MEMS-Based Platforms for Multi-physical Characterization of Nanomaterials: A Review

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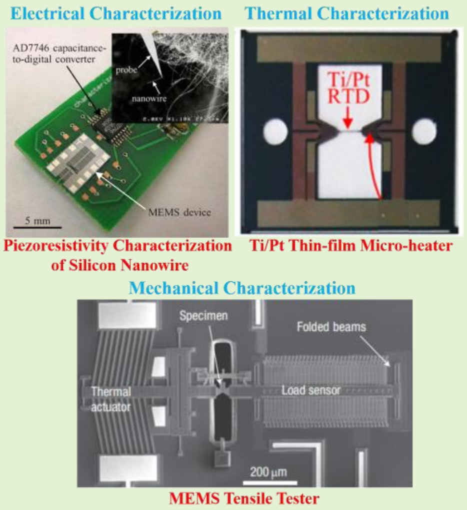
Abstract—**Functional nanomaterials possess exceptional mechanical, electrical and thermal properties which have significantly benefited their diverse applications to a variety of scientific and engineering problems. In order to fully understand their characteristics and further guide their synthesis and usage, the multi-physical properties of these nanomaterials need to be characterized accurately and efficiently. Among various experimental tools for nanomaterial characterization, micro-electro-mechanical systems (MEMS) based platforms provide merits of high accuracy and repeatability, well-controlled testing conditions, small footprint, and compatibility with high-resolution imaging facilities (e.g., electron microscope and atomic force microscope), thus, various MEMS-enabled techniques have been well developed for characterizing the multi-physical properties of nanomaterials. In this review, we summarize existing designs of MEMS-based platforms for nanomaterial characterization, outline critical experimental considerations for nanomaterial characterization using MEMS devices, and discuss applications of the MEMS-based platforms to characterizing multi-physical properties of the nanomaterials.**



Index Terms—**MEMS-based platforms, MEMS-enabled techniques, multi-physical characterization, nanomaterials**

I. Introduction

THE last two decades have witnessed the extensive research on nanomaterials by virtue of their exceptional promise in science and technology. Based on structural dimensions, existing nanomaterials fall into four categories of nanostructures: zero-dimensional nanostructures (e.g., nanoparticles, nanospheres, and isolated molecules) [1, 2], one-dimensional nanostructures (e.g., nanowires, nanobelts, nanotubes, and nanoribbons) [3-5], two-dimensional nanostructures (e.g., nano-films, grapheme, and molybdenum disulfide) [6, 7], and three-dimensional nanostructures (e.g., nanocombs, nanoflowers and nanocups) [8-10]. Due to their superior physical properties and unique nanoscale morphologies, these nanomaterials have been widely used for a variety of applications such as next-generation electronics, nanocomposite synthesis, sustainable energy and biosensing [11-14]. The mechanical, electrical, and thermal properties of these nanomaterials play critical roles in their practical uses, and the experimental determination of these properties is thus of major concern from the perspective of both nanomaterial synthesis and applications.

Based on the type of properties to be measured, a variety of experimental techniques have been employed for nanomaterial characterization. For instance, *in situ* mechanical and electrical testing is typically performed via nanomanipulation on advanced imaging platforms (e.g., electron microscopes, atomic force microscopes (AFMs), and optical interferometers). An AFM can measure attractive or repulsive forces between a scanning probe tip and the nanomaterial sample at the pico-newton level, measuring the induced nanostructure deformation at sub-nanometer level simultaneously, which enables accurate determination of the nanomaterial's mechanical properties [15, 16]. Microscopes are used to observe the motions or deformations of the tested samples and obtain their mechanical or electrical properties [17-19]. In addition, X-ray emission can also provide the stress state of the samples during loading [20, 21]. For thermal property characterization, heat stabilizer boxes or hot plates are utilized as heat sources [22], while the photothermal measurers, thermoreflectance detectors and thermocouple probe tips are commonly adopted as popular devices for temperature measurement [23, 24]. However, there are still some limitations for these traditional characterization methodologies. To name a few, AFM-induced strain is not scalable, and it is difficult for AFM to apply uniaxial strain to the samples [1]. In addition, the setup size of the AFM characterization system is usually very large. Sometimes both the nanomaterial sample's deformation

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and load sensor's deflection need to be imaged, but they cannot be obtained simultaneously when high magnifications are employed [25].

The explosive growth of micro-electro-mechanical systems (MEMS) provides new methods to characterize nanoscale materials, as described in several review articles [25-27]. The broad displacement and load ranges offered by the MEMS platforms can be easily applied to strain-stress tests of most nanomaterials [28-31]. The MEMS platforms possess high load and displacement resolutions and thus can achieve precise sample alignment and manipulation [32-39]. Moreover, the MEMS platforms have the potential to package the testing setup into a monolithic chip.

There are some reported reviews regarding the topics of MEMS platforms and nanomaterial characterizations: Bell et al. [40] and Algamili et al. [41] reviewed the actuation and sensing mechanisms in MEMS-based device, Zhu et al. reviewed the MEMS platforms [42] and experimental techniques [43] for nanomaterial mechanical characterization. The mechanical characterization of materials at micro/nano scales was successively reviewed by Pantano et al. [44], Kujawski et al. [45] , the book written by Yang and Li [46], and the book chapter by Bhushan [47]. Haque and Saif reported reviews of MEMS-based micro/nano scale tensile and bending testing [27, 37], and summarized lessons learned from nanoscale specimens tested by MEMS-based apparatus [48]. Srikar and Spearing [26] discussed about MEMS-based microscale mechanical testing methods. Bhowmick et al. [49] discussed the latest advances in MEMS-based nanomechanical testing techniques that go beyond stress and strain measurements under typical monotonic loadings. Pan et al. [50] summarizes existing MEMS-based platforms developed for cell mechanical manipulation and characterization. Also Wang et al. [51] reviewed the MEMS-based testing apparatus which can be actuated and measured inside SEM and TEM with ease. All the above reviews provide inspirations and insights for this paper's writing.

This paper aims at providing an up-to-date review of the MEMS-based platforms for multi-physical characterization of nanomaterials, including mechanical, electrical and thermal characterization respectively. Firstly, based on the actuation and sensing mechanisms, the performances of representative MEMS platforms for nanomaterial characterization are summarized. Then, regarding the issue of coupling noises in the signal measurement process, we reviewed the typical design theory and methodologies adopted to address the issue, and summarized 5 analytical models employed for the measurement decoupling design of MEMS platforms. Finally, we focus on the applications of MEMS-based platforms in characterizing multi-physical properties of nanomaterials, and have reviewed reported representative works on mechanical, electrical and thermal characterization of nanomaterials using MEMS-based platforms. Also the limitations of existing MEMS-based nanomaterial characterization techniques and platforms are analyzed.

This review could serve as informative guidelines for experimentalists and practitioners engaged in the multi-physical characterization of nanomaterials adopting MEMS-based platforms, what's more, the performance summary of MEMS platforms, analytical models for decoupling design and experimental considerations of multi-physical characterization summarized in this review could facilitate more advanced development and improvement of MEMS-based platforms for nanomaterial characterization.

II. CONFIGURATION OF MEMS-BASED NANOMATERIAL CHARACTERIZATION PLATFORMS

In the perspective of device configuration, the MEMS-based nanomaterial characterization platforms generally consist of three components: excitation part, sensing part and connection part, which will be discussed in detail in the following sections.

