showed increase in yield strength with sample size reduction for SCS samples. The fundamental mechanism for the size dependent plasticity is associated with large Peierls stress for SCS, size dependent stress gradient in bending, and the corresponding dislocation population within the slip planes. To theoretically study the role of size on yield behavior of SCS samples, we considered SCS samples with variation of sample
thickness in pure bending. For a given maximum bending stress at the free surface ($\sigma_{max}$), a smaller sample has significantly larger stress gradient than a larger sample as bending stress varies from $\sigma_{max}$ to zero within the half thickness of the samples. Therefore, the local bending stress or equivalently the corresponding local resolved shear stress along a slip direction on a slip plane sharply decreases for a smaller sample. With the larger stress gradient within the smaller sample, the local resolved shear stress balances with Peierls stress within small distance from a dislocation nucleation source at free surface. In such case, it is difficult to have the following dislocations after nucleation of the first dislocation from the surface dislocation source due to the following reasons. First, for the further movement of the dislocation against the larger stress gradient, it is required to apply much larger bending stress. Second, due to the large stress gradient, the leading dislocation is located near the surface dislocation source at the equilibrium and hence introducing a following dislocation requires much larger stress as the repulsive force between the leading dislocation, which is effectively pinned near the source by the interplay of large Peierls stress and large stress gradient, and the following dislocation is inversely proportional to the distance between them. On the other hand, for larger samples with the maximum bending stress $\sigma_{max}$, the leading dislocation can move further away from the dislocation nucleation source due to smaller stress gradient and as a result, it becomes easier to nucleate the following dislocations as the repulsive interaction force between two dislocation sharply decreases with the distance. With the larger number of dislocations within dislocation pile-up, a stress concentration factor at the leading dislocation increases which facilitates further movement of the leading dislocation even when the local resolve shear stress is substantially smaller than Peierls stress. Hence it is easier to yield larger samples as we validated with experimental results.
7.4 Figures

Figure 7.1: The schematic of surface dislocation nucleation in SCS. The SCS sample with the length (L) and thickness (d) is shown in (a). The sample is subjected to pure bending moment M. The zoom-in view (b) of area C in (a). Dislocations nucleate from A and move along slip plane (A-B). s is a coordinate along the slip direction from A. ΔL is a mean average distance between slip bands. (c) shows stress state in the sample due to M. In (d), the resolved shear stress along s-coordinate is shown.
Figure 7.2: (a) and (b) show dislocation density \( n^*(s) = n(s)/P \) for \( \tau_{cr}/\tau_s = 0 \) and \( = 1 \) cases within \( 1 \geq s/(l_{cr}/2) \geq -1 \), respectively, where \( P = 2(1 - \nu)/Gb \).

\[ \eta = s/(l_{cr}/2) \]

\[ n^*(\eta) = n(\eta)/P \]

\[ \tau_{cr}/\tau_s = 0 \]

\[ \tau_{cr}/\tau_s = 1 \]

\[ \text{Quadratic approximation} \]

\[ \text{Case with } \kappa = 100 \]
Figure 7.3: (a) shows nondimensional resolved shear stress \( \tau^*_{\text{Lead}} = \tau_{\text{Lead}} / Q \) at the leading dislocation of dislocation pile up \( s = -l_{cr} / 2 \) as a function of stress gradient \( 1 \geq \tau_{cr} / \tau_s \geq 0 \) where \( Q = (1 - \nu)\tau_s^2 l_{cr} / Gb \). (b) shows nondimensional yield stress \( \tau^*_s = \tau_s / \tau_N \) as a function of non dimensional sample size \( l^* = l / b \) with variation of nondimensional Peierls stress \( \tau^*_{\text{peierls}} = \tau_{\text{peierls}} / \tau_N \).
Figure 7.4: The schematic of bending sample: (a) overall structure of the sample with eight bending arms, (b) free body diagram of the upper part of the sample (see Area A in (a)), (c) bending stress state at cross section p in (b) due to elastic and elastic-plastic deformation of the sample, (d) the sample geometry at the designated area in (b).
Figure 7.5: (a) Measurement of yield strength for SCS samples using a least squares fitting between the experimental data and the elastic-plastic force-displacement model for $h = 720\,\text{nm}$ sample. (b) shows yield stress as a function of sample size using the least squares fitting.
Chapter 8

Conclusions and Future Research

The main goal of the present work was to study thermally and mechanically coupled behavior of micro/nanomaterials with a special focus on brittle-to-ductile transition in single crystal silicon. For thermo-mechanical study of micro/nano scale SCS samples, we developed the novel micro device and carried out quantitative in situ experiment as we presented in the previous chapters. Using this unique in situ thermo-mechanical testing ability, we have unambiguously shown that BDT behavior indeed depends on sample size and temperature. Also we have revealed that yield strength of SCS samples depends on the sample size. In this chapter we summarize the present thesis with the key findings. We conclude this chapter with brief discussion on the future work and potential research topics associated with the applications in the energy sector.

