RESEARCH DIRECTIONS IN EXPERIMENTAL SOLID MECHANICS AT SMALL SCALES

Research Trends in Mechanics
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Editor

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Introduction

Experimental Mechanics is the subgroup of Theoretical and Applied Mechanics that feeds, via observation and measurement, the development of new models for the rich variety of phenomena that are still areas of open enquiry and research in solid and fluid mechanics and dynamics. Over the years, advances in technology have sharpened the perspective of these eyes of the mechanics community. One area where this has been particularly true has been in the elucidation of phenomena associated with the mechanical behavior of materials at small scales.

The goal of this report is to identify major research topics in this area that are likely to be the focus of enquiry for the next decade. Since these topics will invariably spur the development of new tools, some attention will also be directed towards current trends in this arena. This report gathers the input of a number of leaders in topics of interest and the development of new tools. The contributors are identified (Table 1) and gratefully acknowledged for their short reviews that follow.

Table 1: Topics of Interest and Contributors

	TOPIC	CONTRIBUTOR	AFFILIATION
1	Research Directions in Tribology	Robert W. Carpick	University of Pennsylvania
2	Mechanobiology	Taher Saif	University of Illinois at Urbana-Champaign
3	Molecular and Cell Biomechanics	Gang Bao	Georgia Institute of Technology
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5	Mechanical Characterization of Nanostructures	H. D. Espinosa	Northwestern University
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1. Research Directions in Tribology

Robert W. Carpick

Tribology is the study of interacting surfaces in relative motion and the resulting phenomena of friction, adhesion, lubrication, and wear. It is intrinsically interdisciplinary, as sliding interfaces involve solid mechanics (elasticity, plasticity, fracture, fatigue, and atomic-scale bond breaking), thermal and fluid sciences (hydrodynamic lubrication, thermal transport), materials science (crystallization, amorphization, diffusion, phase transitions), physics (phononic and electronic excitations), chemistry (chemical bond formation and dissociation, adsorption) and even biology (when considering processes in the body or orthopedic implants). Mechanics lies at the center of tribology, not only historically[1] but also today, where leaders in tribology consistently draw upon mechanics to describe and understand behavior at sliding interfaces.

Three broad issues motivate supporting mechanics research efforts in tribology. The first concerns the impact of tribology on energy use. A significant amount of energy is wasted annually due to friction and wear[2, 3], and the economic, environmental, and safety costs of wear-induced failures can be extensive, and even life-threatening[4]. As well, many new energy-efficient technologies are obstructed by tribological issues. For example, Carnot efficiency can be greatly improved with higher temperature operation, but materials that withstand such severe conditions are lacking. Crucial applications include automotive systems, power generation and conversion (*e.g.*, wind turbines), and energy extraction (*e.g.*, drilling, hydraulic fracturing),

The second broad issue arises in nanoscale applications, where the surface-to-volume ratio becomes so large that surface and interfacial interactions like adhesion (bonding), friction (energy dissipation), and wear (material modification and removal) are dominant. This point is painfully appreciated by the microelectromechanical systems (MEMS) community, since MEMS device performance is critically limited by adhesion-, friction- and wear-related failures[5, 6]. This is even more severe for nanoelectromechanical systems (NEMS)[7, 8]. NEMS switches, for example, offer orders of magnitude lower power consumption than conventional solid state transistors. However, to be commercially viable, these switches must open and close more than 10^{16} times without degrading. However, failures arising from adhesion, contamination, and wear are a barrier. Other nanoscale applications where tribology is critical include next-generation hard disks which need to operate at high local temperatures[9], and nanomanufacturing schemes involve contact interactions between materials, such as tip-based nanolithography[10, 11]. Nanoscale wear limit these applications [12, 13], and new scientific insights into nanoscale contact could help advance these technologies.

The third broad issue pertains to biological systems. For example, the orthopedic implant market continues to grow as the population ages, but with this comes the need for implants to last longer. Recent wear-related failures of metal-on-metal hip implants have resulted in medical complications, patient suffering, undesirable replacement surgeries, and large pending lawsuits[14]. Durable, biologically-compatible materials that can withstand demanding conditions for extremely long times, or whose lifetimes can be reliably predicted, are needed. More generally, in living systems, interactions at interfaces control much of the function of biological systems as a whole. Examples where tribological effects are critical include all places where cartilage is located, the operation of the eyelid, the development of engineered tissues, the

mechanism of touch, all forms of cell-surface interactions which are controlled by focal adhesions[15], the operation of molecular motors[16], and the packing of DNA into viruses[17]. In all these cases, the predictive understanding of biomaterial interfaces is critical.