A. Excitation Part

The excitation parts are used to provide mechanical/electrical/thermal loads on tested samples. For mechanical excitation, according to the actuation principle, the excitation part can be divided into electrostatic actuation [31, 33, 35, 52], thermal actuation [25, 34] and piezoelectric actuation [53, 54]. The temperature loading parts are mainly composed of thin film microheaters [55, 56] or small heating stages [57].

1. Mechanical loading mechanisms

The MEMS platforms involving electrostatic, thermal or piezoelectric actuation are commonly adopted to test nanomaterials with thickness below 250 nm [28], thus, for these on-chip MEMS actuators, their output forces and displacements are limited by small structural sizes. The typical actuation structures are shown in Fig. 1.

1) *Electrostatic actuation*

The electrostatic actuator typically employs the comb-drive configuration, which can provide large displacement output but relatively small force output (typically on the order of 1-10 μN at tens of volts for devices fabricated via surface micromachining) [31, 33, 35, 52]. As the small output forces may be not enough to produce large strain and test the failure properties of tested nanomaterials, to tackle this problem, high-aspect-ratio comb-drive actuators were fabricated using bulk micromachining [36]. Naraghi et al. [30] proposed a stepped electrostatic actuation structure capable of generating both large force and displacement output, the design of actuator's mechanical motion is inspired from "moving inchworm".

2) *Thermal actuation*

A thermal actuator typically consists of a series of silicon beams supporting a free-standing shuttle [58], and utilizes the thermal expansion caused by resistive heating of the beams to produce displacement output [25, 34]. Conversely, the cooling effect of the beams can be also utilized to load the tested sample [29]. V-shaped beams have been widely used in thermal

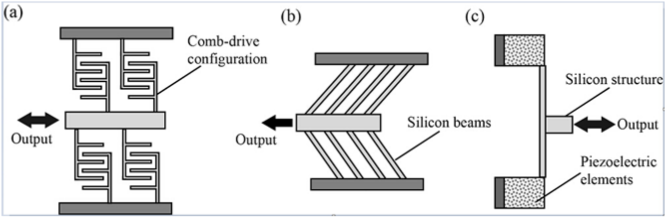

Fig. 1. Typical types of actuators. (a) electrostatic actuator; (b) thermal actuator; (c) piezoelectric actuator.

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actuators since they can easily output large forces. Z-shaped beams can achieve similar function, while their output forces are smaller than those of V-shaped beams [59]. However, the Z-beam actuators are capable of producing bi-directional motions, and can also be used as load sensors due to their wide stiffness range [59]. The thermal actuators typically provide tens of micrometers displacements and tens of milli-newtons forces, which are ideal for testing of brittle nanomaterials with high strength [28]. Two drawbacks of thermal actuation should be noted: (i) the mechanical response of the thermal actuator is relatively slow; and (ii) the heat generated by the actuator could be conducted to the samples [60]. In order to reduce the un-desired heat conduction, heat sink or structural optimization was employed [61, 62], as well as experimental and modeling study on the effect of heat sink beams in temperature control in thermal microactuators [63].

3) *Piezoelectric actuation*

Piezoelectric actuators enjoy advantages of high resolution, wide bandwidth and large density force [53, 54]. However, a main challenge for the piezoelectric actuators is that their fabrication processes are not compatible with most silicon-based MEMS techniques [64]. Some special cases adopting piezoelectric actuation for nanomaterial characterization were also reported eleswhere [53, 65, 66].

2. *Temperature loading mechanisms*

The temperatures loading parts are essential to characterize thermal properties of the nanomaterials. In order to control the characterization temperature, the MEMS platform can be placed into a heat stabilizer box, or glued on a hot plate [22]. But these off-chip heaters can only provide a uniform temperature field. For the sake of accurate temperature control, the silicic, titanic, or platinic thin film microheaters based on MEMS techniques are employed, these films are deposited on the microstructures with various layouts such as spiral and tooth [55, 56], which can manipulate the temperature distribution on the order of micrometers. Two typical temperature loadings are described below:

1) *Thin film micro-heater*

Although furnace or hotplate has been ever used in some past researches of mechanical property testing systems [67, 68], but more and more attention has been paid to the novel thin film micro-heater due to its own several advantages: firstly, the metal thin film micro-heater is able to heat up specimen locally, thus keeping all the other testing system components at room temperature, therefore no additional cooling system is required in the testing system. Besides, the resistively heated micro-heater owns a very fast thermal response, which makes thermal equilibrium between the integrated micro-heater and specimen be attained in much shorter time than using furnace heating system [55]. In addition, the compatibility of the micro-heater with other micro-tensile system for is better than traditional furnace heating mechanism [69]. Detailed applications of thin film micro-heaters are illustrated in Section IV-C.

2) *Heating stages*

In order to investigate the temperature-sensitive nanomaterial's properties, sometimes high temperature is required for the temperature loading part, thus special heating stages allowing high-temperature characterization is often selected, and usually SiC is chosen as the structural material for the heating stage because of its several advantages: (i) outstanding mechanical properties at high temperature (2830 ℃ melting temperature and only 4% elastic modulus decrease at 1000 ℃), (ii) semiconductive characteristics that allow resistive heating to stage, (iii) large heat conductivity that makes heat transferred to tested sample at high efficiency [57].

B. *Sensing Part*

The sensing part is used to measure the load applied to a sample and the resultant sample response. According to the type of load exerted by the excitation part, the employed force, displacement and temperature sensors are summarized below.

1. *Force sensor*

The employed MEMS force sensors include capacitive force sensors, on-chip cantilever-based force sensors, and off-chip force sensors.

1) *Capacitive force sensor*

The widely used MEMS capacitive force sensor shown in Fig. 2(a) is capable of detecting small forces from mN to pN by transferring the input force into detectable capacitance changes. Except for the wide force measurement range, the capacitive force sensor also owns merits such as multi-axes measurements with low noise and high sensitivity, insensitivity to temperature, and easy fabrication [34, 36]. Taking advantages of these merits, capacitive force sensors were adopted in MEMS-based material testing systems used for *in situ* mechanical testing of nanostructures [25] and MEMS-based *in situ* electron and x-ray microscopy tensile testing of nanostructures [70], which are pioneering works from Espinosa group.

Significant improvements in the sensing scheme of a MEMS-based nanomechanical tensile testing technique was recently reported [71], achieving independent and separate measurements of the signals from the two capacitive sensors.

For further simplification, a recent paper [72] reports the realization of independent and separate measurements using a single capacitive readout with no need of electrically isolating the two capacitive sensors, representing a significant advance in the field of capacitive force sensing.

2) *Cantilever-based force sensor*

On-chip flexible cantilevers (Fig. 2(b)) with calibrated spring constants can be also utilized for the load detection, and the input force is calculated by measuring the cantilever deflection [35, 52, 54, 73]. In addition, piezoresistive cantilevers electronically measure the load with high resolution [74], however, they suffer from large thermal drift [33], and the piezoresistive force measurement imposes electrical potential to the sample, requiring proper electrical isolation. Silicon is a common material for piezoresistive sensing, which utilizes the inherent piezoresistive property of polycrystalline silicon, the resistance change can be used to transduce the structures' deflection into an electrical signal, based on this mechanism, the piezoresistive microdisplacement transducer (PMT) [75] and Z-shaped thermal actuator (ZTA) [76] have been successfully demonstrated as force or displacement sensor. Silicon-based piezoresistance has been widely used for various sensors including pressure sensors, accelerometers, cantilever force sensor, inertial sensors, and strain gauges [77].

