MEMS AND ROBOTICS-BASED MANIPULATION AND CHARACTERIZATION OF MICRO AND NANOMATERIALS

by

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A thesis submitted in conformity with the requirements for the degree of Doctor of Philosophy
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Abstract

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Advances in the synthesis of micrometer and nanometer-sized materials have resulted in a range of novel materials having unique properties. Characterizing those materials is important for understanding their properties and exploring their applications. Physically manipulating those materials is important for constructing devices. This thesis develops tools and techniques for the manipulation and characterization of micro and nanomaterials.

A microelectromechanical systems (MEMS) microgripper is developed to pick and place micro-objects, achieving high repeatability, accuracy, and speed. The adhesion forces at the microscale are overcome by actively releasing the adhered micro-object from the microgripper. A microrobotic system is built based on this microgripper and realizes automated pick-and-place of microspheres to form patterns.

To characterize the electrical properties of one-dimensional nanomaterials, a nanorobotic system is developed to control four nanomanipulators for automated four-point probe measurement of individual nanowires inside a scanning electron microscope (SEM). SEM is used as a vision sensor to realize visual servo control and contact detection.

To characterize the electromechanical properties of individual nanowires, a MEMS device is designed and fabricated that is capable of simultaneous tensile testing and current–voltage measurement of a nanowire specimen. A nanomanipulation procedure is developed to transfer a single nanowire from its growth substrate to the MEMS device in SEM. The piezoresistive properties of silicon nanowires are characterized.
A nanomanipulation system is developed that is capable of being mounted onto and de-mounted from the SEM specimen stage without opening the high-vacuum chamber. The system architecture allows the nanomanipulators to be transferred through the SEM load-lock. This advance facilitates the replacement of end-effectors and circumvents chamber contamination due to venting.
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Contents

1 Introduction 1
  1.1 Background and Motivations ................................. 1
  1.2 Objectives ................................................. 4
  1.3 Dissertation Outline ...................................... 4

2 Pick-and-Place of Micro-Objects Using a MEMS Microgripper 5
  2.1 Introduction ................................................ 5
  2.2 Three-Pronged Microgripper ................................ 8
  2.3 Force Analysis .............................................. 11
  2.4 Experimental Results and Discussion ...................... 15
    2.4.1 Repeatability of Active Release .................... 16
    2.4.2 Quantification of Release Performance ............. 17
    2.4.3 Understanding the Curved Trajectory .............. 20
  2.5 Microrobotic Pick-and-Place of Microspheres ............. 22
    2.5.1 Recognition of Microgripper and Microspheres ...... 22
    2.5.2 Contact Detection and Microrobotic Control ........ 23
    2.5.3 Automated Pick-and-Place of Microspheres .......... 26
    2.5.4 Three-Dimensional Assembly of Microspheres ....... 27
  2.6 Conclusions ............................................... 28
3 Automated Four-Point Probe Measurement of Nanowires in SEM

3.1 Introduction .................................................. 29

3.2 Automated Nanomanipulation inside SEM ..................... 31
  3.2.1 Nanomanipulation System ................................. 31
  3.2.2 Recognition of Probes and Nanowires .................... 33
  3.2.3 Feature Tracking ........................................ 34
  3.2.4 Vision-Based Contact Detection .......................... 34
  3.2.5 Look-then-Move Control and Visual Servo Control ...... 35
  3.2.6 Overall Process of Automated Nanomanipulation ......... 37

3.3 Experimental Results and Discussions ....................... 38
  3.3.1 Manual Operation ....................................... 38
  3.3.2 Evaluation of Drift and Noise in SEM Imaging .......... 39
  3.3.3 Contact Detection Results .............................. 41
  3.3.4 Performance of Vision-Based Control System ............ 42
  3.3.5 Four-Point Probe Measurement Results .................. 43

3.4 Conclusions ................................................ 45

4 Piezoresistivity Characterization of Nanowires using a MEMS Device

4.1 Introduction ................................................ 46

4.2 Device Design and Analysis ................................ 48
  4.2.1 Device Design ........................................ 48
  4.2.2 Mechanical Analysis .................................. 50
  4.2.3 Electrical Analysis ................................... 52

4.3 Device Fabrication and Calibration .......................... 53
  4.3.1 Microfabrication ...................................... 53
  4.3.2 Sensor Calibration ................................... 56

4.4 Experimental Results and Discussions ....................... 56
  4.4.1 Synthesis of Silicon Nanowires ......................... 56
4.4.2 Transfer of Nanowire onto MEMS Device .......................... 57
4.4.3 Nanowire Characterization ......................................... 59
4.4.4 Effect of E-Beam Irradiation ......................................... 61
4.5 Conclusions ..................................................................... 62

5 A load-lock-compatible nanomanipulation system for SEM 64
5.1 Introduction ..................................................................... 64
5.2 System Design .................................................................. 67
5.3 System Characterization ..................................................... 69
5.3.1 SEM Imaging ............................................................... 69
5.3.2 Actuator Characterization ................................................. 70
5.3.3 Encoder Characterization ............................................... 73
5.3.4 Control .................................................................... 74
5.4 Results and Discussions .................................................... 77
5.5 Conclusions ................................................................. 81

6 Conclusions .................................................................. 82
6.1 Contributions ................................................................. 83
6.2 Future Directions ............................................................ 83
List of Tables

2.1 Structural Parameters of the Microgrippers .......................... 10
2.2 Summary of Release Accuracy ............................................. 20
3.1 Quantification of SEM Image Drift and Noise ............................ 40
5.1 Resolution of the Optical Encoders ....................................... 74
5.2 Summary of System Performance ......................................... 78
# List of Figures

1.1 Manipulation of nanomaterials for mechanical and electrical characterization. . 3

2.1 SEM image of a three-pronged microgripper. . . . . . . . . . . . . . . . . . . 7
2.2 Microgripper schematic. . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 9
2.3 Microfabrication process. . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 10
2.4 Characterized microactuator performance. . . . . . . . . . . . . . . . . . . . 11
2.5 Adhesion forces acting on a microsphere on a rough surface. . . . . . . . . . 12
2.6 Analysis of forces during grasping and active release. . . . . . . . . . . . . . 14
2.7 Experimental setup for micrograsping and active release tests. . . . . . . . . 16
2.8 Landing positions of microspheres. . . . . . . . . . . . . . . . . . . . . . . . 18
2.9 Landing positions of microspheres released from 2 μm height. . . . . . . . . 19
2.10 Pick-and-place to align microspheres. . . . . . . . . . . . . . . . . . . . . . . 21
2.11 High-speed videography quantifying microsphere trajectories. . . . . . . . 22
2.12 Microsphere reveals a curved trajectory during active release. . . . . . . . 23
2.13 Gripper recognition. . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 24
2.14 Visual determination of which gripping arm the microsphere adheres to. . . 25
2.15 Vision-based contact detection. . . . . . . . . . . . . . . . . . . . . . . . . . 25
2.16 Contact detection results. . . . . . . . . . . . . . . . . . . . . . . . . . . . . . 25
2.17 “U OF T” pattern formed by automated microrobotic pick-and-place. . . . 26
2.18 Pattern formation by automated pick-and-place. . . . . . . . . . . . . . . . . 27
2.19 Microspheres assembled into two-layered structures. . . . . . . . . . . . . . 27
3.1 Four nanomanipulators with probes installed for manipulation in SEM.

3.2 Visual recognition of probes and nanowires from SEM visual feedback.

3.3 Principle of vision-based contact detection.

3.4 SEM vision-based control.

3.5 Characterization of a piezo actuator for look-then-move control.

3.6 Four probes simultaneously move to their target positions on a nanowire.

3.7 Manual operation often causes probe tip damage.

3.8 Automated contact detection and subsequent Z-positioning of a probe.

3.9 Step responses of the vision-based control system.

3.10 Four probes landing on target positions for four-point probe measurement.

3.11 Four-point probe measurement results of a nanowire.

4.1 Schematics of the MEMS device for electromechanical characterization.

4.2 Mechanical model of the device during mechanical characterization.

4.3 Circuit diagram of the testing setup during electrical characterization.

4.4 Microfabrication process flow.

4.5 MEMS device wire-bonded to a circuit board.

4.6 Calibration results of the displacement sensor and force sensor.

4.7 Nanowire transfer.

4.8 Mechanical and electrical characterization results.

4.9 Effect of e-beam irradiation.

5.1 Commercial nanomanipulation systems.

5.2 Load-lock-compatible nanomanipulation system.

5.3 Installation procedure of the nanomanipulation system.

5.4 Quantification of SEM image drift and noise.

5.5 Actuator characterization results.

5.6 Encoder characterization results.
5.7 Contact detection results. ........................................... 77
5.8 Control system performance. ...................................... 79
5.9 Nanowire probing. .................................................. 80
Chapter 1

Introduction

1.1 Background and Motivations

A variety of micro and nanomaterials have been synthesized that exhibit unique mechanical, electrical, and photonic properties, and have been used as functional elements in device applications. For example, self-assembly of silica microspheres resulted in a photonic crystal with a complete three-dimensional bandgap [1]. Semiconductor nanowires were used to construct nanoelectronic circuits [2], solar cells [3], and nanosensors for the detection of biological and chemical species [4].

Device construction usually requires the positioning of micro and nanomaterials. Taking one-dimensional nanomaterials as an example, nanowires/nanotubes need to be positioned between source and drain electrodes for building nanotransistors and biosensors. To position relatively large amounts of materials simultaneously, large-scale methods are used, namely, self-assembly [1], contact printing [5], and dielectrophoresis [6]. However, these methods represent probabilistic strategies and are not capable of precision control of individual materials.

By contrast, mechanical manipulation, despite being slow in comparison with the aforementioned large-scale methods, promises specificity, precision, and programmed motion, and thus, can enable the precise manipulation of individual materials. For the manipulation of mi-
cromaterials, a micromanipulator under an optical microscope is used. The end-effector can be either a microprobe or a microgripper. Owing to the strong adhesion forces (capillary forces, electrostatic forces, and van der Waals forces) at the microscale, manipulation is unreliable and has low repeatability, motivating the development of suitable manipulation tools and strategies.

Different from micromanipulation, manipulation of nanomaterials requires higher-resolution microscopes, typically atomic force microscopes (AFM) or scanning electron microscopes (SEM). AFM nanomanipulation can be conducted in either vacuum, ambient, or aqueous environments. Nonetheless, since AFM uses the same cantilever for both imaging and manipulation [7–9], performing truly simultaneous imaging and manipulation is a challenge. In contrast, SEM nanomanipulation uses nanomanipulators inside an SEM and is conducted in situ with video rate imaging.

An important application of SEM nanomanipulation is to characterize the mechanical and electrical properties of individual nanowires/nanotubes. Figure 1.1(a) shows that two AFM cantilevers stretch a multi-walled carbon nanotube (MWCNT) for tensile testing [10]. Individual MWCNTs are first picked up by one cantilever and then attached to the second cantilever. In Figure 1.1(b), an AFM cantilever buckles a bundle of MWCNTs for mechanical characterization [11]. An InGaAs/GaAs nanospring was tensile-tested between a probe and an AFM cantilever [12], as shown in Figure 1.1(c). Figure 1.1(d) schematically illustrates that a silicon nanowire is stretched for the characterization of its piezo resistivity. The green-colored probe is used to push the freestanding cantilever and stretch the suspended silicon nanowire [13]. The two red probes form electrical connections for measuring the resistance changes of the nanowire during straining.

State-of-the-art SEM nanomanipulation is conducted via teleoperation. An operator monitors the SEM screen and carefully operates a joystick to move a nanomanipulator (thus position an end-effector) inside an SEM. The operation process is time consuming, skill dependent, and poor in repeatability. Thus, developing nanorobotic techniques that facilitate this process and enable automation is desired.
Figure 1.1: Manipulation of nanomaterials for mechanical and electrical characterization. (a) A MWCNT was tensile-tested by two AFM cantilevers [10]. (b) A bundle of MWCNTs was buckled by an AFM cantilever [11]. (c) An InGaAs/GaAs nanospring was stretched between a probe and an AFM cantilever [12]. (d) A silicon nanowire was stretched for piezoresistivity characterization [13].

In addition, the aforementioned nanomaterial characterization techniques [Figure 1.1] rely on SEM imaging for deformation and force measurements. However, the electron-beam irradiation affects the electrical measurement of a nanomaterial. Therefore, it is desirable to have a testing device that is capable of acquiring all the testing data electronically without relying on SEM imaging. Given that MEMS (microelectromechanical systems) actuators and force sensors can deliver nanometer motion resolutions and nanonewton force resolutions, they can be used for nanomaterial testing.

Since end-effectors (e.g., nanoprobes or nanogrippers) can be easily damaged during nanomanipulation, they require frequent replacement. The replacement process has two drawbacks because the high-vacuum chamber of SEM must be opened to access the nanomanipulators. First, exposing the chamber to air incurs contamination. Second, pumping the chamber again
takes a few hours. Therefore, it is desirable to have a nanomanipulation system that does not require the opening of the high-vacuum chamber for end-effector exchange. Since SEM specimens are transferred through the SEM load-lock, using the load-lock also for nanomanipulator transfer is a solution.

1.2 Objectives

The objectives of this thesis are:

- To develop a MEMS microgripper to pick and place micro-objects with high repeatability, accuracy, and speed, and build a microrobotic system for automated pick-and-place.

- To develop nanorobotic techniques and a MEMS device to characterize the electrical, mechanical, and electromechanical properties of nanomaterials in SEM.

- To develop a nanomanipulation system that is capable of being transferred through the load-lock of SEM without opening the high-vacuum chamber.

1.3 Dissertation Outline

An overview of the ensuing chapters is as follows. Chapter 2 presents a MEMS microgripper that is capable of overcoming adhesion forces to release adhered micro-objects, and the demonstration of automated pick-and-place. Chapter 3 presents a nanorobotic system in SEM for automated four-point probe measurement of nanowires. Chapter 4 presents a MEMS device for the characterization of piezoresistive properties of nanowires. Chapter 5 presents a load-lock-compatible nanomanipulation system for use in SEM and the system characterization results. Chapter 6 concludes with a summary and contributions of this thesis, and possible future research directions.
Chapter 2

Pick-and-Place of Micro-Objects Using a MEMS Microgripper

2.1 Introduction

Physical pick-and-place of micro-objects promises specificity, precision, and programmed motion, features making micromanipulation amenable to automation for the construction of microsystems [14–17]. For instance, micromanipulation has been used to build a diamond-shaped structure by assembling microspheres into a lattice [14]. Based on a combination of microfabrication and micromanipulation [16], novel photonic crystals were also demonstrated.