The complexity of tribological interactions requires quantitative, multi-pronged approaches. There is potential for major advances due the development of new *materials* and *methods* that enable a new generation of tribological advances. Regarding *materials*, coatings with unrivaled tribological properties such as diamond-like carbon have emerged from the laboratory to the marketplace[18]; and several nanoparticles can function as remarkably effective lubricant additives[19]. However, widespread use of these materials is inhibited by factors including environmental factors, scale-up, allowable loads, and uncertain lifetimes. Mechanics research to address these issues is needed.

Regarding *methods*, the length- and time-scales of highly accurate, atomistically-informed simulation methods have recently begun to intersect those of advanced experimental approaches[20], including powerful *in situ* experimental techniques[21, 22]. This convergence is crucial because it is inherently difficult to observe phenomena at the hidden sliding interface. Close coupling of experiments with theory to take advantage of new materials is a key direction where mechanics researchers can take a leading role, particularly by applying the recent knowledge and instrumentation developed largely in the mechanics community in pursuing small-scale mechanical testing approaches[23, 24].

A key approach worthy of continued support is to perform studies at the level of single asperity contacts. Measurements and simulations using this approach have already provided a range of insights. The primary tool experimental tool is atomic force microscopy (AFM) [25, 26] and related local probe methods, which is has reached an advance level of development. Atomistic simulation techniques such as molecular dynamics (MD) have been used to model single-asperity contacts as well as their dynamics during sliding [27-31].

A key major challenge pertains to the time scale gap: simulations time scales are orders of magnitude faster than experiments. Matching of sliding speeds has only recently been demonstrated in specific cases [32, 33]. Further efforts should take advantage of opportunities massive parallelization, accelerated MD techniques[34], and high-speed AFM experiments. This should be coupled with continued refinement of potentials used in MD, coupling to *ab initio* methods, and efforts to match length scales, materials, and test environments. It is highly desirable to investigate extreme conditions both in experiments and simulations, such as high temperature environments, high stresses, and high strain rates, where the highly non-equilibrium conditions limit the application of traditional theoretical approaches. Coupled experimental and simulation studies could aid in computer-aided discovery and design of new materials with outstanding tribological properties, as envisioned by the Materials Genome Initiative[35].

These efforts are needed since reduced funding opportunities and academic focus in the U.S. in tribology are threatening to lead to a loss of knowledge, expertise, and talent gained over the last few decades. With a renewed focus, major advances can be achieved by leveraging the

convergence of length scales and time scales in experiment and simulation that is presently occurring.

2. **Mechanobiology** Taher Saif

Concepts and methods of mechanics have been used in diverse fields for centuries. Biomechanics, for example, has contributed significantly to the fields of cardiology, tissue engineering, and prosthetics. However, biological functions of cells - the fundamental units of life - have long been understood as determined by biomolecular and genetic processes. This perception is beginning to change. There is increasing experimental evidence suggesting that extracellular and intracellular mechanical forces have a profound influence on a wide range of cell behavior such as growth, differentiation, apoptosis, gene expression, adhesion and signal transduction. It is now understood that cells respond to both biochemical and mechanical cues and determine their functionalities.

Cells generate forces by employing molecular motors and cytoskeletal structures [36] and apply them to the extracellular matrix and to the neighboring cells. Cells sense these forces and their micro-environment, and in response tune their own functionalities and forces, and remodel the micro-environment. This cell-cell and cell-substrate cross talk begin at embryogenesis, and sustain through development, disease progression and aging. A few specific examples will clarify.

Early development: It is now known that differentiation of mesenchymal stem cells can be determined by the mechanical stiffness of their substrate alone. Cells on 0.1-1 kPa substrate (brain stiffness) differentiate to neurons, on 10 kPa substrate (muscle stiffness) to muscle cells, and on 40 kPa substrates (collagenous bone stiffness) to obteoblasts (bone cells) [37]. Neurons develop tensile forces within the first two hours of synaptogenesis (formation of synaptic junction) in vivo, and this tension is essential for the maturation of the functional synapse [38]. Neurons maintain this tensile force actively during development. Such tension might be critical for memory and learning in animals. Significant stresses and stress gradients develop during organ development. The force fields and their kinetics play a critical role in guiding cell migration and morphogenesis [39], as well as gene expression with high degree of spatio-temporal specificity [40].

Disease progression: Several diseases have now been identified with mechanical signatures. Malaria infected red blood cells are more than five times stiffer that healthy cells which prevent them from squeezing through small capillaries [41]. Increased stiffness of breast tissues due to collagen crosslinking promotes breast cancer malignancy [42]. Colon cancer cells with low metastatic potential express many of the hallmarks of metastasis by simply being cultured on appropriately soft substrate for seven days [43]. Here, the cells sense the mechanical micro-environment, and in response decide on their fate.