3) *Off-chip force sensors*

A typical type of off-chip force sensors is AFM probes that can measure the applied loads to the sample [28, 53, 78]. AFM utilizes a laser reflection setup to measure the deflection of a pre-calibrated cantilever probe. Due to the amplification effect during laser reflection, measurement resolution of the probe deflection could be 1000s times higher than regular optical measurement setup [79].

2. *Displacement sensor*

Displacement sensor is another key component in MEMS nanomaterial testing platforms. In nanomaterial mechanical characterization, the displacement sensor is mainly used for measuring specimen deformations, and the accommodation of force and displacement sensors on the same MEMS device facilitate the simultaneous measurement of stress-strain response in some qualitative *in-situ* nanomaterial tests [65, 80]. Capacitive displacement sensors have been mainly used on the MEMS chip for measuring the displacement at the excitation side [81]. The displacement at the sensing part could be measured by the force sensor since nearly all the force sensors quantify an input force by measuring a displacement. Instead of using on-chip displacement sensors, some works also employed high-resolution imaging platforms (a SEM or an optical microscope) to directly measure the deformation of the specimen [36, 82].

TABLE I
PERFORMANCE SUMMARY OF MEMS PLATFORMS

Performance	Disp. Reso.	Disp. Range	Force Reso.	Force Range	Ref
Actuation Methods					
Electrostatic	20 nm	16 μm	60 nN	60 μN	[31]
	1 nm	2.25 μm	34 nN	315 μN	[33]
	0.17 nm	5 μm	108 nN	468 nN	[35]
	1 nm	22.5 μm	14 nN	180 μN	[36]
	20 nm	100 μm	30 nN	300 μN	[30]
Thermal	1 nm	14 μm	12 nN	2.5 mN	[25]
	0.25 nm	1.6 μm	N/A	N/A	[34]
	50 nm	N/A	N/A	600 μN	[58]
	81.5 nm	6.68 μm	1 μN	490 μN	[59]
	N/A	5 μm	0.65 μN	N/A	[60]
	sub-nm	1.6 μm	N/A	0.4 mN	[61]
Piezoelectric	< 50 nm	25 μm	84.5 nN	3 μN	[53]
	100 nm	1.25 μm	5 μN	28 μN	[54]
Force Sensing Methods					
Capacitive	1 nm	22.5 μm	14 nN	180 μN	[36]
	1 nm	14 μm	12 nN	2.5 mN	[25]
	3 nm	N/A	35 nN	N/A	[70]
Cantilever	0.17 nm	5 μm	108 nN	468 nN	[35]
	100 nm	1.25 μm	5 μN	28 μN	[54]
	1 nm	2.25 μm	34 nN	315 μN	[33]
	N/A	37 μm	5 nN	750 μN	[74]
Piezoresistive	4.6 nm	N/A	1.64 μN	128 μN	[76]
Off-chip	< 50 nm	25 μm	84.5 nN	3 μN	[53]
	1 nm	10 μm	10 nN	1.34 μN	[78]

N/A: not applicable; Disp.: Displacement; Reso.: Resolution

3. *Temperature sensor*

For thermal characterization of nanomaterials, temperature sensors such as bimetallic sensor, Ti/Pt resistance temperature detector (RTD) are commonly included in the sensing systems [55, 56]. Micro-Raman spectroscopies and thermocouple probe tips can be employed as temperature measurement tools as well [61]. For the resonance-type MEMS platform, optical interferometers could be used to detect the sample vibration [83].

C. *Connection Part*

The connection parts are components connecting the actuation parts, sensing parts and the samples with each other. Sometimes the connection parts can be very simple or even omitted when the displacement/force sensing resolution is enough. However, in some cases, the connection parts are necessary to improve the performance of the MEMS platforms. Compliant amplifier mechanism is a type of connecting structure which can be integrated in connection part to enlarge the displacement range or improve the displacement resolution of the platform [28, 84]. It is well considered that the compliant amplifier mechanisms can easily increase the displacement or force resolution by an order of magnitude. By using an O-shaped amplifier mechanism, it was reported that the displacement resolution of an electrostatic MEMS platform could reach up to level of 0.2 nm [84]. Also V-shaped and rhombic amplifier mechanisms have been adopted in the MEMS platform as well [62]. The basic structures of the compliant amplifier mechanisms are shown in Fig. 3(a).

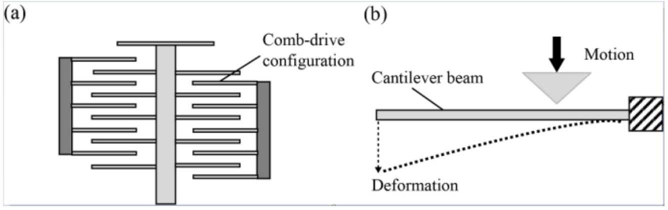

Fig. 2. Typical types of force sensing parts. (a) electrostatic sensor; (b) cantilever load sensor.

Gripping structure is another type of mechanism which can be used in the connection part. Hazra et al. [29] utilized an insertion gripping structure to assemble the sample and the actuation part, as shown in Fig. 3(b). This connection design is able to reduce the negative influence of residual stress, and to eliminate the alignment error to a great extent.

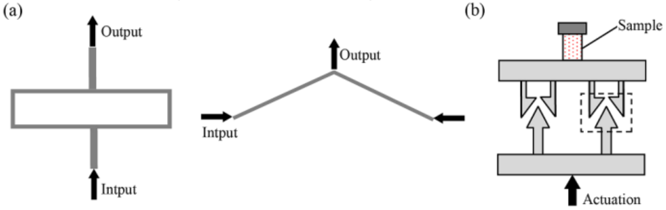

Fig. 3. Typical types of connection parts. (a) compliant amplifier mechanisms; (b) gripping mechanism [29].

TABLE II
REPRESENTATIVE ANALYTICAL MODELS IN MEASUREMENT DECOUPLING DESIGN OF MEMS PLATFORM

Model type	Description	Representative Formula	Parameters	Ref.
Electro-structure coupling model	Capacitive change due to displacement variation	$\Delta C = 2N\varepsilon_0 Ax\frac{1}{d - x^2}$	ΔC: capacitance change N: number of sensing electrodes ε_0: permittivity A: overlapping area d: electrode gap x: displacement	[33]
Thermal-structure coupling model	Output displacement of V-shaped thermal actuator	$U_V = \alpha\Delta Tl\frac{\sin\theta}{(\sin^2\theta + \cos^2\theta\frac{12I}{Al^2})}$	U_V: output displacement α: thermal expansion coefficient ΔT: temperature change I: moment of inertia l: beam length; θ: angle	[81]
	Output displacement of Z-shaped thermal actuator	$U_z = \frac{12\alpha\Delta TL^3}{l^2 + 6L(l + \frac{w^2}{3l})}$	U_V: output displacement α: thermal expansion coefficient ΔT: temperature change L, l, w: structure parameters	[59]
Electro-thermal coupling model	Temperature distribution	$k_p\frac{d^2T}{dx^2} + J^2\rho_e = \frac{S}{h}\frac{T - T_s}{R}$	k_p: thermal conductivity ρ_e: resistivity J: current density S: shape factor T, T_s: temperature R: thermal resistance h: beam thickness	[85]
Resonance model	Young's modulus and residual stress prediction	$f = S_1\sqrt{\frac{E}{\rho}}\sqrt{1 + \frac{\sigma}{E}S_2}$	f: resonant frequency E: Young's modulus ρ: density σ: residual stress S_1, S_2: parameters related to structures	[86]