Analogous to manipulation in the macroworld, manipulating micrometer-sized objects necessitates gripping devices with end structures comparable in size to objects to be manipulated. Enabled by MEMS technologies, many microgripping devices have been reported, including two-fingered devices [18–28] and multi-fingered devices [29–35].

Despite the availability of MEMS gripping tools and the significant progress made in automation techniques for eventually autonomous operation, micromanipulation is still largely skill dependent and entails repeated trial-and-error efforts. Among the challenges, a long-standing difficulty is the release of micro-objects from the end effector due to strong adhesion
forces at the microscale. Force scaling causes surface forces (i.e., adhesion forces) including the capillary force, electrostatic force, and van der Waals force to dominate volumetric forces (e.g., gravity) [36]. In pursuit of rapid, accurate release methods, several strategies have been proposed in the past decade.

These release techniques can be classified into two categories, passive release and active release. Passive release techniques depend on the adhesion forces between the micro-object and the substrate to detach the micro-object from the end effector. In consideration of adhesional and rolling-resistance factors [37, 38], microspheres were rolled on an Au-coated substrate for both pick and release, causing the fracture of the microsphere–substrate interface and the microsphere–tool interface, respectively. Similarly, it was also demonstrated that substrates with a ultraviolet cure adhesive [39] or a gel film [40] were used to facilitate release. Another passive release technique uses the edge of the substrate to scrape the adhered object off the tool [41]. A commonality of passive release techniques is the dependency on surface properties of substrates, time-consuming, and poor repeatability.

By contrast, active release methods intend to detach the micro-object from the end effector without touching the substrate. By applying a voltage between the probe and the substrate [42–45], an electric field was created to detach the object from the probe. Nevertheless, this method requires the micro-object, the probe, and the substrate all to be conductive. More importantly, the released micro-objects landed at random locations on the substrate, resulting in a poor release accuracy.

The second type of active release makes use of mechanical vibration [46, 47]. Requiring a large bandwidth of the manipulator, the vibration-based method takes advantage of inertial effects of both the end effector and the micro-object to overcome adhesion forces. The release process has been modeled and simulated to predict the landing radius of the released object [48]; however, the accuracy has not been experimentally quantified. The third type of active release employs vacuum-based tools [49] to create a pressure difference for pick and release. However, miniaturization and accurate control of vacuum-based tools can be difficult, and its
Figure 2.1: SEM image of a three-pronged microgripper capable of both grasping and active release.

use in a vacuum environment can be limited. Finally, micro peltier coolers were used to form ice droplets instantaneously for pick-and-place of micro-objects [50–52]. Thawing of the ice droplets was used to release objects. The freezing-heating approach requires a bulky, complex end effector and is limited to micromanipulation in an aqueous environment.

In summary, no techniques exist for easy, rapid, accurate, and highly repeatable release of micro-objects in micromanipulation. This chapter presents an active release strategy using a MEMS microgripper that integrates a plunging structure between two gripping arms, as shown in Figure 2.1. While this method retains the advantage of double-ended tools for picking up micro-objects, the plunger is capable of thrusting a micro-object adhered to a gripping arm to a desired destination on a substrate, enabling highly repeatable release with a high accuracy of 0.45±0.24 µm.

This chapter also presents the theoretical analyses of the micromanipulation process in order to understand the microphysics behind this active release technique. All the results in this chapter were obtained under an optical microscope with 7.5–10.9 µm borosilicate microspheres on glass and steel substrates in an ambient environment. No surface treatments were conducted
to the microspheres, microgripper, or substrates.

Enabled by the grasping and release capabilities, a microrobotic system is built that achieved fully automated pick-and-place of microspheres at a speed of 10 spheres/min. This speed is an order of magnitude higher than the highest speed reported in the literature [53]. Image processing is used to recognize features such as the gripping arms and microspheres. The system detects the contact between the microgripper and the substrate purely through visual feedback without using additional force/touch sensors. Automated pick-and-place was performed through vision-based control.

2.2 Three-Pronged Microgripper

Figure 2.2 shows a schematic of the microgripper. The monolithic device integrates three electrostatic microactuators for driving two normally open gripping arms as well as a plunger for active release. In this design, electrostatic actuation was chosen over electrothermal actuation because temperature rise of the gripping arms can influence adhesion forces [54] and reduce the consistency of device performance. Furthermore, electrostatic actuation was also chosen for driving the plunger because it exhibits a much higher bandwidth than electrothermal actuators and is able to deliver a much faster speed, representing an important advantage for thrusting off an adhered micro-object.

This design is different from existing microgrippers that have either only one actively actuated gripping arm [20–22] or two interdependently active gripping arms [18]. Since to which gripping arm a micro-object adheres is random, both gripping arms in our design have an independent actuator for positioning the adhered object to properly align to the plunger for release. Structural parameters of the microgrippers are shown in Table 2.1. The thickness of the gripping arms and actuators is 25 µm, the thickness of the device layer. The initial opening between the gripping arms is 17 µm, intended to accommodate microspheres of around 10 µm in diameter. The widths of the gripping arm tips (6 µm) and plunger tip (8 µm) are slightly smaller
than the microsphere to be grasped so that the microgripper does not contact microspheres in proximity. The number of combs for each gripping arm is 280 to ensure the gripping arms can be fully closed at an actuator voltage of 60 V, according to the electrostatic force calculation results. The number of combs for the plunger is 540 to ensure the plunger tip can pass the gripping arm tips at an actuator voltage of 60 V.

The devices were microfabricated using a modified DRIE on SOI process [21] with a 25 μm thick device silicon layer, as illustrated in Figure 4.4. Two-step DRIE etching of the handle layer creates a step difference between the central suspended structure and the device frame, which greatly reduces the risk of device breakage during device operation and handling. Figure 2.4 shows the experimentally characterized device performance as well as fitted lines.

The device also permits experimentally estimating adhesion forces between the gripping arms and a grasped microsphere. After the microsphere is gently but securely grasped, the actuation voltages for the gripping arms are released in a continuous and synchronous manner until the voltage, $V_2$ at which the gripping arms are opened is obtained. The adhesion forces can then be estimated as

$$F = \frac{1}{2} \frac{N \epsilon t}{b} \left( V_1^2 - V_2^2 \right),$$

where $\epsilon$ is the permittivity of air, $N$ is the number of comb finger pairs, $t$ is the comb fin-
Table 2.1: Structural Parameters of the Microgrippers

<table>
<thead>
<tr>
<th>parameter</th>
<th>value</th>
</tr>
</thead>
<tbody>
<tr>
<td>device Si layer thickness (t)</td>
<td>25 µm</td>
</tr>
<tr>
<td>width of gripping arm tips</td>
<td>6 µm</td>
</tr>
<tr>
<td>width of plunger tip</td>
<td>8 µm</td>
</tr>
<tr>
<td>initial opening of gripping arms</td>
<td>17 µm</td>
</tr>
<tr>
<td>gap between opposing comb fingers (b)</td>
<td>4 µm</td>
</tr>
<tr>
<td>finger pair number of each gripping arm</td>
<td>280</td>
</tr>
<tr>
<td>finger pair number of plunger</td>
<td>540</td>
</tr>
<tr>
<td>width of all flexible beams</td>
<td>5 µm</td>
</tr>
<tr>
<td>length of flexible beams of gripping arms</td>
<td>580 µm</td>
</tr>
<tr>
<td>number of flexible beams of each gripping arm</td>
<td>2</td>
</tr>
<tr>
<td>length of flexible beams of plunger</td>
<td>630 µm</td>
</tr>
<tr>
<td>number of flexible beams of plunger</td>
<td>4</td>
</tr>
</tbody>
</table>

Figure 2.3: Microfabrication process.
Chapter 2. Pick-and-Place of Micro-Objects Using a MEMS Microgripper

Figure 2.4: Characterized microactuator performance.

...ger thickness, \( b \) is the gap between opposing comb fingers, and \( V_1 \) is the voltage applied to both gripping arms for creating a gap of the size of the micro-object. Note that when the initial grasping force applied to the micro-object is increased, the actuation voltage \( V_2 \) required for opening the gripping arms becomes smaller, resulting in a larger estimate of the adhesion forces. Despite the initial grasping force variations as well as microfabrication induced imperfections, the adhesion forces obtained through this actuation force estimation proved useful for understanding purposes.

2.3 Force Analysis

Adhesion forces in an ambient environment include three types of attractive forces, namely, the van der Waals force, the electrostatic force, and the capillary force, all of which depend on the separation distance, \( \delta \), between a microsphere and a flat surface it adheres to. Figure 2.5 shows a microsphere adhered to a flat surface with surface roughness exaggerated.

Van der Waals forces are caused by the instantaneous polarization of atoms and molecules due to quantum mechanical effects. The van der Waals force between a microsphere and a flat surface is [55]

\[
F_{vdw} = \left( \frac{\delta}{\delta + r/2} \right)^2 \left( \frac{Hd}{16\pi\delta^2} + \frac{H\rho^2}{8\pi\delta^3} \right),
\]

(2.2)

where \( r \) is the roughness of the flat surface, \( H \) is the Lifshitz-van der Waals constant which
ranges from 0.6 eV for polymers to 9.0 eV for metals, $d$ is the microsphere diameter, and $\rho$ is the radius of the adhesion surface area.

To estimate the van der Waals force between a 10 µm borosilicate microsphere and the sidewall of a gripping arm, $\delta$ is assumed to be 0.35 nm [54], $\rho$ is assumed to be 0.65% of the radius of the microsphere [54], $H$ is assumed to be 7.5 eV [54], and $r$ is assumed to be 100 nm. Thus, the van der Waals force is calculated to be $1.51 \times 10^{-4}$ µN.

The electrostatic force for microspheres smaller than 100 µm is predominantly the electrostatic double layer force, which is [56]

$$F_{\text{elec}} = \frac{\pi \varepsilon d U^2}{2\delta}, \quad (2.3)$$

where $\varepsilon$ is the permittivity of air, and $U$ is the voltage difference between the microsphere and the flat surface. When $U$ is assumed to be 0.40 V [54], the electrostatic force between a 10 µm microsphere and the sidewall of a gripping arm is calculated to be $6.36 \times 10^{-2}$ µN.

The third type of attractive force is the capillary force, which is composed of the capillary pressure force and surface tension force [57, 58]. The capillary pressure force dominates the surface tension force for microspheres larger than 1 µm [57]. For the microsphere-plane model, the capillary force is [59, 60],

$$F_{\text{cap}} = \frac{2\pi d y \cos \theta}{1 + \delta/(2r_K \cos \theta - \delta)}, \quad (2.4)$$
where $\gamma$ is the liquid surface tension, which is 0.073 Nm$^{-1}$ for water at 22°C, $\theta$ is the contact angle of the meniscus with the microsphere, and $r_K$ is the Kelvin radius, which is defined as the mean radius of the curvature of the liquid-vapor interface.

For estimating the capillary force exerted on a 10 $\mu$m microsphere by a water meniscus at room temperature, $\theta$ is assumed to be 10°, $\delta$ is still assumed to be 0.35 nm as for the calculation of the van der Waals force, and $r_K$ is assumed to be 1 nm. The capillary force is calculated to be 3.71 $\mu$N.

For comparison purposes, the gravity of the 10 $\mu$m microsphere is calculated to be $1.31 \times 10^{-5}$ $\mu$N, using the density of borosilicate glass, 2.55 g/cm$^3$. In summary, the pecking order is

$$F_{\text{cap}} \gg F_{\text{elec}} \gg F_{\text{vdw}} \gg F_{\text{grav}}.$$ (2.5)

It can be seen that the van der Waals force is the smallest among the three attractive forces. The van der Waals force heavily depends on the roughness of the surface. Since devices were formed through deep reactive ion etching, which produces scallop structures on the sidewalls of the gripping arms, the rough surface makes the van der Waals force negligible. The electrostatic force depends on voltage differences, which are difficult to accurately estimate when the microsphere is nonconductive. Unlike the van der Waals force and electrostatic force, neither of which requires physical contact, the capillary force in the air results from a phenomenon called capillary condensation [56]. Liquid from the vapor phase condenses between sufficiently close asperities and forms menisci that cause the capillary force. Thus, there exists a working range, beyond which the capillary force as well as the liquid menisci disappear.

Schematic diagrams in Figure 2.6 illustrate forces exerted on a microsphere by the gripping arms and/or the substrate during grasping and release. Figure 2.6(a)-(c) are drawn from the side view, and Figure 2.6(d)-(f) are from the top view. Figure 2.6(a) shows the microgripper approaches the microsphere and uses the gripping arm to laterally push it in order to break the adhesion bond between the microsphere and the substrate. $F_s$ is the adhesion forces, and $N_s$ is the normal force from the substrate. $N_r$ is the lateral pushing force applied by the right gripping arm, and $F_r$ is the adhesion forces from the gripping arm in the normal direction. Upon the
application of $N_r$, the stress distribution in the contact area between the microsphere and the substrate becomes nonuniform, which creates a rolling resistance moment, $M_s$ [61,62]. Besides the adhesion forces $F_s$ and $F_r$ that are normal to the flat surfaces, $f_s$ and $f_r$ are additional capillary forces from the substrate and the gripping arm, respectively. $f_s$ ($f_r$) resists the relative motion between the microsphere and the substrate (gripping arm), through the menisci. In this situation, the total capillary forces from the substrate and gripping arm are not perpendicular to the flat surfaces.

After the microsphere is moved laterally from its original position, the two gripping arms close and grasp it, as shown in Figure 2.6(b). The normal force and adhesion forces, $N_l$ and $F_l$,
are from the left gripping arm. Similarly, $N_r$ and $F_r$, are from the right gripping arm. Besides $F_l$ and $F_r$, there can also be additional capillary forces parallel to the substrate surface and gripping arm surface, although they are not shown in the diagram for clarity.

The microgripper is then raised, as shown in Figure 2.6(c) to lift up the microsphere. The additional capillary forces from the gripping arms, $f_l$ and $f_r$, overcome the adhesion forces from the substrate, $F_s$, which decreases gradually as a function of the distance between the microsphere and the substrate.

When the microsphere is up in the air (Figure 2.6(d)), the adhesion forces from the substrate become negligible. Upon reaching a desired destination, the gripping arms are opened, during which all of the adhesion forces and normal forces from the gripping arms decrease. Consequently, the microsphere separates from one gripping arm and keeps adhering to the other gripping arm by adhesion forces, as shown in Figure 2.6(e).

For release, the gripping arm with the adhered microsphere is properly positioned relative to the plunger, as shown in Figure 2.6(f). The plunger is then controlled to move forward to thrust out and collide with the microsphere. The microsphere eventually escapes from the adhesion forces from the gripping arm by its own inertia and lands on the substrate. In Figure 2.6(f), $N_p$ is the pushing force applied by the plunger, $F_p$ is the adhesion forces from the plunger, and $M_r$ and $f_r$ are respectively the rolling resistance moment and additional capillary force from the gripping arm.