Aging: It is well known that lung stiffness increases, bone density decreases, cartilage degrades, and blood vessel walls stiffen with age, which increases the risk of atherosclerosis, hypertension and other diseases. Only recently has the mechanics connection to atherosclerosis has been revealed. Endothelial cells that line the blood vessels increase their contractility in

response to increased stiffening of the vessel walls. This degrades endothelial cell-cell junctions and promotes leukocyte extravasation leading to plaque formation, known as atherosclerosis [44].

Even though it is clear that mechanics matters at the single cell scale, the detailed mechanisms of mechanotransduction, i.e., how cells transduce the mechanical signals to cell functionalities, remain elusive. Questions such as, do cells use the time history of the force signal (magnitude, frequency, and spatial information) to achieve functional specificity and future fate, are their mechanical signals that catalyze the transformation of a healthy cell to a malignant one, are currently under intense investigations. The central question of mechanotransduction poses a grand challenge to cellular and molecular biology, and offers a new paradigm for mechanics. A unique and an exciting feature of this paradigm is that the "constitutive relations" are yet unknown. The very notion of constitutive relation needs to be re-interpreted in light of molecular biology, cell signaling and genomics.

In this new paradigm, experimental mechanics can make a significant difference, just as it did and continues to do so in deciphering the structure-property relation in materials. Not too long ago mechanicians adopted tools from materials science, such as TEM (Transmission Electron Microscope) and AFM (Atomic Force Microscope) in conducting mechanics experiment. Similarly it can be envisioned that the tools of biology, such as transfection, PCR, Western Blot, and RNA-seq will also be part of their methods of investigations. The challenge, however, lies in identifying biologically relevant questions related to mechanics that are fundamental in nature and have potential clinical impact. Close collaborations with biologists and clinicians, together with sufficient knowledge in biological science, are essential in defining such questions and in designing cell mechanics experiments.

Powerful concepts and tools of mechanics, if applied with minimal empiricism, and in conjunction with careful experiments, simple and insightful theoretical modeling, and testable hypothesis, will play a significant role in understanding cellular mechanotransduction, and in identifying its key parameters. The new insights may contribute to early disease detection, therapeutics, drug discovery and delivery, and quantitative prognostics.

3. **Molecular and Cell Biomechanics** Gang Bao

Mechanosensing and Mechanotransduction: As the basic unit of life, living cells perform an enormous variety of functions through synthesis, sorting, storage and transport of biomolecules; expression of genetic information; recognition, transmission and transduction of signals; and conversion between different forms of energy. Many of these cellular processes can generate, or be regulated by, mechanical forces at the cellular, subcellular and molecular levels. For example, during cell migration, contractile forces are generated within the cell in order for the cell body to move forward. These contractile forces, in combination with the adhesion of cells to extracellular matrix (ECM) through focal adhesion complexes, enable cells to sense the stiffness of the surrounding substrate and respond to it. Many normal and pathological conditions are dependent upon or regulated by their mechanical environment. Some cells, such as

osteoblasts and vascular cells, are subjected to specific forces as part of their 'native' physiological environment. Others, such as muscle and cochlear outer hair cells, perform their mechanical function either by converting an electrical or chemical stimulus into mechanical motion or vice versa.

Of particular importance is the ability of cells to sense mechanical force or deformation, and transduce these mechanical signals into a biological response [45]. For example, endothelial cells can recognize the magnitude, mode (steady or pulsatile), type (laminar or turbulent) and duration of applied shear flow, and respond accordingly, maintaining healthy endothelium or leading to vascular diseases including thrombosis and atherosclerosis. Vascular smooth muscle cells in the arterial wall remodel when subjected to pressure-induced wall stress. Fibroblast cells 'crawl' like an inchworm by pulling the cell body forward using contractile forces. Bone alters its structure to adapt to changes in its mechanical environment as occurs, for example, during long bed rest. Stem cells sense the elasticity of the surrounding substrate and differentiate into different phenotypes accordingly. These and other examples demonstrate the ability of cells to sense and respond to their local mechanical environment. However, little is currently known about the fundamental molecular mechanisms by which cells sense mechanical force or deformation, and transduce the mechanical signal into a biological response. Answering this fundamental question in biomechanics will provide a quantum leap in our understanding of the essential roles of mechanical forces in biology and medicine. A possible unifying mechanism for mechanosensing and mechanotransduction in living cells is protein deformation, broadly defined as protein conformational change under force [46, 47]. It has been well established that the three-dimensional conformation of a protein largely determines its function. However, the conformation of a protein can be altered by applied mechanical force, resulting in changes of the functional states of the protein and inducing down-stream biochemical and biological effects. Therefore, protein conformational change under mechanical force is an