D. Performance Summary of Representative MEMS Platforms for Nanomaterial Characterization

It should be noted that each excitation and sensing mechanism has its own merits and drawbacks, so the optimal choice of them for MEMS platforms should be based on the certain type of characterization, system specifications and testing requirements. For instance, if your characterization type requires large actuation force, then thermal actuators will be better choice than piezoelectric actuators, but if the sample is thermosensitive, then choosing thermal actuators will bring deleterious effects. Similarly, if the characterization requires large displacement output, then electrostatic comb drives actuator could be better choice. Therefore, getting fully understand of different MEMS platforms' performances is critical for choosing the suitable actuators and sensors for a specific characterization, here we summarize and list some typical MEMS platforms' performances in Table-I classified by the resolution and range of force and displacement output of MEMS platforms, as guidelines for practitioners in the field of nanomaterial characterization.

III. MULTI-PHYSICAL-FIELD MEASUREMENT DECOUPLING DESIGN IN MEMS PLATFORMS

Since the MEMS platforms are usually complicated systems including mechanical, electrostatic, and thermal fields etc., various coupling noises may be generated during measurement process [87]. In order to decouple these multi-physical-field disturbances during signal measurement process, some noise reduction treatments were attempted. Electrostatic actuators' beam structure is grounded to avoid electrostatic interference [88]. The dielectric layer of microheaters is covered for electrical isolation. The design of electrostatic/thermal actuators employs electrical/thermal isolation such as deep trenches and insulation gaps in the device layer of silicon-on-insulator (SOI) [33]. Heat sinks are designed for the thermal actuators to eliminate the heat conducting to the other parts of the MEMS platforms [62]. In the design of capacitive sensors on MEMS platforms, the existences of Brownian noise, Johnson noise and flicker noise may lead to the sensing signal instability [33], as these noises are closely related to electrical and thermal transfers in the structures, thus appropriate design of thermal conduction between MEMS platforms and the supporting stages can minimize these noises.

A. Analytical Models for Decoupling Design

Classified by the structure types, the analytical models employed for the measurement decoupling design of MEMS platforms accordingly fall into 5 categories: mechanical dynamic model, electro-structure coupling model, thermal-structure coupling model, electro-thermal coupling model, and resonance model. In Table-II, we summarize the fundamentals of these five analytical models serving as guidelines in the structural decoupling design of MEMS platforms.

1. Mechanical dynamic model

The dynamic model is used to predict the mechanical behavior of the MEMS platform. Among various models, the spring mass lump model is usually employed as a simplified dynamic model to estimate the system stiffness or motion [34, 36, 61]. Since beam structures are basic components in most MEMS platforms, bending beam models based on the elastic theory are commonly used to analyze the structural stiffness, deformation, amplification ratio and misalignment error, etc.

[25, 28, 62]. It is considered that for very small displacements, roughly until one-fourth of the beam width, the beam model is linear [89, 90]. However, for large displacements, nonlinear effects of the bent beams should be taken into account. The linearity of the loads can be modeled by using the terminal linearity definition [91]. The nonlinear displacement and stiffness function of beams has been studied in [28, 92]. In a recent work [93], the dynamic response of a MEMS-based nanomechanical testing device was systematically investigated in both air and near vacuum environment, in which an analytical model was derived to thoroughly investigate the dynamic response to AC actuation force and ramp force.

2. *Electro-structure coupling model*

The electro-structure coupling model is used in the design of electrostatic actuation or sensing MEMS platforms. The electrostatic force generated by the electrodes has been well modeled by many researchers [33], and the relation between electrical parameters (e.g., voltage and current) and the structure motions can be obtained by substituting the electrostatic force into the mechanical dynamical models. For electrostatic sensing, the capacitance change of the movable electrodes (due to displacement variation) can be calculated using the capacitance model. In this model, the electrostatic fringing field can be neglected when the heights of the capacitive plates are much larger than the gaps between them [33]. For piezoelectric nanomaterials, piezoelectric effect models were employed to estimate their piezoelectric coefficients [33, 94].

3. *Thermal-structure coupling model*

The thermal-structure model based on the thermoelastic theory is widely used in the thermal-actuator-based MEMS platforms. For typical V-shaped and Z-shaped beams, their displacements under temperature changes are discussed in [59, 62, 81]. It should be noted that the loads on the beams will significantly influence their thermal displacements, thus it is suggested that the establishment of thermal-structure coupling model should also consider the loads from the connection or sensing parts.

4. *Electro-thermal coupling model*

The electro-thermal coupling model is employed to analyze the temperature distribution in the MEMS platforms [95]. Components such as beams and plates in the platform can be firstly modeled as resistors in a lumped model [57], then the electrical power and the Joule heating in this model can be calculated by using the Kirchhoff voltage and current laws, in this way the temperature distribution can be obtained. It is noted that the designed thermal actuation platforms should not have large temperature gradients across them, since the mechanical response of the samples may be altered accordingly. The basic equations for electrothermal coupling including the influence of air were also discussed elsewhere [63, 85, 96], specifically, a combined experiment-modeling methodology [63] was presented reporting experimental measurement and multiphysics modeling of the temperature profiles of a V-shaped electrothermal actuator (ETA), also demonstrated that the heat sink beams could play a critical role in reducing the temperature in the ETA.

5. *Resonance model*

The resonance model is based on the resonant vibration of the structure. In general, the measurement of the nanomaterials' resonant response is used to evaluate the Young's modulus and residual stress [26]. Nanomaterials are usually attached on beam structures during resonance characterization, the fundamental expressions of the resonant frequency for beam structures are presented as well [86, 97].

B. *Simulation Methodology*

In some cases, the structures of the MEMS platforms may be over-complicated, leading to the adoption of multi-functional simulation tools in the design. Finite element analysis (FEA) is the most commonly used to simulate the temperature distribution, mechanical deformation and system stiffness of the platform systems [28, 36, 85]. Different boundary conditions should be applied on the FE model based on the specific physical field that is affecting the MEMS platforms, the mechanical boundary condition is the displacement on the MEMS structure; the electrical boundary condition is the voltage applied on the metallic pads of the platforms [98]; and the thermal boundary condition is usually the structural or environmental temperature loading [99].

IV. MEMS-BASED MULTI-PHYSICAL CHARACTERIZATION OF NANOMATERIALS

Nanomaterials is playing more and more important roles in miniaturized electronic, thermal, and electromechanical systems, it is quite necessary to gain thorough understanding of their mechanical, electrical and thermal properties.

A. *Mechanical Property Characterization*

Due to the scaling effects and geometric differences, when the surface-to-volume ratio increases along with the decreased size of structures, nanostructures such as nanowires, carbon nanotubes, and ultrathin films tend to exhibit significantly different mechanical properties compared with their bulk counterparts. This means that we can't easily deduce nanomaterial mechanical properties from bulk properties. Besides, the well-established techniques for mechanical characterization at macro-scale can't be totally transplanted to nanoscale in the respect of equipment and resolution limitations [44]. As a consequence, more and more attentions have been paid to nanomaterial mechanical characterization in last two decades.