### 2.4 Experimental Results and Discussion

The experimental setup (Figure 2.7) consists of an optical microscope (Motic PSM-1000) with a CMOS camera (Basler A601f). A custom-made circuit board with a wire bonded microgripper was mounted on a 3-DOF micromanipulator (Sutter MP285) at a tilting angle of 25°.

Borosilicate glass microspheres (diameters: 7.5–10.9 µm) were manipulated at room temperature of 22°C with relative humidity of 50±5%. A droplet of microspheres in isopropanol
was micropipetted onto the substrate and let dry in air. The surface tension of isopropanol (0.021 N/m at room temperature) is smaller than that of water. However, due to the volatility of isopropanol and because the microspheres were let dry in air for a prolonged period, water was assumed to constitute most of the liquid menisci between the microspheres and the substrate. Therefore, the surface tension of water was used in (2.4) in Section 2.3 for estimating the capillary force.

Two types of substrates, wipe cleaned with isopropanol and let dry in air, were used in the experiments, including an electrically conductive substrate (steel) and a non-conductive substrate (glass). These two substrates were expected to exert different electrostatic forces and van der Waals forces on the microsphere while it is traveling in air during release, which might affect the release accuracy.

### 2.4.1 Repeatability of Active Release

After the gripping arms opened, the microsphere randomly adhered to a gripping arm in all cases. The overall adhesion forces between the gripping arms and microsphere were estimated...
to be 3.6 µN to 5.8 µN through measuring actuation voltages required to open the gripping arms after a gentle yet secured grasping of a microsphere, as described in (2.1) in Section 2.3.

For successful release, the microsphere must gain a sufficient amount of momentum from the collision with the plunger in order to overcome the adhesion forces. The speed of the plunger can be varied by controlling the rising profile of the actuation voltage. When a sharp increase in actuation voltage was applied to the plunger (e.g., from 0 V to 50 V within 0.1 s), release of the microsphere was guaranteed (i.e., 100% success rate, \( n = 700 \)). A high plunging speed alleviates careful sample preparation requirements (e.g., baking) or environmental control requirements (e.g., humidity). Quantification using high-speed videography (13,000 frames/sec) revealed that a plunging speed of 65.24 mm/sec produced a microsphere speed of 105.01 mm/sec with a momentum of \( 1.40 \times 10^{-13} \text{ kg \cdot m/s} \). This plunging speed guaranteed the successful release for all trials. High-speed videography also demonstrated that a microsphere was separated from the plunger upon impact.

### 2.4.2 Quantification of Release Performance

To quantitatively characterize the release performance, single microspheres were repeatedly picked and released from different heights (2–30 µm) above the substrate. Figure 2.8(a) shows representative data of landing positions on a glass substrate. The results show a fairly linear and predictable relationship between landing positions and heights from the substrate, indicating that forces including the van der Waals forces and the electrostatic forces from both the substrate and the microgripper, as well as the gravitational force, do not have a significant effect on the high-speed microsphere that travels a short distance in air.

Figure 2.8(a) also shows that the precision of landing is inversely proportional to the height from the substrate. When the height was over 20 µm, random landing locations were observed, which should be partly due to the more pronounced air flow effect. To investigate the influence of substrate differences on release performance, experiments were also repeated using a steel substrate. Compared to data in Figure 2.8(a), results shown in Figure 2.8(b) confirm that the
active release approach does not have observable substrate dependence.

As mentioned earlier, adherence of the microsphere to which gripping arm is random. Figure 2.8(a)(b) show experimental data collected when the microspheres adhered to the right gripping arm. Similar data were captured but not shown for microspheres that adhered to the left gripping arm.

Given the above findings, the release height was set to 2 µm above the substrate for quantifying release accuracy. The small distance of 2 µm from the substrate reduces the distance/time the microsphere travels in air, making the landing location less sensitive to environmental disturbances. Figure 2.9 shows the recorded landing positions of the microsphere, proving an accuracy of 0.70±0.46 µm. A summary of the characterized release accuracy is given in Table 2.2. The 0.55 µm standard deviation of landing positions can be due to either (1) slight variations of initially adhered lateral and/or vertical positions of the microsphere on the gripping arm or (2) imperfect control of the microgripper height above the substrate.

By using an automated substrate contact detection method (Section 2.5.2) to accurately control the release height, the release accuracy was further improved to 0.45±0.24 µm.

Besides a high accuracy, the active release technique enables easy, fast pick-and-place op-
Figure 2.9: Landing positions of microspheres when the gripping arms were placed 2 µm above the substrate. (a)(b) Glass substrate. (c)(d) Steel substrate. (a)(c) Microspheres adhered to the left gripping arm. (b)(d) Microspheres adhered to the right gripping arm.

Operation in micromanipulation. Figure 2.10 shows the result of a series of pick and release of microspheres. While grasping was manually conducted, which is skill dependent, positioning the microsphere properly for plunging was rapid and took less than 1 s with the use of calibration results shown in Figure 2.4. The actual release takes 0.17 ms, according to high-speed videography.
Table 2.2: Summary of Release Accuracy

<table>
<thead>
<tr>
<th></th>
<th>glass substrate release accuracy</th>
<th>steel substrate release accuracy</th>
</tr>
</thead>
<tbody>
<tr>
<td>microspheres adhered to left arm</td>
<td>$0.70 \pm 0.46 \mu m$ $(n = 18)$</td>
<td>$0.67 \pm 0.55 \mu m$ $(n = 18)$</td>
</tr>
<tr>
<td>microspheres adhered to right arm</td>
<td>$0.64 \pm 0.46 \mu m$ $(n = 18)$</td>
<td>$0.67 \pm 0.55 \mu m$ $(n = 20)$</td>
</tr>
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2.4.3 Understanding the Curved Trajectory

Interestingly, it can be seen from Figure 2.8 that the microspheres all landed to the right/left side of the plunger (plunger was along the $y$ axis) depending on which gripping arm they adhered to. High-speed imaging verified that the flying path of the microsphere was indeed curved. Images shown in Figure 2.11 were taken through high-speed videography when the gripping arms were 20 $\mu m$ above the substrate before the release of the microsphere.

According to the brief force analysis in Section 2.3, the van der Waals force and electrostatic force decrease with increased distances between the microsphere and gripping arm. Additionally, the capillary force vanishes beyond a certain distance. Thus, it is assumed that the gripping arm has an adhesion force effective region around it, as indicated by dashed lines in Figure 2.12.

During release, the plunger first impacts the microsphere along the sidewall of the gripping arm at a high speed as shown in Figure 2.12(a), where the dotted lines represent the adhesion force effective region. When the traveling microsphere approaches the gripping arm corner, which was rounded by deep reactive ion etching, the adhesion forces create a radial acceleration towards the corner, which curves its travel direction. While the microsphere is within the adhesion force effective region, there exists resistance $f_r$ (additional capillary force) in the tangential direction caused by menisci. Eventually, the microsphere leaves the gripping arm...
Figure 2.10: Pick-and-place to align microspheres (7.5 µm to 10.9 µm). Note that the microgripper was titled 25°. (a) The microgripper approaches a microsphere, and uses one gripping arm to laterally push it to break the initial adhesion bond between the microsphere and the substrate. (b) Two gripping arms are closed, grasping the microsphere and lifting it up. (c) The microsphere is transported to the target area where some microspheres have already been aligned. (d) The gripping arms are opened and the gripping arm that the microsphere adheres to positions the microsphere properly to the right position in relation to the plunger. (e) The plunger thrusts out the microsphere that lands accurately on the substrate. (f) Microgripper retracts to repeat the pick-and-place process.

tip and hence the adhesion force effective region. It then travels straightly and lands on the substrate, as depicted in Figure 2.12(b).
Figure 2.11: High-speed videography (13,000 frames/second) quantifying microsphere trajectories upon release from a height of 20 μm above the substrate.

2.5 Microrobotic Pick-and-Place of Microspheres

2.5.1 Recognition of Microgripper and Microspheres

The microspheres on the substrate were recognized using a Hough transform to determine their centers and radii. Contours formed from Canny edge detection readily recognize the gripping arms and the plunger. As shown in Figure 2.13(a), M₁, M₂, and M₃ denote the centroids of the two gripping arms and the plunger. By comparing the y coordinates of their centroids, the left gripping arm, right gripping arm, and plunger were distinguished.

Minimum bound rectangles (MBRs) were used to further define the positions of the two gripping arms, as shown in Figure 2.13(a). Point D was then taken as the overall position of the microgripper, which is the intersection of the horizontal line going through the plunger centroid, M₃, and the line connecting the left adjacent corners of the top and bottom MBRs.

To attain secured grasping, the system aligns the grasping position of the gripping arms with respect to a microsphere, as illustrated in Figure 2.13(b) where \( g \) is the width of the gripping arm (denoted by \( k \) in Figure 2.13(a)). \( r \) is the radius of the microsphere. The contact position of the gripping arm with the microsphere is on the segment AB. In particular, the middle position
Figure 2.12: Microsphere reveals a curved trajectory during active release. (a) The plunger thrusts the microsphere that reaches the roundish corner of the gripping arm. (b) The microsphere escapes from the effective range of the adhesion forces. The trajectory is drawn under the assumption that there are no disturbances when the microsphere is in the air.

C provides the most security for grasping when microspheres slide during grasping (Figure 2.13(b)). According to the geometry, the distance from the microgripper position D to the optimal grasping position C is 

\[ l = t \sin \alpha + \frac{r}{2} \cos \alpha - r \cot \alpha, \]

which is a function of the size of the microsphere to be grasped.

When the gripping arms open, the microsphere randomly adheres to one of the two gripping arms. As shown in Figure 2.14, the boundary of the gripping arm to which the microsphere adheres is connected with that of the plunger. Thus, only two contours are detected with the larger contour containing the microsphere. By comparing the \( y \) coordinates of the centroids of the contours (\( M_1 \) and \( M_2 \) in Figure 2.14), the system determines to which gripping arm the microsphere adheres.

### 2.5.2 Contact Detection and Microrobotic Control

Knowledge of relative depth positions of the gripping arms and microsphere is gained through the detection of the contact between the gripping arms and the surface of the substrate. Ob-
Figure 2.13: (a) Recognized gripping arms and plunger. (b) Sidewall of a gripping arm for determining the secured grasping position, C. (c) 3D schematic showing the grasping of a microsphere.

viating the need for additional force/touch sensors, the system employs a vision-based contact detection algorithm [63] that provides a detection accuracy of 0.2 µm. The contact detection process completes within 5–8 seconds.

The microgripper was controlled to move downwards at a constant speed (e.g., 20µm/sec) to establish contact with the substrate while the algorithm ran in real time. Since further lowering the gripping arms after the contact is established causes the gripping arms to slide on the substrate (Figure 3.3), monitoring the $x$ coordinates of the gripping arms result in a V-shaped curve, as shown in Figure 2.16. The global minimum represents the initial contact of the microgripper with the substrate.

The microrobotic system is a “looking-and-moving” system. Transformation between the image frame ($x$-$y$) and the microrobot frame ($X$-$Y$) was achieved with calibrated pixel sizes.
Figure 2.14: Visual determination of which gripping arm the microsphere adheres to after the gripping arms open.

Figure 2.15: Vision-based contact detection. Gripping arms slide on the substrate after contact is established.

Figure 2.16: Contact detection by monitoring $x$ coordinate of a gripping arm in the image while lowering the microgripper at a speed of 20 $\mu$m/sec.
2.5.3 Automated Pick-and-Place of Microspheres

To quantify the operation speed of the microrobotic system, microspheres were picked and placed to form patterns. The system starts with the contact detection to determine the depth position of the gripping arms relative to the substrate surface. The microgripper was then moved upwards by 15 µm above the substrate, ready for the pick-and-place operation.

Microspheres in the field of view were visually recognized. Their positions in the image frame, sizes, and optimal grasping positions were determined. Then, by using the contact detection result and coordinate transformation, the target X-Y-Z positions were determined by the system. The microspheres were picked up from the source area in the order of their x coordinates in the image frame. According to the actuation calibration results (Figure 2.4), the system determined actuation voltages for the gripping arms for secured grasping while ensuring no excessively large actuation voltages were applied.

The microrobot lifted the securely grasped microsphere to 15 µm above the substrate. When a pre-planned target position was reached, the microrobot moved downwards and stopped at 2 µm above the substrate for release. The gripping arm to which the microsphere adhered was first visually detected and then aligned the microsphere accurately in front of the plunger based
Figure 2.18: Pattern formation by automated pick-and-place. (a) Microspheres before pick-and-place. (b) A circular pattern with circularity of 0.52 µm.

Figure 2.19: Microspheres assembled into two-layered structures.

on the calibration results shown in Figure 2.4. The plunger was then actuated to release the microsphere, after which the microgripper was raised 15 µm above the substrate and return to the source area for picking up the next microsphere. Figure 2.18 shows that microspheres were arranged into a circular pattern with a circularity of 0.52 µm, which is defined as the standard deviation of the distances from the microspheres to the center of the circle. Figure 2.17 shows an assembled “U OF T” pattern. The average pick-and-place speed was 10 spheres/min.

2.5.4 Three-Dimensional Assembly of Microspheres

The technique can be extended to building three-dimensional structures (e.g., Figure 2.19). The difficulty involved in such tasks is that the microgripper tips, when positioning a microsphere
for release, may collide with other microspheres in close proximity. To overcome this difficulty, a rotational degree of freedom is required in the system, either for the substrate and thus the microspheres, or for the microgripper to avoid collision between the microgripper tips and microspheres.

2.6 Conclusions

This chapter presented a new MEMS microgripper that integrates both gripping and release mechanisms. The microgripper was applied to the grasping and active release of microspheres. The plunger provides the microsphere with sufficient momentum to overcome adhesion forces, resulting in highly repeatable release (100% of 700 trials) and a release accuracy of 0.45±0.24 μm. The tested borosilicate microspheres varied from 7.5 μm to 10.9 μm in size. Within this size range, release accuracy was found independent of microsphere sizes. Release performance was also found independent of electrical conductivity of substrates (steel and glass). Considering structural dimensions of the present device (e.g., thickness of gripping arms and plunger: 25 μm and initial gripping arm opening: 17 μm), we speculate that the reported release accuracy should be consistent for microspheres ranging from a few micrometers up to 17 μm. This study revealed that the most important operating parameters are plunging speed and the height from the substrate. The highly controllable active release capability represents an important progress for reliable pick-and-place micromanipulation.