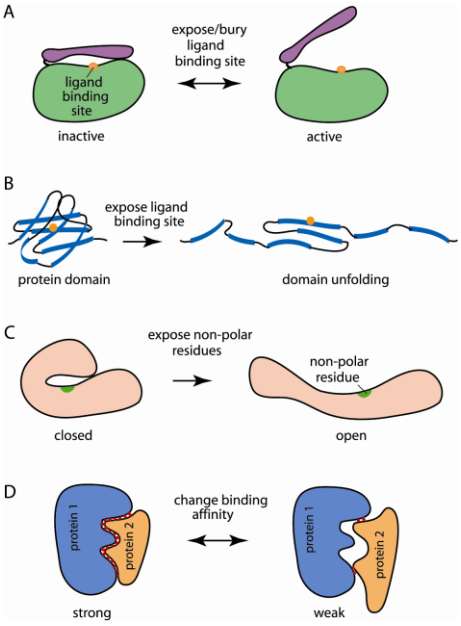

Figure 1: Examples of biological consequences of protein deformation. Mechanical forces can (**a**) switch a 'lid' in a protein from 'close' to 'open' position, or (**b**) unfold a protein domain, thus exposing the ligand binding site. Protein deformation could also (**c**) expose the non-polar residues, causing non-specific interaction between the protein domain and other biomolecules; or (**d**) induce a change in binding affinity, altering protein-protein interactions.

excellent candidate as the unifying molecular mechanism of mechanosensing and mechanotransduction in living cells [48].

Shown in Figure 1 are some examples of the possible effect of protein deformation in a living cell. Many proteins have specific ligand binding sites buried initially by a protein domain or a peptide (a 'lid'). As illustrated in Fig. 1a, upon applying mechanical forces to such a protein, the 'lid' opens, exposing the ligand binding site. The reverse is also true: protein deformation can close a 'lid' that is initially open, thereby burying the ligand binding site. Alternatively, a protein globular domain could unfold under mechanical force, exposing the ligand binding site that is buried inside the globular domain (Fig. 1b). Mechanical forces could also unfold a globular domain and thus expose the non-polar residues (Fig. 1c), which may cause non-specific interaction between the protein domain and other biomolecules, and thus alter protein function. It is well known that proteins interact with each other based on conformational matches: good conformational match leads to high binding specificity and affinity between two proteins, while poor conformational match does the reverse. As shown schematically in Fig. 1d, when proteins 1 and 2 have good conformational match, they have strong interactions to realize their functions, for example, to activate a signaling cascade, or facilitate an enzymatic activity. However, when one of the proteins, say, protein 2, sustains a force-induced conformational change, the interaction between proteins 1 and 2 becomes weak due to the poor conformational match, thus altering the function of protein 2. The reverse is also true: deformation of a protein could increase its affinity to another protein that otherwise would not interact due to the poor conformational match in its native state. This concept is not limited to protein-protein interactions; protein-DNA, protein-RNA and protein-small molecule interactions can be altered by force-induced protein conformational change as well.

Deformation and Constitutive Behavior of Cells: Over the last few decades, extensive experimental and modeling/simulation studies have been performed to determine the deformation of cells and tissues under applied force, and their constitutive behaviors [49]. Typical experimental set-ups for single-cell mechanical testing are shown in Figure 2. However, in most of the

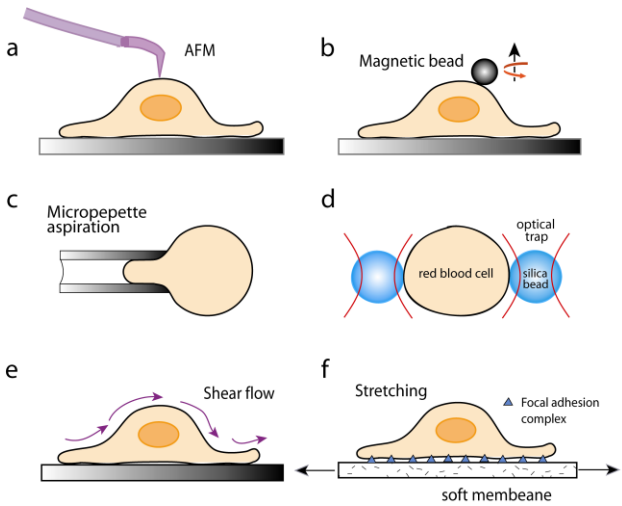

Figure 2: Schematic representation of the three types of experimental techniques used to probe living cells. Atomic force microscopy (AFM) (**a**) and magnetic twisting cytometry (MTC) (**b**) are Type A methods which can probe cell components at force resolution of 10^{-10} and 10^{-12} N, respectively, and displacement resolution of at least 1 nm. Micropipette aspiration (MA) (**c**) and optical trap (OT) (**d**) are type B techniques that can deform an entire cell at force resolution of 10^{-10} and 10^{-11} N, respectively. Shear flow (**e**) and substrate stretching (**f**) methods are capable of mechanical response evaluation of a population of cells.