Experimental platforms for mechanical characterization of nanomaterials demand high-resolution sensors to accurately measure the mechanical load applied to a nanoscale sample and the resultant displacement [27, 44]. Meanwhile, experimental challenges exist such as how to mount nanometer-sized specimens onto the testing platform [27] with satisfactory precision. Similarly, the specimen alignment is also challenging and needs to be overcome especially in nano-tensile testing, where just a slight misalignment between the specimen axis and loading direction may lead to undesired bending moment and cause non-uniform stress across the specimen width [27, 44, 45, 100].

Due to the aforementioned platform design and experimental challenges in nanomaterial mechanical characterization, only several simple experimental methods were adopted in early stage including resonance testing [101], thin-film bulge testing [102, 103], and nano-indentation testing [104]. With the advancement of MEMS technology, more and more characterization techniques for nanostructures and thin films emerge owing to MEMS test platforms' advantages of easy fabrication, small size, short response time, high performance and low energy requirements [44, 105].

Generally, MEMS-based characterization of mechanical properties for nanomaterials mainly can be divided into static tests and dynamic tests [44], where static tests includes tensile test and bending test, while fatigue test and resonance test can be classified into dynamic tests group.

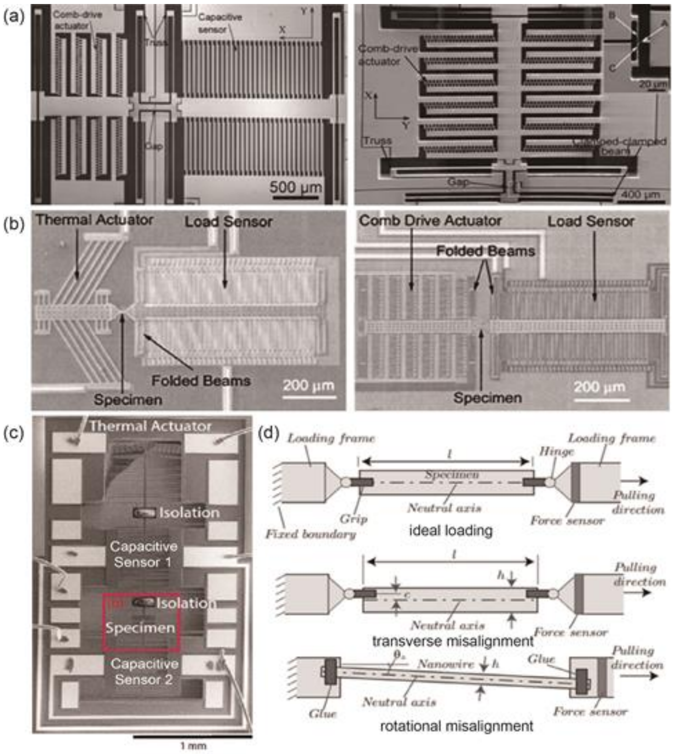

Fig. 4. (a)(b)(c) Photographs of typical MEMS nanotensile platforms [34, 36, 70]. (d) Schematic of a specimen alignment mechanism [100].

1. *MEMS-based tensile testing*

The MEMS-based tensile testing has been applied to characterizing different types of nanomaterials (e.g., nanowires, nanotubes, nanoribbons and nanofibers), and can measure the sample's mechanical properties such as Young's modulus, failure strain, fracture strength and the brittle-to-ductile transition process [72, 106-115]. In a tensile test, a one-dimensional (1D) nano-sample is mounted across a micrometer-sized gap on a MEMS device, and an on-chip micro-actuator stretches the sample from one side of the gap, a micro force sensor measures the tension force of the sample on the other side of the gap. The elongation (thus tension strain) of the sample can be quantified via high-resolution imaging (using an optical or electron microscope) [34, 81, 116] or on-chip measurement of the sample-mounting gap size [117].

Several previous works have engaged in solving above problems and challenges: frictional and electrostatic attraction forces, glues, tapes and connecting ring are often utilized in specimens' mounting process. Several customized force sensors were developed because commercial load cells can't attain nano-Newton resolution, meanwhile commercial capacitance-based displacement sensors were adopted to meet high resolution requirements. In order to alleviate all abovementioned problems, MEMS-based testing systems equipped with thermal actuator (or electrostatic comb drives actuators) and capacitive force sensor begin to emerge to deal with existing challenges. In a recent work, Pantano et al. [118] investigated the problem of load sensor instability and conducted optimization of MEMS-based tensile testing devices.

Some typical MEMS platforms for tensile test are shown in Fig. 4. Zhang et al. [36] developed electrostatically actuated tensile stages (Fig. 4(a)) using comb-drive actuator and two types of force sensors (differential capacitive sensor or clamped-clamped beam sensor), for *in-situ* electron microscopy mechanical testing of 1-D nanostructures. Zhu et al. [70, 81] initially designed a MEMS device for the tensile testing of nanostructure with two types of actuators: thermal and electrostatic actuators (left and right panels of Fig. 4(b), respectively). The device with a thermal actuator was used for displacement-controlled testing, and the one with a comb-drive electrostatic actuator for force-controlled testing. Espinosa et al. [25] developed the first MEMS-based material testing scheme that can continuously observe specimen deformation with sub-nanometer resolution and simultaneously measure tension force with nano-newton resolution. Recently, B. Pant et al. [34] proposed a versatile MEMS material testing setup, as shown in Fig. 4(c), that supports both *in-situ* and *ex-situ* testing of nanomaterial with high accuracy and precision. Except for abovementioned work, there are also MEMS material testing systems for characterizing nanoscale films [65, 119] and 1D nanomaterials [32, 120]. Specially, the tensile testing of constrained carbon nanofibers was *in-situ* conducted inside the SEM using a specialized MEMS-based testing platform [121]. Lu's team developed a 3D printed micro-mechanical device (MMD) for in situ tensile testing of micro/nanowires [122], suggesting the potential to revolutionize micro/nanomechanical characterization of low-dimensional materials by 3D printing of MEMS devices. It's noted that nanomechanical experiments on 1D and 2D materials are typically conducted at quasi-static strain rates of 10^{-4}/s, high strain rate tests were conducted by a piezoMEMS device [123] (adopt piezoelectric actuation, achieving ultra-high strain rates of $\sim 10^{6}$/s) and a MEMS-based nanomechanical testing device [93] employing electrostatic comb drive actuator (strain rate up to 10/s for tensile testing of gold nanowires). To investigate the temperature effects on mechanical properties of 1D nanostructures, an integrated MEMS [115] with an on-chip heater was developed for *in-situ* tensile testing of Si nanowires [114, 115] from room to elevated temperatures, revealing brittle to ductile transition in nanoscale Si, which is of great scientific and technological interest.

To mount a specimen onto a MEMS device, researchers have adopted epoxy gluing [124] or electron-beam ion deposition (EBID) of metals (e.g., aluminum, chromium, platinum and titanium) [54, 61, 124, 125] to mechanically connect the specimen across the testing gap. Some researchers also directly deposited thin-film specimens onto the testing gap of the MEMS device [126] or co-fabricated thin-film specimens with the MEMS device [65]. Another method of fastening the nanomaterial is assembling the sample on the platform through a grip [100]. The tools used to manipulate the nanomaterials on the MEMS platforms include electrostatic grips, probes and nanomanipulators, etc. [127].