Enabled by this releasing technique, an automated robotic pick-and-place system was realized using vision-based techniques for the recognition of the microgripper and microspheres, determining the height of the microgripper above the substrate, and motion control of the micromanipulator. The system demonstrated a pick-and-place speed of 6 spheres/min, which is much faster than a skilled operator and an order of magnitude faster than the highest speed reported in the literature thus far. Three-dimensional structures were also built with microspheres, demonstrating the capability of three-dimensional assembly.
Chapter 3

Automated Four-Point Probe Measurement of Nanowires in SEM

3.1 Introduction

Using electron microscopy as an imaging platform, nanomanipulation inside a scanning electron microscope (SEM) has been employed to maneuver and characterize the properties of nanomaterials. For instance, carbon nanotubes, InGaAs/GaAs nanosprings, and silicon nanowires were deformed via a nanomanipulator inside an SEM to characterize their mechanical and/or electrical properties [10–13, 64, 65]. Through nanomanipulation, nanomaterials were also placed on MEMS devices for tensile testing [66–70].

To date, SEM nanomanipulation has largely been performed manually by an operator using a joystick and/or a keypad while constantly monitoring SEM images, which is time-consuming, skill-dependent, and often breaks end-effectors. As this technology becomes increasingly relevant for device prototyping [11, 71–75] as well as the aforementioned material testing at the nanometer scale, progress is being made toward automated nanomanipulation in order to achieve high reliability, efficiency, and repeatability.

Existing nanomanipulators are driven by piezoelectric actuators and usually have only three
translational degrees of freedom. Because of creep, drift, and hysteresis of piezoelectric actu-
ators, open-loop control cannot suffice in precision for automatic nanomanipulation tasks, ne-
cessitating feedback control. Integrating position sensors (e.g., optical encoders or capacitive
sensors) appears to be a straightforward solution and has been achieved for a few commercial
nanomanipulators manufactured by, for example, SmarAct GmbH and Attocube Systems AG.
However, the integration of high-resolution encoders increases the cost significantly; furth-
more, sensor drift can be significant at the nanometer scale. Thus, visual feedback from SEM
becomes essential for closed-loop control of nanomanipulators with/without encoders.

To visually obtain the $XY$ position of an end-effector, visual tracking of the end-effector
in a sequence of SEM images has been implemented using feature-based methods [76, 77] or
correlation-based methods [76, 78]. For example, a rigid-model-based visual tracking method
was reported, which applies domain-specific constraints and was evaluated by tracking a mi-
crogripper inside an SEM [78]. Based on SEM visual tracking, visual servo control can be
realized to control the in-plane position of the nanomanipulator, which, however, has not been
reported in the literature.

Besides position control along $XY$, precise positioning along the $z$ axis is also essential but
more challenging since it is difficult to extract depth information from SEM visual feedback. To
address this issue, a few techniques were proposed. The depth-from-focus method commonly
used under an optical microscope was extended to SEM for a coarse estimate of the $Z$ position
of an end-effector [79]. A touch sensor based on piezoelectric ceramics [79] and a shadow-
based depth detection method [80, 81] were used for a fine estimate of the $Z$ position. MEMS
capacitive sensors for contact detection under optical microscopes [82] can also be used inside
an SEM. However, the precisions of these methods have not been quantified.

In addition, stereoscopic SEM images can be generated by tilting the electron beam [83],
which requires the installation of specialized hardware and needs more thorough studies in
order to be used for automated nanomanipulation. Tilting the specimen stage can also be
used for SEM stereoscopy [84], but has not been evaluated for nanomanipulation purposes. In
summary, a convenient technique is needed which is capable of precisely estimating Z positions or detecting contact between an end-effector and a substrate.

In this chapter, automated nanomanipulation is demonstrated inside an SEM by performing a well-structured nanomanipulation task for probing the electrical properties of individual nanowires. Four-point probe measurement of nanomaterials has been reported using scanning tunneling microscopy inside an SEM [85–87], using four-probe devices [88–91], and using focused ion beam deposition or electron beam lithography to pattern electrodes [92, 93]. Nonetheless, existing techniques require expensive equipment and lack flexibility; moreover, they all entail tedious trial-and-error manual operation.

The nanomanipulation system controls four nanomanipulators inside an SEM to realize automated four-point probe measurement on individual nanowires lying on a substrate. Nanowires as well as nanoprobes installed on the nanomanipulators are visually recognized from SEM imaging. The XY positions of the nanoprobes are controlled via look-then-move control followed by visual servo control. Their contact with the substrate is also detected via visual feedback. Current–voltage (I–V) data of tin oxide nanowires are obtained after the nanoprobes positioned on the nanowires.

### 3.2 Automated Nanomanipulation inside SEM

#### 3.2.1 Nanomanipulation System

A nanomanipulation system (Zyvex S100) is integrated into an SEM (Hitachi S-4000) by mounting its head assembly onto the specimen stage of the SEM. The head assembly, as shown in Figure 3.1, is composed of four quadrants of 3-DOF nanomanipulators, each of which is composed of a coarse positioning stage and a fine positioning unit. The coarse positioning stage contains three identical piezoelectric slip-stick motors, each of which has a travel of 12 mm with 100 nm resolution. The fine positioning unit contains a piezoelectric tube having travel ranges of 10 μm along the axis of the tube and 100 μm along each of the two transverse
directions with 5 nm nominal resolution.

The nanomanipulators do not have integrated position sensors for either coarse or fine positioning, as in most commercial and academic nanomanipulation systems. In Figure 3.1, each nanomanipulator has a tungsten probe installed as an end-effector for interacting with the specimen placed at the center of the specimen stage. The probes have direct electrical connections to an interface located outside the SEM for applying or measuring electrical signals. Prior to loading the probes into the nanomanipulators, the probes are chemically cleaned to remove the native tungsten oxide using KOH solutions and HF. After the cleaning procedure, the probe tips are 150–200 nm in diameter.

SnO$_2$ nanowires being probed in this chapter are synthesized for use as anode materials in Li-ion batteries. The nanowires are prepared by the CVD (chemical vapor deposition) method in a horizontal quartz tube. Sn powder is chosen as the starting material and is loaded in a ceramic boat. The ceramic boat is placed in the center of a quartz tube mounted in an electric furnace. A silicon wafer with a Au film of 5 nm thick is placed close to the starting powder. An Ar flow is introduced into the furnace as the gas carrier. The furnace is heated up to 700 °C, which is maintained for two hours. Finally, the furnace is cooled down to the room temperature under the Ar atmosphere. The nanowires are scratch-removed and placed on a SiO$_2$-covered silicon substrate. Consequently, the nanowires lie directly on the substrate surface, which
Figure 3.2: Visual recognition of probes and nanowires from SEM visual feedback. (a) Four probes and nanowires. (b) Probes and nanowires are recognized from image processing.

constitutes a well-structured manipulation condition and makes automated probing possible.

### 3.2.2 Recognition of Probes and Nanowires

When the four probes and one or multiple nanowires are in the field of view [Figure 3.2(a)], they are visually recognized and identified [Figure 3.2(b)]. The contours of the objects in the image are recognized through a sequence of low-pass Gaussian filtering, adaptive thresholding, and morphological operations. The probes and nanowires are distinguished by comparing areas surrounded by the contours, since nanowires have smaller areas. The four probes are distinguished from each other by comparing the positions of the centroids of their contours, and the positions of their tips are determined as either the highest or lowest point of a probe’s contour. The target positions on the nanowire for probing are determined relative to the positions of the rightmost and leftmost points of the nanowire’s contour, according to desired separations between the probe tips during measurements.
3.2.3 Feature Tracking

Visual tracking provides motion/position information of the probes in the image frame as position feedback for vision-based contact detection and visual servo control. The sum-of-squared-differences (SSD) algorithm is employed to track the tip of a probe. The system first conducts visual recognition in a frame of image $I_1$ to obtain the coordinate $(x_1, y_1)$ of the probe tip. A rectangular patch of the image containing the probe tip is recorded as a template. In subsequent frames, the SSD measure is calculated for each possible displacement $(\Delta x, \Delta y)$ within a search window. Specifically, in the $k$th frame,

$$SSD(\Delta x, \Delta y) = \sum_{i,j \in N} [I_k(x_{k-1} + \Delta x + i, y_{k-1} + \Delta y + j) - I_{k-1}(x_{k-1} + i, y_{k-1} + j)]^2,$$

(3.1)

where $(x_{k-1}, y_{k-1})$ is the coordinate of the probe tip in the $(k - 1)$th frame of image $I_{k-1}$. The displacement $(\Delta x, \Delta y)$ producing the minimum SSD value is considered to be the displacement of the probe. The system simultaneously tracks all the four probes using the SSD algorithm.

To achieve a subpixel tracking resolution, eight neighboring pixels as well as the selected pixel are used to fit a curved surface in terms of their SSD values. The pixel coordinate corresponding to the valley of the curved surface is used to determine the displacement of the feature [94].

3.2.4 Vision-Based Contact Detection

Manipulation of a nanoobject lying on a substrate requires knowledge on relative vertical positions between the end-effector and the object/substrate. When a probe is moved downwards, the system detects the contact between the probe tip and the substrate via a vision-based contact detection method, which was extended from a previous method we developed for optical microscopy [63]. This method makes use of the phenomenon that a downward-moving probe slides on the surface of the substrate after contact is established, as illustrated in Figure 3.3.

Inside an SEM, when a nanomanipulator moves a probe along its $z$ axis to approach a substrate, the position of the probe in the image frame also moves along a certain direction, due
Figure 3.3: Principle of vision-based contact detection: probe slides on the substrate after contact establishment.

to the perspective projection model of the SEM [78]. When the probe contacts the substrate and begins to slide, an abrupt shift in the moving direction of the probe in the image frame is recognized as the contact point. Therefore, by monitoring the occurrence of this phenomenon, visually tracking a downward-moving probe is capable of detecting the contact between the probe and the substrate.

### 3.2.5 Look-then-Move Control and Visual Servo Control

To achieve closed-loop positioning of a nanomanipulator in the XY plane, visual tracking of the end-effector is used to provide position feedback for a visual servo controller. Visual feedback from SEM has a low sampling frequency (typically <15 frames per second (fps)). This issue was previously addressed by selectively scanning a smaller region of interest [77, 78]. This approach, however, requires the installation of new hardware for accessing the scan controller of the SEM.

To quicken the system response, this thesis uses open-loop control [Figure 3.4(a)] to quickly bring the probe to the vicinity of the target position, without the reliance on visual feedback. Subsequently, visual servo control [Figure 3.4(b)] is used to bring the probe precisely to the target position. For the open-loop control, since piezo actuators have hysteresis, a mathematical model (denoted by $H$) of piezo actuators is used that takes into account the effect of hysteresis. $H$ is a static model that relates the actuation voltage to the output displacement.
Figure 3.4: SEM vision-based control. (a) Look-then-move control. It quickly brings the probe to the vicinity of the target position. (b) Image-based visual servo control. It brings the probe precisely to the target position.

To use the model, the piezo actuator is characterized first, as shown in Figure 3.5. A voltage is incrementally applied to an actuator and then incrementally released with the corresponding displacements in the image frame recorded by the visual tracking algorithm. Figure 3.5 shows the characterization results of one piezo actuator at a magnification of 3000×. The applied voltage is denoted by $u$, with its lowest and highest values denoted by $U_0$ and $U_n$. The ascending and descending curves are denoted by functions $P_n(u)$ and $D_n(u)$, and are respectively fitted with a fourth-order polynomial. If the piezo actuator is at any point on the ascending (descending) curve, and the applied voltage is monotonically increasing (decreasing), the output displacement is $P_n(u)$ ($D_n(u)$).

However, if the applied voltage starts to decrease (increase) when the actuator is on the ascending (descending) curve, the actuator will stray away from that curve due to hysteresis. In that case, a mathematical model is used to calculate the output displacement. When the applied voltage increases from point $(u_1, D_n(u_1))$ on the descending curve [Figure 3.5], the output displacement (dashed curve in Figure 3.5) is [95]

$$ P_1(u) = k \cdot P_n(m \cdot (u - U_n) + U_n) + D_n(U_1) - k \cdot P_n(U_0), \quad (3.2) $$

where $k = (D_n(U_n) - D_n(u_1))/(D_n(U_n) - D_n(U_0))$ and $m = (U_n - u_1)/(U_n - U_0)$.

When the actuator is on the descending curve and a certain displacement is desired, the
corresponding applied voltage is calculated via the Newton-Raphson method to invert \( D_n(u) \) or the mathematical model (3.2), as the output of the inverse model \( H^{-1} \) in Figure 3.4(a). A similar mathematical model is used for the scenario where the applied voltage starts to decrease while the actuator is on the ascending curve to calculate the output of the inverse model.

### 3.2.6 Overall Process of Automated Nanomanipulation

The four probes are first brought into the field of view under a proper magnification using the coarse positioning stages, after which only the fine positioning units are controlled to move the probes. The probes and nanowires are visually recognized. The longest nanowire is selected for testing, and four target positions on the nanowire are determined. The system moves the probes downwards to establish their contact with the substrate through vision-based contact detection. After contact detection, the probes are positioned at a certain height (e.g., 200 nm) above the substrate, ready for subsequent in-plane movement.

The four piezoelectric tubes are actuated in their respective \( x \) and \( y \) axes to obtain parameters for their mathematical models to be used in the look-then-move controllers. Through look-then-move control followed by visual servo control, the probes are simultaneously moved toward their target positions on the nanowire [Figure 3.6]. Then the probes move downwards
Figure 3.6: Four probes simultaneously move to their target positions on a nanowire by vision-based control.

to land on the nanowire for measurements. The separation distance between the inner pair of probes can be automatically adjusted by repositioning the probes for measuring the electrical properties of different lengths of the nanowire.

Aside from the sharp probes used in this thesis, probes with flat tips of hundreds of nanometers in width can also be microfabricated and used for probing nanowires. Despite higher costs, they can provide more secure contact with nanowires. For installation, their orientation should be carefully adjusted to ensure they are parallel to the substrate surface.

### 3.3 Experimental Results and Discussions

#### 3.3.1 Manual Operation

For comparisons between automated nanomanipulation and manual operation, a nanomanipulator was manually controlled to perform the task. An experienced operator used a joystick and/or a program via mouse clicking to control actuation voltages for a piezoelectric tube while monitoring the imaging screen. When approaching and contacting the substrate, the probe tip is often bent [Figure 3.7] before the operator realizes that the contact has been established since it is difficult for a human operator to promptly perceive the initial contact between the probe
Figure 3.7: Manual operation often causes probe tip damage and sometimes inadvertently severs a nanowire.

Aside from probe damage, manually controlling a probe to probe a nanowire is also time consuming and can be destructive to the nanowire. Bringing the probe tip to a desired location takes much time since the x and y motions must be carefully coordinated. When the probe contacts and then is lifted off the nanowire, the probe sometimes inadvertently severs the nanowire, as shown in Figure 3.7, since the contact between the probe tip and the nanowire is difficult to accurately determine by naked eyes observing noisy SEM imaging.