modeling studies and constitutive equations developed for living cells, the active feature of living animal cells has been either ignored or poorly captured. It has been well established that most of the living animal cells are 'active' materials and structures, i.e., their structure, morphology and thus constitutive behaviors change with applied mechanical load. Cell structural changes, including structural alterations in cytoskeleton and changes in density and/or distribution of local adhesion complexes, may happen within a few minutes upon loading [50, 51]. Therefore, it is likely that as mechanical measurement of cells is being conducted, significant changes in cell structure and/or surface contact occur concurrently, leading to an altered force-deformation response of the cell. The degree of changes in cell deformation behavior depends on both the magnitude and rate of applied force. Adding to the complexity is that certain cells also have force-generating functions, which should be considered in the constitutive behavior of cells as well. Therefore, there is a critical need to develop better constitutive models for single-cell mechanical behavior, taken into account the active behavior of cells. However, it remains to be very challenging to quantify accurately the distribution of forces among various subcellular structures inside a living cell. It is well known that a significant portion of forces is supported as well as generated by the cell cytoskeleton, but cells are active and the cytoskeletal structures are dynamic; they could undergo remodeling or re-organization in response to mechanical perturbations. Further, the measurement of mechanical behavior of individual cells may give rise to different results, which may depend on cell morphology, stage in the cell cycle, as well as how different subcellular structures respond to mechanical perturbation. This raises a fundamental paradox: How can we measure mechanical behavior of living cells if they react to our measurement tools? These issues are fundamental to the study of the mechanics of living cells.

4. **Challenges in the Manufacturing of Nanostructured Materials** Mostafa Bedewy and A. John Hart

Research in nanomanufacturing aims to overcome the roadblocks to large-scale realization of novel materials and products utilizing the nanoscale material building blocks. On one hand, fundamental scientific contributions (*basic research*) are required for understanding the nanofabrication processes, and for developing lab-scale methods/instruments to precisely control the process and deterministically design the nanomaterial. This science aspect of nanomanufacturing is heavily reliant on advanced characterization techniques for studying the evolution of morphology during processing (*in situ*), as well as the correlation of the morphological evolution to real-time process kinetics (*operando*). On the other hand, these scientific findings

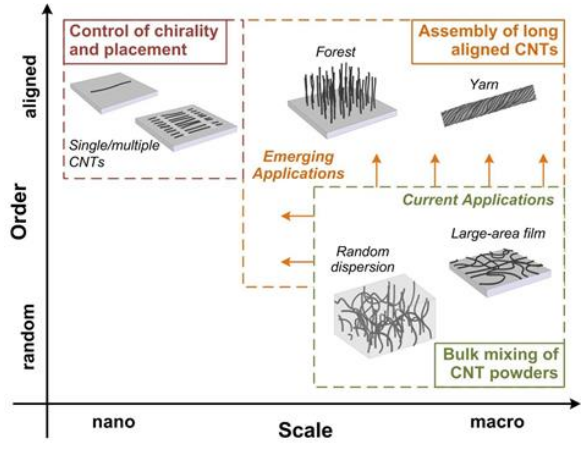

Figure 3: Mapping of current and emerging applications of CNTs, showing need for ordered and precisely controlled hierarchical CNT structures. Reproduced from De Volder et al. [52].

pave the way for developing high-throughput and cost-effective production technologies (*applied research*) that scale-up the lab method into either a continuous or batch production process. Ultimately, both fundamental and translational research are required to overcome roadblocks towards commercialization.

For example, commercial production of carbon nanotubes (CNTs) has rapidly increased over the past few years, reaching more than 2000 tons/year in 2011[52]. Nevertheless, the majority of commercial products containing CNTs utilize randomly oriented CNTs as additives to a matrix to improve the overall properties of the mixture (Fig. 3). Examples include adding CNTs to a polymer to render it conductive for use as electrostatic shields, or adding CNTs to a resin matrix to improve the mechanical properties for use as structural composites. On the other hand, many emerging applications are based on ordered CNT structures. Macroscopic structures that are composed of self-aligned CNTs forming a hierarchical morphology, such as CNT forests and yarns, are promising for integration in high-performance structural composites [53], electrical interconnects [54, 55], thermal interfaces [56], filtration membranes [57], gas sensors [58-60], oil-sorbent materials [61] and other applications.