In order to eliminate potential measurement errors caused by misalignments (Fig. 4(d)) of the 2D nano-specimen and the testing gap, Kang et al. proposed a self-alignment method for the MEMS platforms, and limited the alignment error within 1% by using a hinge mechanism [100, 128]. Siddharth S. Hazra et al. designed a prehensile gripping mechanism to fully relax the residual stress in the structure, and its alignment error was reduced to 3% [29]. DC and AC/DC electric fields can be also used for the alignment of nanomaterials as an external manipulation [129, 130].

Finally, for the MEMS platforms employing thermal actuation, the temperature of the tested specimen will be elevated due to the heat transfer from the thermal actuator to the specimen. In order to compensate the thermal expansion of the sample, thermal drift correction was adopted by some researchers [32]. Zhu et al. reduced the thermal effect imposed on the specimen by integrating heat sinks and optimizing device geometries [62].

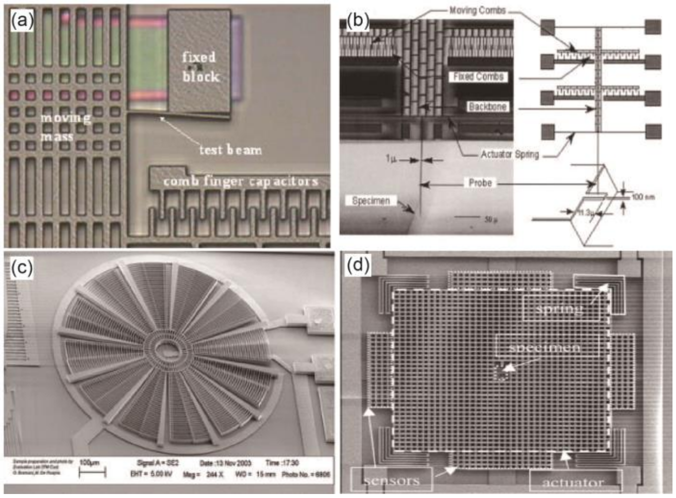

Fig. 5. Typical MEMS bending test platforms: (a) [46], (b) [37], (c) and (d) [131].

2. *MEMS-based bending testing*

Similar to tensile tests, mechanical properties of nanomaterials can also be evaluated via MEMS-based bending testing, which represents another type of widely used experimental techniques [46, 47]. Compared with tensile tests, sometimes bending tests are even preferred because this kind of test require relatively smaller force and can generate larger deflections [44], thus the bending test setup requires more stringent force resolution and larger spring constant of the loading mechanism.

For bulky materials' bending tests, various kinds of methods exist such as axisymmetric test, microbeam test, membrane bulge test, M test, wafer curvature test, membrane deflection experiment (MDE) as well as the on-chip bending test. But as the existed big difference in geometries along with more stringent requirements on force and displacement measurement resolution, bending tests method deviates a lot in nanoscale.

Earlier lateral bending test of suspended nanostructures employs AFM to measure the Young's modulus, strength and toughness of MWNTs and SiC [132], in order to eliminate the negative effects of adhesion and friction in certain bending tests where the nanostructures are lying on a substrate, Walters et al. [133] and Salvetat et al. [134] achieved some improvements by suspending nanotube and dispersing MWNTs respectively. In another representative work, Sundararajan and Bhushan [135] developed a quasi-static bending test technique using AFM to evaluate mechanical properties of Si-based nanoscale structures. Except for AFM-based bending test method, quasi-static bending test can also be performed using nanoindenter [104] that can load up to 500 mN on sample, which is much higher than that in AFM-based testing system (up to about 100 μN). Nevertheless, problems and challenges (e.g., not well-controlled loading process and uncertain boundary conditions) still exist and may affect the test performance and accuracy.

A typical MEMS-based bending test setup [46] is shown in Fig. 5(a), where a cantilever beam (co-fabricated with the MEMS device) moved by a comb-drive electrostatic actuator is bent against a fixed block. Haque and Saif [37] proposed a MEMS-based setup, as shown in Fig. 5(b), which employs a comb-drive electrostatic actuator to bend a 100 nm thick aluminum cantilever beam, the applied force was calculated based on the pre-calibrated loading equation of the actuator, and the beam deformation was measured via high-resolution imaging. Corigliano et al. [131] proposed a rotary comb-drive actuator (Fig. 5(c)) and a parallel-plate electrostatic actuator (Fig. 5(d)) for in-plane and out-of-plane bending test of thin-film (700 nm) polysilicon micro-structures, respectively, the micro-structures were co-fabricated via a commercial surface micromachining process, which allows for nanometer-thick polysilicon structures to be attached to the bottom of micrometer-thick polysilicon MEMS structures.

3. *MEMS-based fatigue testing*

The phenomenon of fatigue failure in micro- and nanoscale material is critical because it can severely impact the durability and reliability of devices made of micro- or nanoscale material (such as MEMS devices made of microscale polycrystalline silicon). The reliability issue is extremely significant because as structure dimensions shrink down to nanoscale, it is more likely move, decompose, aggregate or change shape [60], and the continuous change of property caused by the fatigue may also lead to failure mechanism. Although many studies have been done on the static mechanical properties research of nanoscale materials [136], in order to improve the long-term service performance of them, it is necessary to pay more attention and efforts on dynamic behavior such as fatigue property.

Fatigue tests are usually performed to characterize the fatigue behavior of small-scale material samples. Commonly the methods of fatigue tests can be classified into two types: the uniaxial cyclic test and dynamic bending test, where the former is most common and it can further be divided into tension-tension and tension-compression modes, while the latter is less common because the risk of buckling [44]. And the commonly tested materials include thin metal wires and silicon free-standing films.

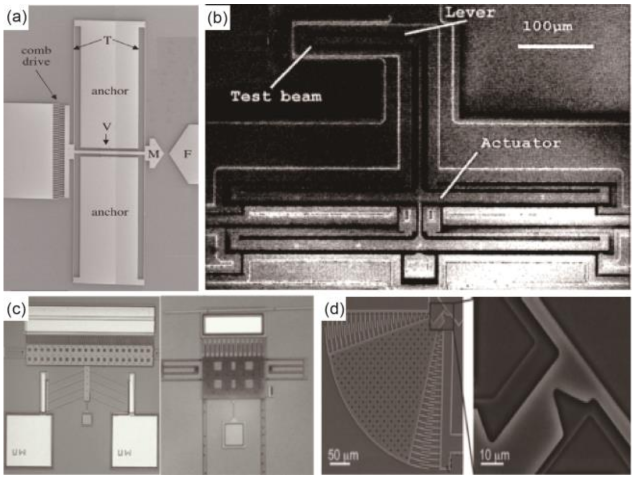

Fig. 6. Typical MEMS fatigue testing platforms.

An important category of fatigue test method is the on-chip testing, compared with off-chip test method, the on-chip test method owns advantages of loading frequency, specimen centering and high measurement precision, so it's commonly adopted.