### 3.3.2 Evaluation of Drift and Noise in SEM Imaging

Drift and noise are inherently present in SEM imaging in the fast scanning mode, both of which affect the performance of visual tracking. SEM image noise refers to random variations of pixel values and arises from a few sources. Image drift refers to the movement of the entire image, such as from electron beam drift, charge drift on a specimen, and/or electromagnetic interferences from the environment.

In order to quantify the magnitudes of image drift and noise, the subpixel visual tracking method was used to track stationary features on a substrate. TABLE 3.1 summarizes standard
deviations for different magnifications at the highest scanning rate of 13 fps. Besides showing the combinatorial effect of image drift and noise, TABLE 3.1 also shows the effect of image noise alone, which was obtained by simultaneously tracking two stationary features in the image to remove the effect of image drift. It can be seen from TABLE 3.1 that at higher magnifications, image drift and noise are more significant and are generally smaller along the y direction than along the x direction.

Since the effect of image drift can be eliminated, image noise is the primary source of errors in visual tracking and automated nanomanipulation, and therefore, of primary concern. In TABLE 3.1, the image noise at the magnification of 3000× is approximately 0.68 pixel along the x axis, which corresponds to 45 nm considering the pixel size of 66 nm/pixel. It can also be observed from TABLE 3.1 that although a higher magnification generally gives rise to higher image noise in pixels, the noise level in terms of nanometers is actually reduced at increasing magnifications. Thus, for manipulating nanoobjects smaller than 45 nm, a higher magnification than 3,000× is necessary. Since the nanowires in this study have diameters ranging from 70 nm to 100 nm, the 3,000× magnification was sufficient in terms of tracking resolution and hence, automated probing requirements.

While image noise can be reduced by choosing slow scanning modes of the SEM, lower frame rates are inadequate for automated nanomanipulation relying on the SEM visual feed-

<table>
<thead>
<tr>
<th>magnification (pixel size)</th>
<th>30,000× (6.6 nm)</th>
<th>10,000× (20 nm)</th>
<th>5,000× (40 nm)</th>
<th>3,000× (66 nm)</th>
<th>1,500× (133 nm)</th>
<th>600× (330 nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>drift and noise (pixel)</td>
<td>x 4.46</td>
<td>2.97</td>
<td>2.20</td>
<td>1.23</td>
<td>0.77</td>
<td>0.65</td>
</tr>
<tr>
<td></td>
<td>y 4.42</td>
<td>1.56</td>
<td>1.38</td>
<td>1.14</td>
<td>0.68</td>
<td>0.69</td>
</tr>
<tr>
<td>noise (pixel)</td>
<td>x 1.27</td>
<td>0.97</td>
<td>0.72</td>
<td>0.68</td>
<td>0.77</td>
<td>0.60</td>
</tr>
<tr>
<td></td>
<td>y 0.96</td>
<td>0.76</td>
<td>0.49</td>
<td>0.40</td>
<td>0.41</td>
<td>0.49</td>
</tr>
</tbody>
</table>
3.3.3 Contact Detection Results

Contact detection was performed in an area of the substrate where there were no nanowires or contaminants below the probe to ensure contact detection accuracy. The system moved a probe downwards at a constant speed. Meanwhile, the position of the probe tip in the image frame along its moving direction was monitored using visual tracking. As an example, Figure 3.8 shows the results of automated contact detection and subsequent Z-positioning for one of the piezoelectric tubes via both pixel and subpixel tracking methods.

Each of the two data curves in Figure 3.8 is comprised of five characteristic segments, representing five different stages of the process. First, in segment $AB$, the pixel coordinate of the probe tip in the image frame increases, corresponding to the stage that the probe tip approaches the substrate prior to contact. Second, in segment $BC$, the pixel coordinate quickly decreases after point $B$, which represents that the probe is sliding forward on the substrate surface after contact. At point $C$, contact is determined by the system to have occurred since the pixel coordinate has decreased by five pixels, which is the preset threshold in consideration.
of the image noise level. Thereby at point C the probe is stopped from descending, and the initial contact point is determined to be point B.

Third, in segment CD, the pixel coordinate increases again, which corresponds to the stage that the probe tip is sliding back as the probe is being raised. Fourth, in segment DE, the pixel coordinate decreases, which represents that the probe tip is ascending after having been lifted off the substrate. Fifth, in segment EF, the pixel coordinate remains unchanged within a margin of 0.6 pixel, which represents that the probe is positioned at a height above the substrate. By comparing the two curves in Figure 3.8, it can be seen that the subpixel tracking method demonstrates a superior resolution for processing noisy SEM images.

### 3.3.4 Performance of Vision-Based Control System

A step signal (the desired displacement in pixels) was input to the control system. The responses of the system to a 100-pixel-step input with and without using the open-loop control first are shown in Figure 3.9. The use of the open-loop control shortens the rise time from 3.51 s to 0.44 s, and the settling time from 5.23 s to 0.61 s. The XY positioning accuracy was determined to be 42 nm at 3000× magnification. The much faster response is because the open-loop
control incorporates the mathematical model of the piezoelectric actuator and does not rely on any feedback. Low-frame-rate SEM visual feedback is only used after the probe tip is within the vicinity of the target position, for fine positioning.

3.3.5 Four-Point Probe Measurement Results

A source-measure instrument (Keithley System SourceMeter 2602) was connected to the four probes through its two channels (a current source and a voltmeter). After contact detection, the probes were positioned at approximately 200 nm above the substrate, with their corresponding $xy$ coordinate changes (vs. the $xy$ coordinate when a probe is in initial contact with the substrate and when a probe is 50 nm above the substrate) in the image frame recorded.

The system then horizontally servoed the probes to their target positions on a nanowire, taking into account the pixel coordinate changes of the probes from the height of 200 nm to the target height of 50 nm above the substrate. A target height of 50 nm above the substrate was chosen to ensure secured pressing on our nanowires that range from 70 nm -100 nm in diameter. After the probes landed on the nanowire, as shown in Figure 3.10, a current sweep was applied to the outer pair of probes (probe 1 and probe 3), and the inner pair of probes (probe 2 and probe 4) measured the resultant voltages. To quantify the repeatability of this technique, the entire automation process from contact detection to the final $I-V$ measurement.
Figure 3.11: Four-point probe measurement results of a nanowire. (a) $I$–$V$ data of a nanowire with regard to different separations between the two inner probes. (b) Separation-resistance relationship.

was attempted 50 times on 5 nanowires with 100% success rate.

The $I$–$V$ data from a nanowire (83 nm in width) is shown in Figure 3.11 (a). The five data curves correspond to five different separations between the two inner probes. The five corresponding resistance values are proportional to the separations (i.e., the portions of the nanowires between two inner probe tips), as shown in Figure 3.11 (b). Assuming the nanowire has a circular cross section, the resistivity was determined to be $9.88 \times 10^{-4} \ \Omega \cdot \text{m}$. When a higher current was applied, and the nanowire finally failed when the current reached 7.4 $\mu$A.
The breakdown current density of the nanowire was calculated to be $1.36 \times 10^9$ A/m$^2$.

### 3.4 Conclusions

This chapter demonstrated automated nanoprobing using SEM as a vision sensor. A method for vision-based contact detection was developed to detect the contact between a probe and a substrate for determining relative vertical positions of the probe tip and a nanoobject to be manipulated. A visual servo control system was built for closed-loop control of multiple nanomanipulators, following the open-loop control that brought the probe to the vicinity of the target position. Four-point probe measurements were conducted on individual tin oxide nanowires lying on a substrate. This technique can also be useful for electrical testing of nanostructures, such as nanodevices comprised of multiple nanoelectrodes and one-dimensional nanomaterials.
Chapter 4

Piezoresistivity Characterization of Nanowires using a MEMS Device

4.1 Introduction

Characterization of nanomaterials is important for understanding their properties and exploring their applications. Among a range of properties of nanomaterials, the piezoresistive effect is of interest because of its potential utilization in electromechanical sensors and strain engineering for nanoelectronics applications. For instance, individual single-walled carbon nanotubes were used as active transducer elements in a pressure sensor [96] and a displacement sensor [97].

To quantify the piezoresistivity of individual nanomaterials, a number of experimental techniques have been reported. The experiments in common require simultaneous mechanical loading and electrical measurement of a nanomaterial. For example, the cantilever tip of AFM was used to laterally push a single-walled carbon nanotube suspended over a trench between two solid electrodes [98], which results in large local deformation of the nanotube at the tube-tip contact point. To achieve more uniform tensile stretching, carbon nanotubes were suspended between a solid terrace and a suspended beam, which was pushed downwards by an AFM cantilever [99]. This scheme also results in large local deformation of carbon nanotubes at the
edges of the terrace and suspended beam, which undesirably contributes to the characterization results.

Uniaxial or almost uniaxial tensile strains were also produced. The four-point bending method was used to deform silicon nanowires that were epitaxially grown across a trench, producing low level uniform strains on the order of $10^{-4}$ [100]. To produce severe strains, silicon nanowires were grown between a silicon pad and a cantilever beam, which was pushed by a probe along the longitudinal direction of the nanowire [13]. A single-walled carbon nanotube was adsorbed on top of a membrane and was electromechanically characterized through bulge testing [96]. A silicon nanowire was embedded close to the anchor of a cantilever, which was pushed down by a stylus [101]. Individual InGaAs/GaAs nanosprings were stretched by two nanomanipulators inside a scanning electron microscope (SEM) for piezoresistivity characterization [102].

Besides the aforementioned methods, MEMS actuators are capable of delivering adequate motion resolutions and ranges for deforming nanomaterials, a number of MEMS devices were developed for mechanical characterization of nanomaterials [66–70, 103–113]. For example, electrostatic actuators [66, 70, 105] and electrothermal actuators [105, 111] were utilized to stretch nanomaterial specimens. MEMS capacitive sensors were incorporated into some of the MEMS devices [67, 70] to measure tensile forces of the specimen. The elongation measurements of the nanomaterial were obtained via SEM imaging.

While most of existing MEMS devices are only capable of mechanical characterization of nanomaterials, two of them have electrical testing capabilities [107, 108]. However, the device reported in [107] can only characterize a pair of nanowires in conjunction, rather than individual nanowires, because the two electrical terminals are both on the stationary portion of the device. The device reported in [108] contains an electrothermal actuator, which causes temperature increase in the specimen and affects nanomaterial characterization results. Additionally, force sensing mechanisms of both devices [107, 108] are coupled with actuators, which can introduce large errors in comparison with the use of an independent force sensor.
This chapter reports on a MEMS device and experimental techniques for the electrical and mechanical characterization of individual one-dimensional nanomaterials. Different from previous MEMS tensile-testing devices, this device is capable of acquiring both force and elongation data of a nanomaterial specimen electronically without relying on SEM imaging. In addition, since this MEMS device is capable of performing electrical measurements on a nanomaterial under controllable mechanical strains, the piezoresistive property of the nanomaterial is able to be characterized. The effect of electron beam (e-beam) irradiation on the characterization results is also presented. Electrical insulation on the suspended structures was created through a microfabrication process, enabling the electrical measurement of a nanomaterial. Individual silicon nanowires were integrated to the MEMS device via pick-and-place nanomanipulation inside an SEM, and characterized for their mechanical and piezoresistive properties.

4.2 Device Design and Analysis

4.2.1 Device Design

As schematically illustrated in Figure 4.1(a)(b), the device is composed of two suspended shuttles, namely, the actuator shuttle (on the left) and the force sensor shuttle (on the right), with a small gap in between to be bridged by a nanomaterial specimen. The actuator shuttle includes an electrostatic actuator and a capacitive displacement sensor (lateral comb-drive configuration) that measures displacements of the actuator. The force sensor shuttle contains a capacitive force sensor (transverse-comb configuration), which measures tensile forces of the specimen as well as its own displacement. When the actuator shuttle moves leftward, the specimen is stretched and the force sensor shuttle is also pulled leftward. Thus, the amount of specimen elongation is the displacement of the actuator subtracted by that of the force sensor.

This electronic method of elongation measurement has advantages over electron microscopy (EM) imaging as used in previous MEMS-based nanomaterial testing devices. First, it offers a
Fourth, the specimen can be characterized outside the EM vacuum chamber, making it easier

Figure 4.1: Schematics of the MEMS device for electromechanical characterization. Force and
displacement data of the nanomaterial are both acquired electronically, obviating the reliance
on electron microscopy imaging. Electrical insulation within suspended structures enables
simultaneous electrical characterization during tensile testing. (a) Planar schematic showing
device compositions. (b) 3D model of the device.

higher sampling rate than EM imaging (EM typically has a rate of 13 frames per second in fast
scanning mode). Thus, data points during rapidly developing events (e.g., plastic deformation
and failure) can possibly be captured. Second, EM imaging can be relieved from observing the
entire nanomaterial, and hence, can be used to focus on a section of the nanomaterial with a
higher magnification (e.g., for in situ study of deformation mechanisms). Third, EM imaging
affects electromechanical characterization, owing to e-beam irradiation. We experimentally
verified the e-beam irradiation effect, which will be discussed quantitatively in Section 4.4.4.
Fourth, the specimen can be characterized outside the EM vacuum chamber, making it easier
to study the effect of environmental factors, such as gas, light, or temperature, on nanomaterial properties.

The probe [Figure 4.1] protruding out from the device frame is for the stiffness calibration of the force sensor. An electrostatic actuator was chosen over an electrothermal actuator to avoid unwanted temperature increase of the specimen owing to heat conduction, which can alter material properties. The lateral-comb configuration was used for displacement sensing, since the displacement sensor should have a larger linear displacement sensing range than the force sensor. Additionally, there is an open window below the suspended structures in the area surrounding the gap in order to achieve electron transparency, so that transmission electron microscopy can be used to observe the nanomaterial specimen, if desired.

Figure 4.1(a)(b) also show four electrical insulation cuts on the actuator shuttle and the force sensor shuttle. These cuts electrically separate structures of different electrical functions. Meanwhile, mechanical connection is maintained by using the insulation layer material below the device silicon layer of the SOI wafer, as shown in Figure 4.1(b). The specific functions of these insulation cuts are as follows. First, the insulation cut between the displacement sensor and the actuator ensures that capacitance sensing is decoupled from the actuation voltage. Second, the two electrodes at the gap are respectively insulated from the actuator and the force sensor, in order to provide independent electrical connections to the specimen for two-point electrical measurement. During the electrical characterization, an electric current flows in series through beam ‘b’, the specimen, and beam ‘a’ [Figure 4.1(a)]. Third, the calibration probe is insulated from the force sensor for valid calibration.