CNT forests are typically grown by chemical vapor deposition (CVD), owing to the scalability and versatility of this method. Nevertheless, the actual morphology of a typical CVD-grown forest is composed of highly intertwined bundles of tortuous CNTs that exhibit a distribution of diameters and numbers of walls. Most promising applications of CNT forests require CNTs with high monodispersity, a high degree of alignment, and dense packing. For example, spatial mapping of the morphology of vertically aligned CNT forests by Synchrotron X-ray scattering and attenuation has revealed a continuous decrease in the average CNT diameter across the height of as-grown CNT forests [62-64]. Moreover, both the quantified alignment and density initially increase then decrease towards the forest bottom, following an S-shaped Gompertz curve of population growth (Fig. 4) [62, 65, 66]. These morphological features have recently been shown, both qualitatively and quantitatively, to limit the thermal and electrical

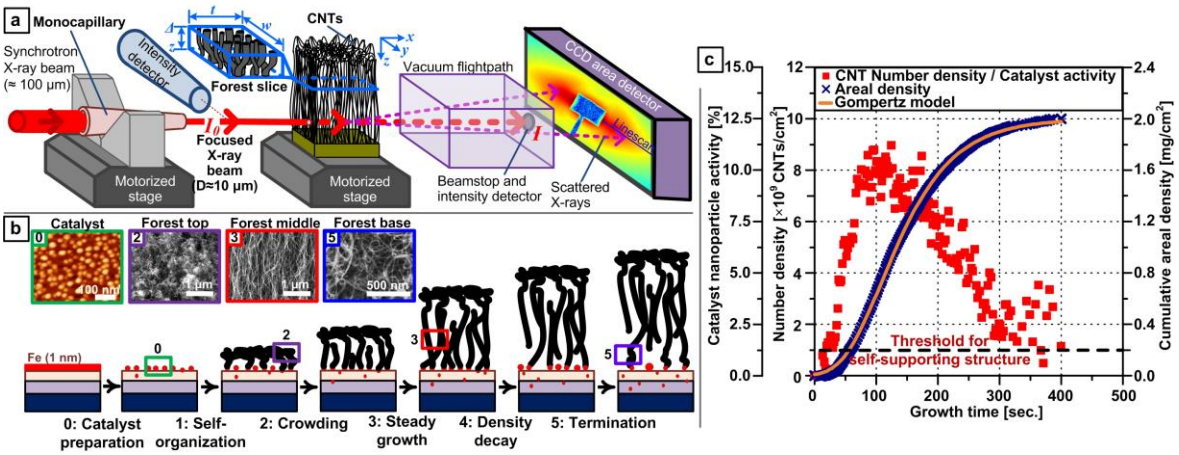

Figure 4: Spatiotemporal evolution of CNT forest morphology during CVD growth. (a) Schematic of CNT forest characterization by Small Angle X-ray Scattering (SAXS). (b) Successive stage of CNT growth according to a collective growth model. (c) Time evolution of forest density and catalyst activity, as well as the mass kinetics of growth. Reproduced from Bedewy et al. [65].

conductivities of CNT forests, highlighting the need for engineering the morphology of aligned CNTs during growth and self-organization [67, 68]. Importantly, only a small fraction of the catalyst nanoparticles bear CNTs and the rest are inactive, producing low-density structures (Fig. 4c), limiting the transport properties of CNT forests and preventing their utilization as electric interconnects [69].

Because a large number of CNTs grow simultaneously in a typical CVD process (more than 10^9 CNTs/cm^2), understanding the mechanical coupling effects during growth is key to overcoming the limiting growth mechanisms, as well as to engineering the collective mechanical properties. CNT forests behave like foams under mechanical loads, yet the characteristics of individual CNTs as well as the interactions among neighboring CNTs dictate both the deformation behavior and energy absorption. Accordingly, engineering the hierarchical morphology is required for tailoring the strength and toughness of multifunctional CNT assemblies [70-72].