In field of compression-tension fatigue test, Eppell et al. developed a MEMS device for direct measurement of fatigue behavior of nanoscale fibers [137], as shown in Fig. 6(a), this device consists of an electrostatic comb-drive actuator and an integrated Vernier scale (labelled V) with 0.25 mm resolution to detect the displacement, the choice of electrostatic actuator benefits the device and enables the specimen be fatigued by cyclic loading superimposed on a mean stress [137]. As for another type of actuation mechanism, Larsen et al. [138] designed an *in-situ* MEMS fatigue test device (Fig. 6(b)) with integrated electrostatic actuator to evaluate nano-nickel property. Fischer and Labossiere [60] designed an MEMS-based fatigue tester using electrothermal actuator shown in Fig. 6(c), the comb-drive array located at the top of each device is used to measure the corresponding displacement, the advantage of this device is that any reasonable force can be achieved simply by arranging the electrothermal actuators in parallel, but this device also has drawback of relatively slow response time. In the field of dynamic bending fatigue test, Alsem et al. [139] proposed an polysilicon MEMS fatigue life characterization resonator as shown in Fig. 6(d) to examine the polycrystalline silicon thin films' susceptibility to fatigue, comb drive actuator and capacitive displacement sensor combs are utilized in the work. Recently, a digital micromirror device (DMD)-based MEMS-type device [140] was developed by Lu's team for high-cycle tensile fatigue testing of 1D nanomaterials.

4. *MEMS-based resonance testing*

The resonant tests for nanomaterials allow one to investigate the elastic constants (e.g. Young's modulus) and residue stresses by measuring the resonance frequencies of the material samples.

Among various methods used to characterize mechanical properties, atomic force acoustic microscopy (AFAM) is one of the most important applications, which is based on the observation of the changed resonant frequencies of the AFM cantilever when it contacts with the sample surface [44], a basic AFAM experimental setup is comprised of standard AFM apparatus and equipped with a piezoelectric transducer that excites ultrasonic acoustic waves towards sample. Passeri et al. [141] adopted AFAM technique to perform the characterization of SnSe ultrathin film and deduced the indentation modulus of it. And Stan et al. [142] used contact resonance atomic force microscopy (CR-AFM) to accurately determine the radial elastic moduli of ZnO nanowires with diameters smaller than 150 nm.

Compared with abovementioned testing methods using AFM cantilever beams, MEMS-based resonant structures provide more robust and repeatable ways to estimate and evaluate nanomaterial properties, and these resonant devices own many advantages because they can apply electrostatic cyclic loading to micro- or nanoscale film structure at high frequencies and high measure resolution [143]. Jeong et al. [143] fabricated a testing-ready resonating device shown in Fig. 7(a) that is driven by electrostatic force, the temperature dependency of the elastic modulus of Si thin film is investigated. Nguyen [144] developed a polysilicon micromechanical resonator (Fig. 7(b)) which is laterally comb-driven and folded-beam suspended. Taking advantages of interdigitated electrostatic comb drive, Tang et al. [145] presented the designs of a linear resonant structure with a mass suspension and a torsional resonant structure using a spiral-spring suspension as shown in Fig. 7(c).

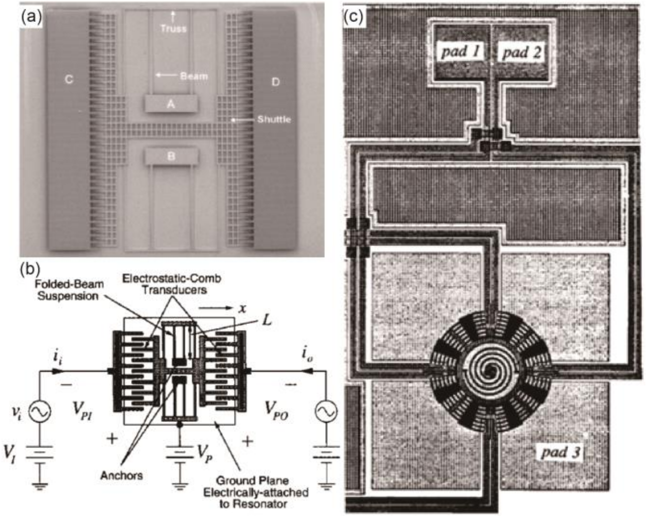

Fig. 7. Typical MEMS resonance testing platforms.

The excitation in the resonance test can also be generated by a piezoelectric plate or laser diode [86, 146]. An optical interferometer can be used to detect the vibration of the sample. It is noted that the air damping and squeezed-film damping can significantly influence the resonant frequencies of the structures, thus the damping effect should be taken into account in a resonance test.

B. *Electrical Property Characterization*

Better understanding of the electrical properties of nanomaterials will contribute to the development of next-generation nanoelectronics and nano-sensors which promise ultrahigh performance [147]. In addition, the intrinsically coupled electromechanical properties of nanomaterials (e.g. 1-D ZnO nanowires that are piezoelectric [148] and piezoresistive [149, 150]) also provide special routes of detecting mechanical loading from the electrical change of the nanomaterial and controlling mechanical deformation of nanomaterials via electrical excitation [46]. Therefore, it is also of great interest to carry out electromechanical characterization of nanomaterials [66, 151]. For example, performing the electromechanical measurements of nanoscale fibers will benefit the development of biosensor [148, 152], characterizing piezoelectric properties of thin films, nanowires, and nanobelts has numerous potential applications in actuators and motion-controllers [46].

In the field of MEMS-based piezoresistive characterization of nanomaterials, piezoresistance of carbon nanotubes and FIB-deposited carbon nanotubes were successfully measured using MEMS-based platforms [149, 150]. In addition, Passeri et al. [153] developed a piezoresistive microcantilever-based sensor for biomedical microelectromechanical system (BioMEMS) application.

In the scope of *in-situ* characterization, mechanical and electrical characterizations of the SiC nanowires were performed simultaneously [73] and an electrostatic tensile device (Fig. 8(a)) was developed to facilitate the characterization. A method (Fig. 8(b)) based on point resistivity measurement was also reported [66], where electromechanical characterization of nanoscale freestanding films was performed on a MEMS tensile stage. The device utilized pre-fabricated electrical isolations (shown in Fig. 8(c)) to allow electrical currents flowing through a prescribed path to eliminate the impact of contact resistance on electrical property characterization. A microscale, thermally actuated uniaxial testing stage was developed for *in-situ* electromechanical characterizations of nanofibers [152].

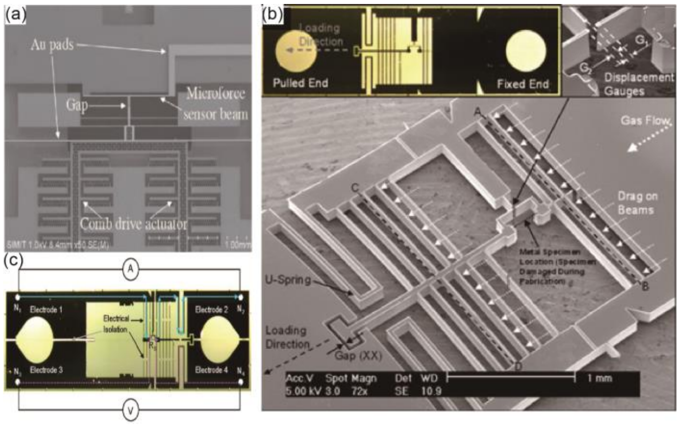

Fig. 8. Typical MEMS electrical characterization platforms (a) [73], (b) [66] and pre-fabricated electrical isolations (c) [66].