### 4.2.2 Mechanical Analysis

Since a nanomaterial specimen becomes part of the mechanical system of the device during testing, the motion ranges and stiffness values of the actuator and sensors must be compatible with the mechanical properties of the specimen. Figure 4.2 shows a spring diagram for
The characteristics (motion range and stiffness) of the actuator and the two sensors must be compatible with that of the target specimen, in order to achieve desired testing conditions. Analyzing deformation compatibility and force equilibrium.

\[
d_a = d_s + d_f \tag{4.1a}
\]

\[
k_s d_s = k_f d_f \tag{4.1b}
\]

\[
F_a = k_a d_a + k_s d_s \tag{4.1c}
\]

where \(d_a\) is the displacement of the actuator shuttle, \(d_s\) is the elongation of the specimen, \(d_f\) is the displacement of the force sensor shuttle, \(k_a\), \(k_s\), and \(k_f\) are the stiffness values of the actuator shuttle, the specimen, and the force sensor shuttle, respectively, and \(F_a\) is the output force generated by the actuator.

The equations were used for parameter design of the device, in consideration of the requirement to produce tensile failure of the specimen. The specimen elongation at the failure point can be estimated using

\[
d_{so} = w_s \varepsilon_o \tag{4.2}
\]

where \(w_s\) is the gap width of the device (original specimen length), and \(\varepsilon_o\) is the approximate failure strain according to the reported data in literature. \(d_f\) at specimen failure is set to be the largest displacement of the force sensor in its linear sensing range. Then \(k_s\), \(k_f\) can be selected according to (4.1b). A slightly larger \(k_f\) can be chosen to ensure specimen failure. Subsequently, (4.1a) is used to select \(d_a\) at specimen failure, which is the largest displacement required for the actuator shuttle. Finally, (4.1c) is used to determine the largest \(F_a\) required, for the actuator parameter design (e.g., number of comb pairs).
Figure 4.3: Circuit diagram of the testing setup during electrical characterization of a nanomaterial specimen. In order to extract the intrinsic electrical characteristics of a specimen, contact resistances as well as the resistances of the two beams are considered.

### 4.2.3 Electrical Analysis

Within the circuit for electrical characterization, the specimen is connected in series with beam a and beam b (these beams are labeled in Figure 4.1(a)). Additionally, contact resistances exist between the specimen and the two electrodes [Figure 4.3]. Thus, the measured current–voltage ($I–V$) characteristics are not from the specimen alone. The contact can be either an ohmic contact or a Schottky barrier. Since an ohmic contact can also be regarded as a Schottky barrier with a low barrier height close to zero [114], Schottky diodes are used in Figure 4.3 to represent contacts. It can be seen that at a given voltage, one Schottky barrier is forward-biased, whereas the other is reverse-biased, playing an important role in the measured $I–V$ data. This contact effect must be considered and is discussed in detail in Section 4.4.3.

The resistances of beam a and beam b can be well calculated, given that beam dimensions and the resistivity of the silicon device layer are known. They can also be experimentally verified by fabricating and testing a device with the actuator shuttle and the force sensor shuttle connected. Additionally, if the device layer silicon is not heavily doped, the piezoresistive effect of beam a and beam b should also be taken into account.

It should be noted that four-point probe measurement is capable of eliminating/minimizing the contact resistance effect [90, 115, 116], and can also be realized herein by modifying the device design. However, such modification would require a longer specimen to span four suspended electrodes.
Figure 4.4: Microfabrication process flow. (a) Grow thermal oxide on the backside of the SOI wafer. (b) Pattern thermal oxide layer by RIE. (c) DRIE-etch handle layer Si. (d) RIE-etch thermal oxide layer. (e) DRIE-etch handle layer Si. (f) BOE-etch buried oxide layer and thermal oxide layer. (g) E-beam-evaporate Al and lift off. (h) DRIE-etch device layer Si and release devices.

4.3 Device Fabrication and Calibration

4.3.1 Microfabrication

A deep reactive-ion etching (DRIE) on silicon-on-insulator (SOI) process, modified from [117], was used for device construction [Figure 4.4]. The starting SOI wafer has a 25-μm-thick silicon device layer that was heavily doped with boron (resistivity: 1.7–1.9×10⁻⁵ Ω·m). The low-resistivity device layer reduces the resistivity of beam a and beam b [Figure 4.1(a) and Figure 4.3] as well as their piezoresistive effect.

Briefly, thermal oxidation is used to grow 1-μm-thick oxide layers on the top and bottom surfaces of the wafer, and then the top oxide layer is stripped with buffered oxide etch (BOE) [Figure 4.4(a)]. The bottom thermal oxide layer is then patterned using reactive-ion etching (RIE) [Figure 4.4(b)]. A thick layer of photoresist is patterned on the bottom side, which is used in conjunction with the thermal oxide layer as an etch mask for the DRIE of the handle
Chapter 4. Piezoresistivity Characterization of Nanowires using a MEMS Device

For mechanically connecting suspended structures with electrical insulation, the two-step DRIE process of the handle layer [Figure 4.4(c) and (e)] creates a step difference between the central suspended structure and the device frame, which significantly reduces the risk of device breakage during device handling and operation. The released devices are glued and wire-bonded to custom-made printed circuit boards [Figure 4.5] with a two-channel capacitive-to-digital converter (AD7746, Analog Devices Inc.) for sampling data from the two capacitive sensors on the device. Since the remained flux on the circuit boards can outgas in SEM, it was removed using an ultrasonic cleaner and an aqueous flux remover, prior to the wire-bonding. The glue and solder used in this work are vacuum compatible.
A few devices whose actuator shuttle and force sensor shuttle are connected by design were also included on the same wafer, for device characterization. The equivalent resistance of beam a and beam b was measured to be 81 \( \Omega \), which is negligible in comparison with the nanowire resistances obtained in Section 4.4.3. Furthermore, the deformation of the two beams by the actuator did not result in resistance change. Therefore, beam a and beam b can be safely ignored in the circuit shown in Figure 4.3.

Figure 4.6: Calibration results of the displacement sensor (a) and the force sensor (b)(c). The displacement sensor exhibits a resolution of 1.7 nm at 45 Hz. The force sensor exhibits a displacement sensing resolution of 1.5 nm and a force sensing resolution of 26.8 nN at 45 Hz.
### 4.3.2 Sensor Calibration

The displacement sensing functions of both the displacement sensor and the force sensor were calibrated. The force sensing function of the force sensor was also calibrated. A data sampling rate of 45 Hz was used during device calibration.

The displacement sensor was deformed by driving the actuator [Figure 4.1(a)], with the displacements measured from microscopy imaging and correlated to the output voltage of the readout circuit [Figure 4.6(a)]. Determined from the noise level of the output voltage, the displacement sensor exhibits a resolution of 1.7 nm at 45 Hz. The force sensor was deformed by using a microprobe under a probe station to push the calibration probe of the device, with the displacements also measured from both imaging and the readout circuit. The calibration results are shown in Figure 4.6(b). The displacement sensing resolution of the force sensor was determined to be 1.5 nm at 45 Hz.

A precision microbalance (XS105DU, Mettler-Toledo Inc.) with a resolution of 0.1 µN was used to calibrate the force sensor. Figure 4.6(c) shows the calibration results. The force sensor exhibits a resolution of 26.8 nN at 45 Hz. Additionally, the stiffness of the force sensor can be obtained by calculating the ratio of the slope of the regression line in Figure 4.6(b) to that in Figure 4.6(c), resulting in 18.2 N/m.

### 4.4 Experimental Results and Discussions

#### 4.4.1 Synthesis of Silicon Nanowires

As a type of piezoresistive nanomaterial, silicon nanowires were chosen for characterization by the MEMS device in this work. Silicon nanowires used in this study were vapor-liquid-solid (VLS) synthesized using low pressure chemical vapor deposition (LPCVD). A gold thin film was thermally evaporated onto a silicon (111) substrate, which was subsequently annealed to form a discontinuous film consisting of 50–100 nm diameter gold islands. The substrate was
then introduced into a reactor and brought to a temperature above the Au-Si eutectic point of 363 °C in a H₂ atmosphere. Deposition occurred at approximately 550 °C using a 10% SiH₄/H₂ as the silicon source with trace levels of phosphine (PH₃) as the n-type dopant source at a total pressure between 10 and 50 Torr. Lengths of the synthesized nanowires were 10–30 µm.

### 4.4.2 Transfer of Nanowire onto MEMS Device

A few methods have been reported for the integration of a nanomaterial to a MEMS device. Nanomaterials can be fabricated directly on MEMS devices [103, 118]). Pre-synthesized nanomaterials were also transferred onto MEMS devices via DEP (dielectrophoresis) trapping [104, 119] [111], FIB (focused ion beam) deposition [106], and direct pick-and-place [67, 70, 105, 108, 120–123]. Due to the flexibility of pick-and-place nanomanipulation inside SEM and no need for nanomaterial pre-processing (e.g., sonication), we transferred individual silicon nanowires from growth substrates onto MEMS devices via direct pick-and-place.

For the experimental setup, a silicon nanowire substrate and a circuit board with a wire-bonded MEMS device were placed on the specimen stage of an SEM (S-4000, Hitachi Inc.), where a nanomanipulation system (S100, Zyvex Inc.) was mounted, as illustrated in Figure 4.7(a). A tungsten probe (tip diameter: 200 nm) was installed to one of the nanomanipulators and used to transfer individual nanowires [Figure 4.7(b1)–(b3)]. Throughout the manipulation process, an acceleration voltage of 2 kV in SEM imaging was typically used.

Briefly, the nanomanipulator first approaches an edge of the nanowire substrate and establishes contact with a single nanowire near its root. Following the contact, the nanowire is ‘soldered’ to the probe tip using electron beam induced deposition (EBID). The deposited material from EBID without injecting precursors is carbonaceous material, which is from the decomposition of contaminants inside the SEM chamber [10, 11, 73]. The probe then retracts and pulls the nanowire off from the growth substrate, as shown in Figure 4.7(b1). A detached nanowire typically fractures near its root, so the upper section of the nanowire to be used for testing does not experience tension and remains intact.
Figure 4.7: Nanowire transfer. (a) Experimental setup: The MEMS device and the nanowire sample are installed to the specimen stage of an SEM integrated with a nanomanipulation system. 1. Nanomanipulator, 2. tungsten nanoprobe, 3. silicon nanowire substrate, and 4. MEMS device. (b) Transfer procedure: A silicon nanowire is transferred from its growth substrate to the testing device via nanomanipulation inside an SEM, followed by electromechanical characterization. (b1) The probe picks up a nanowire by soldering it to the probe tip using EBID and detaching it from the growth substrate. (b2) The nanomanipulator transfers the nanowire to above the device. (b3) The nanomanipulator places the nanowire across the gap of the device and the nanowire is EBID-soldered to the two edges of the gap. (b4) The device electromechanically interrogates the nanowire until its tensile failure.

The nanomanipulator subsequently transfers the nanowire to above the MEMS device [Figure 4.7(b2)] and lowers the nanowire to place it across the gap of the device. The first bond between the nanowire and one edge of the gap is formed again via EBID. The nanomanipulator then orients the nanowire to make it perpendicular to the gap, followed by EBID-soldering the nanowire to the second gap edge [Figure 4.7(b3)]. The purpose of fixing the nanowire to the very edges of the gap, rather than some distance away from the edges, is to prevent static friction between the nanowire and the device surface when the nanowire is stretched.

Finally, the probe is retracted from the MEMS device, during which the nanowire break-
Figure 4.8: Mechanical and electrical characterization results. (a) Stress–strain curve. The nanowire exhibits a Young's modulus of 165.3 GPa and a failure strength of 5.67 GPa. (b) $I$–$V$ characteristics of the nanowire under different strain levels.

Prior to stretching the nanowire, the two bonds are strengthened using EBID once again, until the $I$–$V$ characteristics of the nanowire does not change anymore. This step was also performed to ensure the EBID bonds are secure during nanowire stretching and do not break before the tensile failure of the nanowire.

### 4.4.3 Nanowire Characterization

The nanowire specimen was tensile-stretched until its fracture [Figure 4.7(b4)] with the force–elongation data recorded, during which its $I$–$V$ data curves were also obtained using a source measurement unit (SourceMeter 2602, Keithley Instruments Inc.) at a number of strain levels. A representative stress–strain curve from a nanowire specimen is shown in Figure 4.8(a), from which the Young's modulus (165.3 GPa) and failure strength (5.67 GPa) were determined. It can also be observed from the curve that the nanowire specimen did not experience the phase of plastic deformation before fracture, proving to be brittle. The Young's modulus derived from five silicon nanowires was $165.4 \pm 3.9$ GPa ($n=5$), which is in agreement with findings for VLS-grown [111] silicon nanowires as reported in [70, 124]. In all experiments, the nanowire
misalignment was less than 5° between the axial direction of the nanowire specimen and the stretching direction, resulting in an error of less than 1% in the measured Young’s modulus [67]. The failure strengths of the nanowires were determined to be 5.3±0.6 GPa (n=5). The tested nanowires had diameters of 72–97 nm and lengths of 6.7–8.2 µm between the two EBID bonds.

For coupled electrical characterization at each strain level, a voltage sweep (e.g., from −20 V to +20 V) was applied to a nanowire specimen and the current flow was measured. Figure 4.8(b) shows $I$–$V$ characteristics of a silicon nanowire at different strain levels. It can be seen that straining the nanowire resulted in $I$–$V$ changes.

Figure 4.8(b) also shows that the $I$–$V$ curves are not symmetrical with regard to the origin, indicating the existence of Schottky contacts. When the applied voltage is low (<0.5 V), the voltage is mainly distributed on the two Schottky barriers [Figure 4.3], rather than on the specimen. As the applied voltage increases, the specimen starts to contribute more to the $I$–$V$ characteristics. At a high voltage (>5 V), while the voltage drop across the forward-biased Schottky barrier remains small, the voltage drop across the reverse-biased Schottky barrier becomes saturated. Thus, the slope of the linear section of a $I$–$V$ curve at high voltages approximates the conductance of the specimen [114, 125–127]. When the applied voltage is even higher (e.g., >15 V), the $I$–$V$ curves become more nonlinear since the electrical transport through the nanowire is space charge limited [13, 128].