Hence, overcoming these manufacturing roadblocks requires developing well-controlled synthesis processes, *in situ* characterization techniques, and comprehensive mathematical models. Such concerted effort could be targeted to address the following challenges:

- Understanding the atomic scale mechanism of CNT nucleation and catalytic activation, and its propagation across populations of catalyst particles that are necessary to grow dense self-organized CNT structures as high-performance interfaces.
- Identifying and overcoming the termination mechanism of CNT growth, thus enabling direct synthesis of ultra-long CNTs as mechanical fibers or electrical wires.
- Elucidating how mechanical properties of CNT structures relate to both the characteristics of individual CNTs, as well as their interactions within an ordered, or quasi-ordered, structure.
- Tuning CNT chirality and diameter of single-walled CNTs growing in large populations, and, moreover, achieving this in low-temperature and CMOS-compatible growth processes.
- Translating lab-scale methods to roll-to-roll manufacturing processes for CNT growth and printing, with considerations to quality, rate, and cost.
- Developing online quantitative metrology methods capable of measuring CNT diameter, alignment, and hierarchical organization, such as by electromagnetic methods or using compact X-ray sources.

These research challenges can be generalized to many nanomaterial systems, and are essential to accelerate progress toward both science and technology of nanomanufacturing. There is an important synergy among nanoscale/nanostructure fabrication, metrology, and process design and control. Intersection between these topics is essential to understand and implement strategies for scalable manufacturing of nanostructured materials across a wide range of applications and industries. For example, rigorous analyses of nanostructure synthesis methods such as CVD and chemical self-assembly are needed to establish repeatable outcomes. Quantitative characterization methods are needed for implementation in manufacturing settings,

overcoming the low throughput of electron microscopy and extreme capital investment required for most characterization instruments. This is especially a challenge for multi-phase systems such as nanostructures dispersed in polymers. Last, innovative process and machine designs are needed to scale-up laboratory methods, and to create high quality materials at accessible cost. These approaches must be accompanied with a keen vision toward the most disruptive commercial applications, while understanding the requirements for integration with broader manufacturing infrastructure such as in the composites and electronics industries.

5. **Mechanical Characterization of Nanostructures** H. D. Espinosa

Nanostructures, such as carbon-based nanomaterials (carbon nanotubes-CNTs, graphene and carbon nanofibers-CNFs) and nanowires (metallic and semiconducting), are envisioned as critical components in the next generation of advanced materials, electronic devices and autonomous sensor networks. For example, CNTs and graphene, which exhibit outstanding mechanical and electrical properties, are currently being studied as the building blocks of high-performance composite materials, next-generation electronics, and nano-electromechanical systems (NEMS). Crystalline nanowires that display enhanced mechanical properties, such as high moduli, fracture and yield strengths, as well as active properties, such as piezoelectricity and piezoresistivity, are envisioned as components of future electronics, energy harvesting architectures, and ultra-high density interconnects. Although the potential of all these nanostructures is well recognized, further understanding by means of mechanical characterization is highly needed in order to translate envisioned performances to everyday products.

Carbon-based nanomaterials, used in bio-inspired hierarchical nanocomposites, have the potential to achieve an unprecedented level of both strength and toughness given the outstanding properties of their building blocks (CNTs, graphene, CNFs). However, the engineering of their interactions and characterization across length scales are challenges that prevent the translation of the mechanical properties of the building blocks to larger scales [73].

On the other hand, nanowires, already demonstrated as building blocks of energy harvesting and 3-D non-planar electronic architectures, remain insufficiently characterized. In fact, knowledge of their mechanical and electromechanical properties is critical to achieve optimization of nanosystems and to establish unambiguous synthesis-structure-property relations. However, variations in the fabrication methods, and the resulting dopant concentration and defect density, lead to scatter in their measured properties. This is compounded with experimental challenges to achieve confident characterization of mechanical and electromechanical properties below 100 nanometers [74].

For all the aforementioned nanomaterials, however, significant efforts in the experimental, theoretical, and computational arenas are underway in order to address these challenges. To maximize the impact of these efforts, future research directions should address the following areas:

For CNT materials and nanowires, the understanding of the mechanical and coupled properties (electromechanical, electrothermal) will necessitate further development of novel characterization techniques [75, 76]. For CNT materials, modeling and experimentation of intermediate (meso-) scales, informed by atomistic and nanoscale characterization (i.e., multiscale modeling) is needed in order to facilitate the understanding of lateral interactions and the role of topology and hierarchy in mechanical properties. For nanowires, efforts need to be directed towards the understanding of size-effects of properties and failure modes below 100 nanometers [77], especially for interactions that couple mechanics, and other phenomena, e.g. piezoelectricity, which couples mechanical and electrical properties [74, 75]. New computational approaches need to be developed [78] to achieve accurate modeling of coupled behavior for realistic nanostructure sizes (10nm-100nm) and defect distribution. For both types of materials, rate-dependent studies are likely to have a significant impact, because composite materials usually require high-strain-rate failure tolerance [75], and nanoscale electronic architectures will impose MHz to GHz cycling on the nanostructures [75]. Furthermore, experiments at high strain rate have the potential to bridge the gap existing between the strain rates in experiments and atomistic simulations, thus allowing the validation of the empirical force fields used in computational models. Advances in techniques such as dynamic TEM, where nanosecond scales are already achievable, will allow the exploration of rate-dependent failure mechanisms in nanomaterials [75].