But because it is experimentally verified that the e-beam irradiation from electron microscopy imaging (*in-situ* experiment) can significantly alter the characterization results, in order to solve this problem, Zhang et al. [147] developed a MEMS device using electrostatic actuators and capacitive sensors for electromechanical characterization purpose, in this design the measurement process is independent and not relying on the electron microscopy imaging, making the properties of tested silicon nanowires not affected by the e-beam irradiation. Zhu et al. [148] designed a nanoelectromechanical oscillator to measure the piezoelectric coefficient of ZnO nanowires.

Other devices for electromechanical characterization also include micromachined cantilevers [1, 154, 155], where nanomaterials were grown on or sandwiched in the cantilever structures, and controlled forces are applied on the cantilevers using microbalances and mechanical stylus, etc, hence the resistance of the nanomaterial varies as the bending of the cantilever.

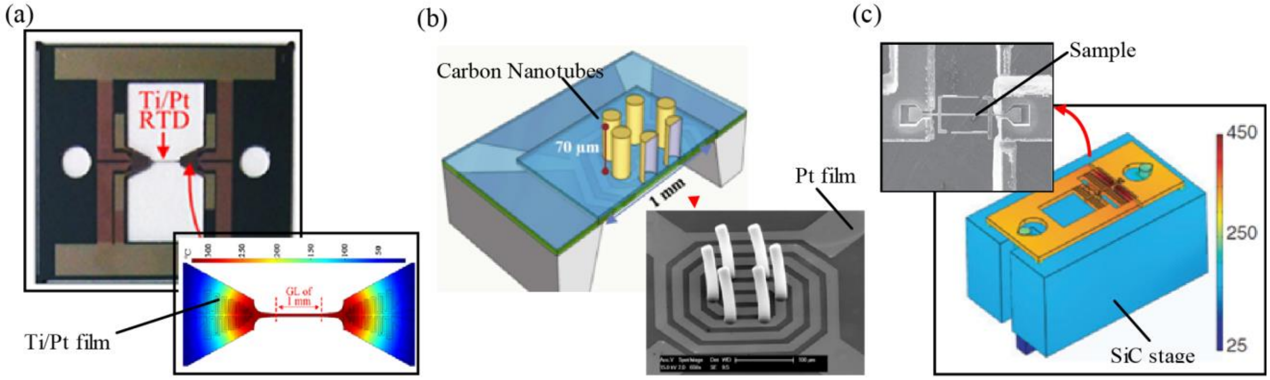

Fig. 9. Typical MEMS thermal characterization platforms

C. Thermal Property Characterization

As rapid development of micron and nanoscale electronic, mechanical devices and the integrated micro/nano-electro-mechanical systems (MEMS and NEMS), it becomes more and more important to predict and characterize the thermal transport properties because thermal conduction of nanomaterials plays a critical role in controlling the performance and reliability of nano/micro devices and systems [156].

The thermal properties of the nanomaterials include thermal expansion, conduction, dissipation, and temperature dependent mechanical properties. Temperature–strain, temperature–stress, and temperature–resistance curves are generally tested in the thermal properties' characterization.

Compared with common characterization using MEMS-based platforms, temperature control devices should be included in the test systems. Different MEMS-based methods and platform for nanomaterials thermal characterizations are summarized below.

Hot plates are simple heating devices but cannot be accurately manipulated in a micro scale [157], thus thin film microheaters were adopted by some researchers because of their good compatibility with MEMS structures. Ang et al. [55] developed a spiral Ti/Pt thin film micro-heater shown in Fig. 9(a) that is integrated in a micro-tensile testing system, and this micro-heater could provide tensile tests with elevated temperatures, ensuring single crystal silicon (SCS) nanofilm being locally heated up. In addition, Che et al. [158] present the fabrication and characterization of molybdenum microheaters for high-temperature gas sensing applications. Silvestri et al. [56] used the spiral Pt film microhotplate to measure the thermal dissipation properties of Carbon Nanotubes arrays, as shown in Fig. 9(b).

Thermal conductivity measurement is a critical component in thermal characterization and play an important role in nanodevice performance. Tunable thermal property of magnetically polarizable nanofluid was characterized and investigated in order to get enhanced thermal conductivity (TC) [159]. In addition, Kwak and Kim characterized the TC of copper oxide nanofluid [160], Che et al. investigated thermal conductivity of carbon nanotubes [156], Cui et al. [161] proposed a novel MEMS-based dual temperature control (DTC) measurement method for thermoelectric properties of individual nanowires.

In order to test samples at a very high temperature, SiC MEMS apparatus was adopted by some researchers, as shown in Fig. 9(c), because it has a large heat conductive coefficient so heat can be transferred to the samples efficiently, and also the mechanical properties of SiC are not sensitive to temperature, so the reported temperature control range was as high as 700 °C [57].

Besides abovementioned characterization in individual physical-field (mechanical, electrical and thermal), multi-physical-field characterization could also be well conducted utilizing functional MEMS platforms, including electromechanical, thermoelectric, and thermomechanical characterization. To name a few, in scope of MEMS-based electromechanical characterization, in situ tensile MEMS stages were developed for electromechanical characterization of nanoscale freestanding films [66], nanofibers [152], single-walled CNT [1], nanowires [70]. Additionally, MEMS devices have been well-demonstrated as a popular platform for piezoresistivity characterization of single nanowires [73, 147, 149, 162, 163] benefited from its capability of simultaneous electrical and mechanical measurements. For other multi-physical-field properties, thermoelectric properties of individual nanowires were measured by a MEMS-based dual temperature control (DTC) method [161]. What's more, an integrated MEMS testing stage was reported for thermomechanical testing of 1D nanostructures, revealing brittle to ductile transition in SCS nanowires [115] and Si nanowires [114].

V. CONCLUSION

Despite the rapid advances of MEMS-based nanomaterial characterization techniques, there still exist some design and technical challenges for developing MEMS-based characterization platforms. For instance, in some MEMS setups [164, 165], the supporting structure of the nanoscale sample is not strictly rigid due to the limitation of the structural sizes, hence the mechanical response of the sample is inevitably influenced by the uncertain boundary condition. Moreover, residual stresses of MEMS devices, caused by temperature gradients or material mismatch, may result in non-negligible geometric deformations of the platform structures [26], which leads to inherent measurement errors during nanomaterial characterization.

In conclusion, this review presents an up-to-date summary of the existing MEMS-based platforms for multi-physical characterization of nanomaterials, including mechanical, electrical and thermal characterization respectively. First of all, we have summarized reported works regarding the fundamental configuration parts (excitation, sensing and connection parts) of MEMS platforms, providing a brief comparison of different MEMS platforms' performances classified by the resolution /range of force/displacement output. Besides, regarding the problem of coupling noises in the signal measurement process, we have reviewed the typical design theory and methodologies adopted to address the issue, and have presented 5 analytical models employed for the measurement decoupling design of MEMS platforms. Last but not the least, we focused on the applications of MEMS-based platforms in characterizing multi-physical properties of nanomaterials, and have reviewed reported representative works on mechanical, electrical and thermal characterization of nanomaterials using MEMS-based platforms, the limitations of existing MEMS-based nanomaterial characterization platforms are analyzed as well.

The present review could serve as informative guidelines for experimentalists and practitioners engaged in the multi-physical characterization of nanomaterials adopting MEMS-based platforms, what's more, the performance summary of MEMS platforms, analytical models for decoupling design and experimental considerations of multi-physical characterization summarized in this review could facilitate more advanced development and improvement of MEMS-based platforms for nanomaterial characterization.

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