Therefore, the voltage range of 5–9 V in Figure 4.8(b) was used to determine the intrinsic resistance of the nanowire at different strain levels. The resistance and resistivity under the unstrained condition were determined to be $5.9 \times 10^{11} \ \Omega$ and 406 $\Omega \cdot$ m. At 3.0% strain, the resistance was determined to be $2.2 \times 10^{10} \ \Omega$, reduced by a factor of 26.8 from the resistance at zero strain. This significant change in the resistance indicates the piezoresistive effect of the silicon nanowire. As another measure to quantify this effect, the gauge factor (ratio of relative resistance change to strain) of the nanowire was determined to be 67.1 at 1.3% strain. The characterization of the piezoresistivity of silicon nanowire was enabled by the capability of the MEMS device for simultaneous electrical and mechanical characterization.
Repeated tensile tests were performed on silicon nanowires, under the conditions of e-beam on and off. The experimental results indicate that e-beam irradiation did not affect the measured characteristics of a nanowire under e-beam irradiation of different acceleration voltages (0 kV, 10 kV, 20 kV, and 30 kV), with nanowire unstrained (a) and 2.1% strain (b), revealing the effect of SEM imaging. (c)(d) Dynamic current response of a nanowire subject to cyclic mechanical loading with different strain levels (0, 2.1%, and 3.0%), under a constant applied voltage of 20 V, with e-beam off (c) and e-beam on with the acceleration voltage of 20 kV (d).

The EBID deposits for nanowire–device contact were carbonaceous materials, since no gas was used. If a gas injection system is available, different metals such as tungsten, gold, or platinum can be deposited. The Schottky barrier height will be different with a different contact material.

### 4.4.4 Effect of E-Beam Irradiation

Repeated tensile tests were performed on silicon nanowires, under the conditions of e-beam on and off. The experimental results indicate that e-beam irradiation did not affect the measured
mechanical properties. However, e-beam irradiation produces significant effect on $I-V$ characteristics of nanowires. In our study, $I-V$ characteristics of the nanowires were quantified with the e-beam off and on with different acceleration voltages of 10 kV, 20 kV, and 30 kV, under 0% and 2.1% strain levels [Figure 4.9(a)(b)]. It can be seen that e-beam irradiation significantly alters $I-V$ data, likely through charge injection from e-beam into the specimen. Thus, for electromechanical characterization of nanowires, the elongation measurement preferably should be performed electronically. When EM imaging must be used, the acceleration voltage should be kept low.

Figure 4.9(c)(d) show data collected on a silicon nanowire that was subject to cyclic loading at strain levels of 0%, 2.1%, and 3.0%. By comparing the e-beam off result (Figure 4.9(c)) with the e-beam on result (Figure 4.9(d)), the current increases due to e-beam irradiation are 107% for 0% strain, 35% for 2.1% strain, and 56% for 3.0% strain, again demonstrating the significant effect of e-beam irradiation. Additionally, cyclic loading was repeated for over 1,000 cycles. No signs of nanowire fatigue were observed.

Of the over 1,000 cycles, the above e-beam on and e-beam off experiments (Figure 9(c)(d)) demonstrated highly repeatable results, indicating that short-term e-beam irradiation affects electrical measurements but does not permanently change the electrical properties of the nanowires.

### 4.5 Conclusions

This chapter described a MEMS device for simultaneous electrical and mechanical characterization of individual nanowires. The device integrates an actuator, two capacitive sensors, and two suspended electrodes for a nanowire to bridge. Tensile forces and elongation measurements are all acquired electronically, without relying on EM imaging. The two suspended electrodes enable $I-V$ characteristics of a specimen to be obtained at different strain levels. Nanomanipulation (pick-and-place) inside SEM was performed to transfer individual silicon
nanowires from their growth substrates onto the MEMS device. Measurements were made to quantify the mechanical and electrical properties, and piezoresistive effect of the silicon nanowires. The significant effect that e-beam irradiation has through EM imaging on the $I-V$ characteristics of nanowires was also revealed.
Chapter 5

A load-lock-compatible nanomanipulation system for SEM

5.1 Introduction

The capability of real-time imaging with nanometer resolutions makes scanning electron microscopes (SEM) an appealing imaging platform for robotic manipulation of nanometer-sized objects. Nanomanipulation in SEM has been used to manipulate carbon nanotubes [10,73,129], InGaAs/GaAs nanosprings [12,102], and silicon nanowires [13,65,130] for characterizing their mechanical and/or electrical properties. Nanoassembly in SEM was also demonstrated for the construction of three-dimensional photonic crystals [71].

A number of nanomanipulation systems have been developed by companies, such as Kleindiek, Zyvex, SmarAct, and Attocube [Figure 5.1], as well as by academic laboratories (e.g., [10, 77, 131, 132]). Piezo motors and actuators are used due to their high positioning resolution, fast response, high force generation, and no magnetic field generation (thus, no interference with EM imaging).

When a piezoelectric element is operated in the stick-slip mode for achieving a large motion range (e.g., millimeters), it is often called a piezo motor. The same piezoelectric element can
also be operated in the fine mode (called a piezo actuator) to produce fine motion of nanometers with a travel range limited to a few micrometers. Using a single piezo element for both coarse and fine positioning is advantageous when a compact system size is desired. On the other hand, when employing two piezoelectric elements, one functioning as a piezo motor and the other independently as a piezo actuator, a nanomanipulation system is capable of extending the travel range of fine positioning from a few micrometers to tens of micrometers.

For installing a nanomanipulation system into an SEM, nanomanipulators are mounted onto, in most cases, the SEM stage [132, 133], or less commonly, the chamber wall [70] or the ceiling [134] of the SEM. SEM imaging and nanomanipulation occur in the high-vacuum chamber. The installation of a nanomanipulation system or exchanging end-effectors (e.g., nano probes and grippers) requires the opening of the high-vacuum chamber. Breaking the high vacuum and exposing the chamber to the ambient environment contaminate the chamber and incur a lengthy pump-down process (e.g., 30 minutes to 2 hours) after closing the chamber.

Besides the high-vacuum chamber, an SEM has a specimen exchange chamber (load-lock,
typically much smaller than the high-vacuum chamber) through which a specimen is transferred into and out of the high-vacuum chamber. The specimen transfer process does not significantly alter the high vacuum in the SEM main chamber; thus, no lengthy pumping is required, and less contamination is incurred in comparison to the opening of the high-vacuum chamber. Therefore, it is desirable to develop a compact nanomanipulation system that can be transferred through the specimen exchange chamber without breaking the high vacuum. This advance would make nanomanipulation systems more SEM-compatible and improve productivity especially for nanomanipulation tasks that require frequent exchange of end-effectors.

SEM nanomanipulation has been largely performed manually by an operator using a joystick and/or a keypad while closely monitoring SEM images, which is time consuming and skill dependent, and results in low productivity and frequent end-effector breakage. Using SEM as a vision sensor, advances were recently made [77, 78, 115, 135, 136] to facilitate automated nanopositioning. However, piezo motor or actuator has inherent hysteresis demanding a high-bandwidth sensor for hysteresis modeling in feedforward control [137] or closed-loop control [138, 139], but SEM imaging suffers from low frame rates, image drift and noise, and image quality variations due to environmental factors and electrical charging of specimens, necessitating the use of additional position feedback, such as high-resolution optical encoders.

This chapter presents a nanomanipulation system [Figure 5.2] that is small in size, capable of being transferred through the specimen exchange chamber of a standard SEM. This feature circumvents the opening of the high-vacuum chamber of the SEM, which is necessary for existing nanomanipulation systems. The system also integrates high-resolution optical encoders to provide position feedback along multiple axes with nanometer resolutions for closed-loop positioning. The system design, system characterization details, and system performance are presented.
Figure 5.2: Nanomanipulation system that is compact in size, capable of being transferred through the specimen exchange chamber of a standard SEM. 1. SEM pole piece, 2. nanomanipulator, 3. specimen holder, 4. nanomanipulator carrier, 5. male electrical connectors, 6. female electrical connector.

5.2 System Design

As shown in Figure 5.2, the nanomanipulation system has a nanomanipulator carrier (4), on which there are two nanomanipulators (2) and two corresponding male electrical connectors (5). The female electrical connectors (6) are installed onto the SEM main chamber stage permanently, and mate with connectors (5) when carrier (4) is transferred through the specimen exchange chamber like a regular specimen. Electrical wires from connectors (6) are connected to the nanomanipulator driver/controller outside the SEM through an SEM feedthrough port. Carrier (4) has a T-shaped structure at its bottom for its mounting to the T-groove of the specimen stage. After the transfer of carrier (4), the specimen holder (3) is transferred to the carrier through the specimen exchange chamber.

The installation of electrical connectors requires one-time opening of the high-vacuum chamber while mounting and demounting of nanomanipulators do not break the high vacuum. Nor does the exchange of end-effectors affect the high vacuum. In addition, in this design architecture, transferring a specimen into and out of the SEM is decoupled from the nanomanipulation system. Thus, during specimen exchanging, the nanomanipulation system stays
Figure 5.3: Installation procedure of the nanomanipulation system. (a) Female electrical connectors (1) are installed to the specimen stage (3) inside the SEM main chamber (2). (b) The nanomanipulator carrier (6) is installed to the specimen exchange rod (5) after the specimen exchange chamber (7) is aired and its door (4) is opened. (c) The nanomanipulator carrier is transferred to the main chamber for mechanical mounting to the specimen stage and electrical coupling to the previously installed female electrical connectors. (d) The specimen holder (8) with a specimen stub (9) is transferred to the manipulator carrier through the specimen exchange chamber.

Figure 5.3 shows the installation procedure of the nanomanipulation system to a standard SEM (Hitachi SU6600). For demounting the specimen holder or the nanomanipulator carrier, the specimen exchange rod is used again to transfer them out through the specimen exchange chamber.

The system contains two nanomanipulators, both assembled from three nanopositioners (SmarAct GmbH) integrated with optical encoders (reported resolution: 1 nm, Numerik Jena GmbH). The size of each nanomanipulator is 36×21×22 mm³. The nanopositioners, as estimated by the manufacturer, when operating in the coarse positioning mode (i.e., as a piezo motor), have a travel range of 10 mm and a resolution of ~50 nm; and when operating in the
fine positioning mode (i.e., as a piezo actuator), have a travel range of 1 μm and a resolution of ~1 nm. The load carrying capability of the nanopositioner is approximately 3.0 N.

The system can be redesigned to contain more than two nanomanipulators. Additionally, it is also feasible to integrate nanomanipulators with both translational and rotational degrees of freedom into the system for more dexterous nanomanipulation. The present nanomanipulation system producing only translational motions was systematically characterized to quantify its performance inside SEM.

5.3 System Characterization

5.3.1 SEM Imaging

SEM imaging is used as a benchmark to characterize the performance of the actuators and encoders of the nanomanipulation system. SEM is known to have issues such as image drift and noise, making stationary features on a specimen appear moving in the image frame. Thus, characterization of drift and noise in SEM imaging was conducted.

SEM image noise refers to random variations of pixel values, arising from a few sources (e.g., limited number of excited secondary electrons from specimens). Image drift refers to the movement of the entire image due to electron beam drift, charge drift on a specimen, and/or electromagnetic interferences from the environment. In order to quantify image drift and noise, two features on a stationary specimen were visually tracked simultaneously over 5 min using a subpixel visual tracking algorithm [94]. A high imaging magnification of 100,000× was used along with other optimal imaging conditions (e.g., a short working distance of 5 mm, a high acceleration voltage of 30 kV, and the use of a magnetic field cancelation device). The pixel size at 100,000× magnification is 2 nm × 2 nm. These imaging conditions were also used in the subsequent characterization of the nanomanipulation system.

The two tracking curves in Figure 5.4 each represent the position change of a feature relative to its original position at 0 s in the image. Those two tracked features were 1 μm apart. The
Figure 5.4: Quantification of SEM image drift and noise. Drift curves (top) were collected by visually tracking two stationary features on the specimen. The noise curve (bottom) results from subtraction of the two drift curves.

two curves essentially overlap, indicating that image drift dominates noise. The relative position change between the two features (subtraction between the two tracking curves) represents image noise, shown as the horizontal curve in Figure 5.4 (mean: 0.05 nm; standard deviation: 0.33 nm).

5.3.2 Actuator Characterization

The step size of the piezo motor (stick-slip mode) in coarse movement depends on the amplitude and frequency of the driving sawtooth signal. The minimum step size is defined as the minimum repeatable movement of the actuator in the coarse mode. As shown in Figure 5.5(a), the minimum step size of the nanomanipulators is 97.2 nm with a standard deviation of 1.1 nm when the amplitude and frequency of the driving sawtooth signal are set to be 48.84 V and 12,000 Hz, respectively.

The input voltage of the piezo actuator ranges from 0 V to 100 V using a 12-bit DAC (digital-to-analog converter), corresponding to 10 nm/V. The piezo actuator often starts at its center position, e.g., 50 V actuation voltage, in a coarse-fine positioning system for the pur-
Figure 5.5: Actuator characterization results. (a) The step size of the stick-slip movement was experimentally determined. The minimum step size that the system is capable of producing with a high repeatability is determined to be 97.2 nm with a standard deviation of 1.1 nm. (b) The resolution of the actuator is characterized to be better than 0.70 nm. (c) Hysteretic behaviors of the piezo actuator when the initial input voltages are 0 V and 50 V, respectively.

Pose of producing forward/backward movement. The piezo actuator was driven by an input voltage increasing from 50 V to 51 V with an interval of 0.024 V (i.e., 100/212 V). The same imaging conditions and subpixel visual tracking algorithm as used in the SEM characterization were applied. As shown in Figure 5.5(b), the subpixel visual tracking algorithm detects the movement of the piezo actuator after each two voltage increments, since one voltage increment results in the calculated movement of 0.24 nm, smaller than the SEM imaging noise level (i.e., 0.33 nm). The mean and standard deviation of the position increments are 0.51 nm and 0.19 nm, respectively, indicating that piezo actuator has a resolution better than 0.70 nm.
As the actuation voltage increases by 20 V and returns to the initial voltage, the piezo actuator exhibits significant hysteretic behavior [Figure 5.5(c)], translating into poor linearity between the displacement and the actuation voltage. For portions of the curve where a voltage increment corresponds to a travel distance larger than the image noise level, one voltage increment can also result in a movement detectable by the tracking algorithm. Figure 5.5(c) also indicates that hysteresis is highly dependent on the starting voltage, necessitating closed-loop control [138–140] or a suitable mathematical model of the hysteretic behavior [95, 141–143] to compensate for the nonlinearity.
5.3.3 Encoder Characterization

Drift, accuracy, and resolution of the optical encoders were characterized inside the SEM. The encoder reading drifts because the light source of the encoder generates heat and causes the optical scale of encoder to expand. This issue is worsened by poor heat transfer in the high vacuum environment of SEM. Figure 5.6(a) shows encoder reading sampled at 100 Hz for 10 min while the nanomanipulators were kept unactuated. It is not clear why the encoders drift in one direction for the first few seconds when they are turned on and subsequently drift in the opposite direction. After the direction reverse, all encoders drift at a near constant rate of 1.2 nm/s. This result is useful for encoder drift compensation when required. It also guides one to limit the duration between two critical encoder readings to below 1 s, in order to avoid/mitigate the effect of encoder drift.