8.1 Conclusion

In chapter 2, we showed the bending stress on the sample or the deviation from the uniform stress (= load/area) scales inversely with the sample size. We first analytically studied the influence of the transverse and rotational misalignment on the stress state of a sample subjected to uniaxial tensile test. For the transverse misalignment, the non-uniform stress error is proportional to the ratio of misalignment to cross sectional characteristic length. As the characteristic length of a sample decreases, the misalignment error increases and the influence of non-uniform stress becomes unavoidable for smaller samples. The error due to rotational misalignment of the sample with respect to the loading frames is also
considered. For a given rotational misalignment, the non-uniformity in cross sectional stress is higher near the support and increases with average axial strain. Due to the influence of these misalignments, non-uniform stress in the specimen results in large error in elastic modulus measurement when strain is measured at the surface of the sample. Hence we conclude that data interpretation requires careful analysis due to intrinsic nonuniform stress at micro/nanoscale.

In chapter 3, we theoretically considered misalignment error from the multiple sources. Our analysis showed that misalignment error can be significant due to limited precision on the grip between the sample and the loading stage in particular at micro/nanoscale. We carried out numerical experiment of a tensile test using the proposed stage. The results indicated inherent nonlinearity between applied loading and material deformation of a material sample even within linear elastic deformation regime. Then we used the novel design for the specimen with self-aligning hinges to overcome this challenge. With analytical and numerical simulation, we showed that the hinges significantly suppress any misalignment errors within the entire gauge length of the sample.

In chapter 4, we experimentally carried out uniaxial test on small scale samples using the novel MEMS stage with in-built grips and self aligning mechanism, and a sample with built-in hinges. With in situ experimental investigation, we validated our theoretical analysis in Chapter 3. For example, we found that the error in elastic modulus measurement from surface strain can be up to 50% within small linear elastic deformation for a silicon sample. We suppressed such large misalignment error using the proposed self-aligning mechanism as we measured elastic modulus of a micro scale silicon with 99% accuracy.

In Chapter 5, we presented the SiC based MEMS stage to test micro/nanoscale materials with control of sample size and temperature. We theoretically and experimentally characterized the stage. We showed that Joule heating of the SiC stage can raise sample temperature uniformly up to 700°C. Using the method, we recovered the known elastic modulus of the SCS sample at room temperature within 1% error and reduced moduli at elevated
temperatures up to 403°C.

In Chapter 6, we showed that BDT can occur at much lower temperature than bulk BDT temperature due to strong surface effect with decrease in sample size. With in situ thermo-mechanical measurement, up to 31.2% reduction in the BDT temperature was measured. The continuous trend between bulk to surface dominant plasticity was explained using the Harmonic Transition - State Theory based model.

In Chapter 7, we explored size dependent yield strength of SCS samples incorporated with their large Peierls stress and stress gradient due to bending. The model predicted that size dependence can be stronger in SCS as large Peierls stress can behave as dislocation obstacles. For experimental study, we used the novel in situ thermo-mechanical testing method using the SiC based microdevice. We, for the first time, experimentally validated size dependent yield strength in SCS.

8.2 Recommended future work

We have shown that the BDT behavior in SCS is sensitive to sample size. For further investigation of surface effects on the plasticity in SCS, different passivation layers such as SiO$_2$ and Si$_3$N$_4$ on SCS samples can be used. It is expected that there will be different effects from these two passivation schemes as schematically shown in Fig. 8.1. In the case of SiO$_2$, which is brittle, cracking of the oxide layer would occur early in the deformation and in the process expose the free surface. Once this happens, we expect plasticity to localize around the cracked region due to the availability of the free surface and the stress concentration created by the crack. The failure of the sample would also most likely initiate from one of the cracks. Si$_3$N$_4$, on the other hand, will accommodate a much higher strain elastically and therefore we expect that surface dislocation nucleation may not be activated. Alternative deformation mechanism, for example, the generation of threading dislocations pinned at the two Si/Si$_3$N$_4$ interfaces may play a key role. In both cases we expect plasticity to be
reduced compared to unpassivated films due the constraints imposed by the interface. These observations would provide direct evidence of the role of surfaces in the plastic deformation of Si at low temperatures.

8.3 Discussion on potential research: Materials in Extreme Environments

The panel on thermomechanical extremes concluded that designing new materials with properties specifically tailored to withstand thermomechanical extremes must begin with understanding the fundamental chemical and physical processes involved in materials failure, extending from the nanoscale to the collective behavior at the macroscale from Report of the Basic Energy Sciences Workshop on Materials under Extreme Environments (2007).

The need for high temperature materials is higher than ever for applications in not only traditional energy sector, but alternative future energy applications. For example, high temperature materials are essential for designing fuel efficient steam turbines, heat exchangers, fuel efficient vehicles, and nuclear reactors as higher operational temperature for those systems is desired for higher energy efficiency. Also, for the future alternative energy application, fundamental study of thermo-mechanical behavior of materials at the microstructure level is critical, e.g., solid oxide fuel cell (SOFC) applications. The increase in thermo-mechanical stress due to high temperature operation of SOFC may cause catastrophic failures of the materials which can lead to tremendous capital loss. In order to prevent such failures and engineer novel materials for energy applications at extreme temperatures, the fundamental understanding of the microstructural activities, i.e., dynamics of dislocations, voids, and grain boundaries, and their impact on material deformation, failure, and degradation are necessary. The in situ method for high temperature material testing in advanced analytical tools will allow to reveal the key roles of these defects in thermo-mechanical material response. The result of the study will provide design criteria of the novel materials for
extended lifespan, enhanced reliability, and increased efficiency even at higher temperature.
8.4 Figures

Figure 8.1: Further investigation of the surface effect: (a) a unpassivated sample, (b) SiO$_2$ passivation with the cracks, and (c) Si$_3$N$_4$ passivation.
References