Encoder accuracy was quantified using SEM imaging. The nanomanipulators were moved in the XY plane in open loop, with the travel distance measured from both the encoders and SEM imaging. The absolute accuracy of the encoders depends on travel distances. For example, when the nanomanipulator traveled by 1,292 nm (measured by SEM imaging), the corresponding encoder reading was 1,280 nm. The encoder error changes proportionally with traveled distances. The results show that the average accuracy of the encoders is 98% of SEM measurements.

Subtracting the drift (e.g., a ramp signal of 1.2 nm/s) from the encoder reading in Figure 5.6(a) yields the noise component of the reading, as shown in Fig. 5.6(b). The noise has a p-p value of 2 nm, limited by the resolution of the encoder. To quantify the minimum motion an encoder (i.e., encoder resolution) is able to detect, a piezo actuator (fine mode) was actuated to travel by approximately 5 nm during 1 s with an interval of 1 nm. The subpixel visual tracking result from SEM imaging is deemed to be the actual displacement, and the encoder reading is the measured output. As shown in Figure 5.6(c) and TABLE 5.1, the difference between the actual position and the encoder reading is less than 2 nm, demonstrating that the encoder has a 2 nm resolution.
It should be noted that when an actuator was actuated to travel a small distance close to the resolution of its encoder, the encoder resolution is the major factor determining the accuracy the encoder reading. When the traveled distance is much larger than the encoder resolution, the effect of the encoder accuracy alone dominates the effect of the encoder resolution.

5.3.4 Control

The integration of encoders enables the implementation of look-then-move control in the XY directions. Considering the large travel range of the piezo motor in the coarse mode and the small travel range of the piezo actuator in the fine mode, two classical discrete PID controllers were implemented to control the coarse-fine system. A switch function was used to determine the selection of the two PID controllers.

However, the accuracy of the optical encoders (e.g., 98% over the travel range) degrades the accuracy of position control in the image frame, demanding an image-based visual servo integrated with the look-then-move control. The combined use of look-then-move and image-based visual servo improves the accuracy while maintaining a reasonable speed.

Denoting \( f_d \in \mathbb{R}^{2 \times 1} \), \( f_i \in \mathbb{R}^{2 \times 1} \) and \( f_c \in \mathbb{R}^{2 \times 1} \) as the desired, initial and current position of the end-effector in the image frame, and \( X_i \in \mathbb{R}^{2 \times 1} \) as the initial position along the XY directions in the actuator coordinate system, the desired position \( X_d \in \mathbb{R}^{2 \times 1} \) of the end-effector in the actuator coordinate system is

\[
X_d = J(f_d - f_i) + X_i, \tag{5.1}
\]
where \( J \in \mathbb{R}^+ \) is the ratio between the displacements in the image frame and the actuator coordinate system by assuming that \( X \) and \( Y \) axes of the actuator coordinate system coincide with column and row of the image frame, respectively. The assumption is feasible because the electron beam and the SEM sample stage can be rotated to align the \( X \) and \( Y \) axes with the column and row of the image frame.

**Coarse-Fine Switch of Look-then-Move Control**

The control input (e.g., \( U_x \) along the \( X \) direction) is switched from coarse mode \( U_c \) to fine mode \( U_f \) by comparing the error \( (E_x) \) with the travel range \( (\Theta) \) of the piezo actuator as follows:

\[
U_x = \begin{cases} 
U_c, & E_x > \Theta \\
U_f, & E_x \leq \Theta 
\end{cases} \tag{5.2}
\]

where \( U_c \) and \( U_f \) are control output from classical discrete PID controllers:

\[
U_c = K_{pc}E_x(n) + K_{ic}\sum_{k=0}^{n} E_x(k) + K_{dc}\Delta(n), \tag{5.3}
\]

\[
U_f = K_{pf}E_x(m) + K_{if}\sum_{k=0}^{m} E_x(k) + K_{df}\Delta(m), \tag{5.4}
\]

where \( n \) (\( m \)) is the discrete step at time \( t \), \( K_{pc}, K_{ic}, K_{dc}, K_{pf}, K_{if}, K_{df} \) are the PID parameters for the coarse and fine control, respectively. \( E_x(n) \) (\( E_x(m) \)) is the error between the current position and desired position, and \( \Delta(n) \) (\( \Delta(m) \)) is the difference between the current position and previous position at time \( n-1 \) (\( m-1 \)).

**Coarse-Fine Switch of Image-Based Visual Servo**

The form of the controller of coarse-fine image-based visual servo is similar as (5.3) and (5.4), except that the error is determined in the image frame. The switch of the coarse-fine control input \( (u_x) \) is as follows:

\[
u_x = \begin{cases} 
u_c, & e_x > \Omega \\
u_f, & e_x \leq \Omega 
\end{cases} \tag{5.5}
\]
where \( u_c \) and \( u_f \) are the coarse and fine control input, and \( \Omega \) (pixel) is often chosen as the ratio between the travel range of the piezo actuator and the pixel size of the SEM image.

### Switch of Look-then-Move Control and Image-Based Visual Servo

The image-based visual servo won’t be triggered until the look-then-move control reaches its steady state, e.g. \( E_x = \tau \) nm (absolute accuracy of the optical encoder corresponding to a travel distance) by choosing the PID parameters in \( U_c \) and \( U_f \) carefully. For example, the control input \( u \) in the \( X \) direction is:

\[
 u = \begin{cases} 
 U_x, & |E_x| > \tau \\
 u_x, & E_x \leq \tau 
\end{cases}, 
\]

\( (5.6) \)

### Positioning along the \( Z \) Direction

The \( Z \) position of a nanomanipulator can also be obtained through SEM imaging, by tilting the SEM stage to achieve views from a different angle. The tilting angle can be determined from the rotary encoder of the SEM specimen stage. The stage tilting induces relative position change between the end-effector and the substrate in the image frame. With the tilting angle and position change being known, the height of the end-effector relative to the substrate surface can be calculated. However, this method depends on the availability of high-resolution rotary encoders and is time costly in implementation. Thus, an SEM vision-based contact detection method is developed for \( Z \)-position determination.

Accurately detecting the contact between an end-effector and the substrate can provide a nanomanipulation system with a reference along the \( Z \) direction. Vision-based contact detection \([63]\) is used. This approach does not require additional equipment, devices or sensors. Using SEM visual feedback, the end-effector (i.e., nanoprobe) is visually tracked in real-time as it descends towards the target surface. Once contact between the end-effector and target surface is established, further motion of the end-effector in the \( Z \) direction causes the end-effector
Chapter 5. A load-lock-compatible nanomanipulation system for SEM

Figure 5.7: The end-effector slides on the substrate after contact is established. A contact is detected if the pixel values difference in current $x_i$ (e.g., B) and previous position $x_{i-5}$ (e.g., A) is greater than a threshold $\Delta d$ (e.g., 4 pixel).

to slide on the substrate surface. This sliding motion is detected by comparing the pixel values difference (e.g., $x_i - x_{i-5}$) and a threshold $\Delta d$ (e.g., 4 pixel) as shown in Figure 5.7.

Upon the detection of the contact (e.g., point B in Figure 5.7), the end-effector is retracted to the actual contact point (point C) by ascending in the Z direction by $\Delta Z$, and then raised by tens of nanometers ($\Delta Z_a$) (to point D). As summarized in TABLE 5.2, encoder accuracy is within 98% of travel distance. Since the motion range along the $\Delta Z$ is typically not beyond 100 nm after contact detection, the nanomanipulation system uses only encoder feedback and standard PID position control for positioning along the Z direction after the first contact detection.

5.4 Results and Discussions

TABLE 5.2 summarizes system specifications and characterized performance. The size of the overall system makes it easily fit through the SEM specimen exchange chamber to enter the high vacuum chamber. The coarse positioning resolution of 97.2 nm is sufficient for the system to manipulate submicrometer-sized objects. However, in order to manipulate nanometer-sized
objects, the system must operate in the fine mode to produce a motion resolution of nanometers.

While the steady state error of the closed-loop controlled nanomanipulation system is determined by the resolution of the encoders, the positioning accuracy depends on the accuracy of the encoders. For example, when the system is controlled to travel 320 nm along the X or Y direction, the system reaches the target position with an error of ±4 nm, benchmarked by SEM imaging. The average encoder accuracy and actuator performance listed in TABLE 5.2 are also applicable to the Z direction. Characterizing the system’s positioning performance along the Z direction was performed by accurately tilting the SEM stage and using SEM imaging from multiple oblique views.

The integrated encoders increase system bandwidth. A comparison was made between look-then-move control and image-based visual servo. The encoder sampling frequency was

<table>
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<table>
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<tr>
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<th>accuracy (look-then-move)</th>
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</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>accuracy (visual servo)</td>
<td>&lt; 1 pixel</td>
</tr>
</tbody>
</table>
CHAPTER 5. A LOAD-LOCK-COMPATIBLE NANOMANIPULATION SYSTEM FOR SEM

Figure 5.8: Control system performance. (a) Step response of the system. Comparison of look-then-move control and image-based visual servo. (b) Tracking a circle using look-then-move control.

set to 100 Hz. SEM imaging at the fast scan mode is 30 Hz as maximum. The control gains were tuned through trial and error. To travel a distance of 250 µm, look-then-move control costs 0.25 s to reach the steady state while visual servo takes 3 s, as shown in Figure 5.8(a).

Figure 5.8(b) shows the tracking of a circle (diameter: 32 µm) with the look-then-move control approach. Tracking the full circle took the system 0.87 s and 10.33 s using look-then-move and image-based visual servo, respectively. Due to encoder inaccuracies (98% of travel distance), the largest error generated by the system was approximately 640 nm. It is also observed that the tracking errors were larger for target positions farther away from the original position, since the absolute encoder error is proportional to the travel distance.

In order to leverage the high speed of look-then-move approach while reducing its errors, a combined use of look-then-move and image-based visual servo was implemented. After look-then-move control reaches the steady state, image-based visual servo can be used to fine tune the final positions, making the tracking accuracy less than 1 pixel with a trade off of a longer tracking time (2.58 s vs. 0.87 s).

A range of end-effectors such as nano probes, AFM cantilevers, and micro-nanogrippers
can be mounted on the nanomanipulation system to perform tasks such as nanomaterial characterization and pick-place assembly. In this chapter, nanowire probing (tin oxide nanowires) is used as an example application to demonstrate the operation of the system.

Both nanomanipulators were mounted with a tungsten nano probe. Electrical connection between the nano probes and outside SEM was established through electrical connectors on the nanomanipulators and the SEM feedthrough port. The probes were chemically treated to remove the native oxide layer using KOH and HF solutions, having an end tip of approximately 150 nm in diameter.

The system is capable of detecting the contact position by monitoring the slope change in the image pattern. Contact detection typically costs 10 s. The system positions the nano probes using look-then-move control along the \( XY \) direction and position control along the \( Z \) direction. Figure 5.9(a) shows the probing of a 90 nm wide Sn nanowire. Exact accuracy of contact detection has not been fully characterized; however, the fact that the nano probes were able to repeatedly land on the 90 nm nanowire confirms that the contact detection accuracy is better than 90 nm. The average positioning time for probing a nanowire is 0.25 s along \( XY \) directions. A SourceMeter (2602, Keithley Instruments Inc.) was used to supply voltages and measure currents. The resistivity of the nanowire was determined from the \( I-V \) data shown in
Figure 5.9(b) to be $1.10 \times 10^{-3} \, \Omega \cdot \text{m}$.

5.5 Conclusions

This chapter presented a compact, closed-loop controlled nanomanipulation system. The system eases end-effector exchange without requiring the opening of the high vacuum chamber of an SEM, eliminating lengthy pumping processes and incurs less contamination. The system was systematically characterized, demonstrating the suitability for nanopositioning. The integrated high-resolution encoders increase system bandwidth. When encoder feedback and SEM visual feedback are combined, the control system is capable of achieving both a high speed and a high accuracy.
Chapter 6

Conclusions

This thesis developed MEMS devices and robotic techniques and systems for the manipulation and characterization of micro and nanomaterials. MEMS actuators and sensors permit the interaction with micro and nanomaterials in a precise and well-controlled manner. Computer vision and automatic control techniques enable the automation of manipulation processes that are skill dependent and time consuming.

A MEMS microgripper was developed that is capable of releasing microspheres with 100% success rate and an accuracy of $0.45 \pm 0.24 \, \mu m$. A MEMS testing device was developed and used to characterize the mechanical and piezoresistive properties of silicon nanowires completely electronically without relying on SEM imaging. The effect of e-beam irradiation from SEM imaging was also revealed and quantified.

Based on the microgripper, automated pick-and-place of microspheres was demonstrated using "looking-and-moving" control. For automated four-point probe on nanowires, four nanomanipulators in SEM were automatically controlled to land on a nanowire using visual servo control. Vision-based contact detection was also implemented for Z position control. A load-lock-compatible nanomanipulation system was developed to make the replacement of end-effectors more convenient.


6.1 Contributions

The contributions of this thesis are:

1. Design and fabrication of a MEMS microgripper that is capable of active release as well as gripping of micro-objects.

2. Demonstration of automated pick-and-place of microspheres with 100% repeatability and high speed.

3. Development of SEM-vision-based servo control and contact detection methods for automated nanomanipulation in SEM.

4. Demonstration of automated four-point-probe measurement of nanowires in SEM.

5. Design and fabrication of a MEMS device for the electromechanical characterization of nanowires.

6. Revealing and quantification of the effect of e-beam irradiation on the $I-V$ measurement of nanowires in SEM.

7. Development and characterization of a load-lock-compatible nanomanipulation system for SEM.

6.2 Future Directions

Many possibilities exist for extending this research. Examples are:

1. To develop a MEMS nanogripper for pick-and-place of nano-objects, such as nanowires and nanospheres. The nanogripper has thin and narrow end tips and can be constructed using combined micro and nanofabrication.
2. To automate the pick-and-place process of nanowires by extending the developed visual servo control and contact detection methods. Nanowire detection and selection algorithms should be developed.

3. To take advantage of the *ex situ* testing capability of the MEMS testing device to study the effects of environmental factors, such as gas, light, or temperature, on the characterization results of nanowire specimens.

4. To modify the design of the MEMS testing device to integrate four suspended electrodes for four-point-probe measurement of nanowires during tensile or compression tests. This new design eliminates the effect of contact resistance on the characterization results and also enables the study of contact effects.

5. To improve on the developed nanomanipulation system by integrating two more nanomanipulators and a rotational specimen stage in the center.
Bibliography