6. **Progress and Future Trends in Nano-Mechanical Testing of Materials** Julia R. Greer

A remarkable increase in the tensile strength of single-crystal metallic whiskers with decreasing whisker diameter was first reported almost a century ago by Taylor [79] and confirmed by Brenner's studies [80, 81] in the 1950s. Their research clearly demonstrated what is now well-known as a size effect in metals. Since then, there has been compelling experimental evidence that the mechanical response, and especially the strength, of nano-sized crystals with different initial microstructures is different from their bulk counterparts. For example, single crystalline metals exhibit a power-law dependence on sample size when dimensions are reduced to sub-micron levels, obeying a smaller is stronger trend [77, 82-84]. In contrast, nanocrystalline samples of the same metals – i.e. same-sized specimens with the same chemical composition but containing multiple grains, display the opposite trend: smaller is weaker [85-87]. Metallic glasses, which lack any sort of long-range or short-range order, were shown to undergo brittle-to-ductile transition when pulled in tension [88-92]. Most of these discoveries were brought to light by the nano-mechanical experiments, mainly uniaxial compression and less frequently, tension, on the so-called "micro- and nano-pillars." In these experiments a nanoindenter or a nanoindneter-like module, is used to compress cylindrical pillars with close to 3:1 aspect ratios that were mainly fabricated by the Focused Ion Beam with few notable exceptions.

The small-scale mechanical testing community has been focused on the problems of unraveling the physical origins of size-dependent strength in nano-scale solids, where the presence of surfaces causes the emergence of unexpected deformation mechanisms in response

to mechanical deformation. It has been shown that when the sample size is reduced not only vertically (i.e. thin films) but also laterally, the mechanical properties of single crystals, for example, drastically differ from those of their bulk counterparts. Such differences are thought to arise from the distinct defect behavior that emerges as a result of reducing material dimensions to the nano-scale and manifest themselves by causing unusual mechanical properties. These characteristics include avalanche-like stochastic stress-strain signatures, size-dependent strength, and tension-compression asymmetry - prevalent only in those structures where the surface area is significantly higher than their volume, i.e. sub-micron scale. These studies provide a powerful foundation for the fundamental deformation processes operating in these materials at small scales, but they are a far cry from representing real materials, whose microstructure is often complex, containing boundaries and interfaces. In fact, both homogeneous (grain boundaries, twin boundaries, etc.) and heterogeneous (phase boundaries, precipitate-matrix boundaries, and free surface) interfaces in size-limited features are crucial elements in structural reliability of most modern materials. Establishing the link between the size-dependent mechanical properties and microstructural evolution represents the grand challenge and scope of existing efforts.

As society moves towards employing architecture to create new materials with unprecedented properties, the critical length scales of individual structural members and of the material microstructure become comparable with one another, and leaps not strides need to be made to understand the governing deformation mechanisms in such material systems. Using architectural features to elicit desired functionality has already started shifting the material creation paradigm from structure/processing/property to property/architecture/fabrication. To ensure feasibility of this “reverse” material construction approach, it is imperative to understand and predict mechanical response of these “metamaterials” (i.e. materials whose properties are controlled by their engineered structure rather than by atomic composition alone), where the combination of feature size and material microstructure plays a critical role.

Modern fabrication techniques push the limit of scalable 3-dimensional architectured materials to smaller and smaller scales such that the characteristic length scale of material microstructure approaches the size of individual structural members. Recent work revealed the development of 3-dimensional architecture nano- and micro-lattices, spanning several length scales with individual members aiming at nanometer scales. For example, the fabrication of ultralight, hollow metallic and ceramic nano-lattices with extremely low densities has recently been reported, whose overall size ranged from hundreds of microns to several centimeters, the individual unit cell size [93, 94]. At these scales it is no longer accurate to individually focus on the mechanical properties stemming from the cellular structural design of the lattice and those from material induced behavior arising from the material’s microstructure; rather models have to be created that account for the material size effect when incorporating properties into structural mechanics models.

The creation of multi-scale predictive models that are experimentally informed and that account for the material and microstructure-induced size effect is likely to be the most important pursuit of the nano-mechanical community in the future. It is particularly important that a

fundamental understanding of the combined effects of feature size, the atomic make-up (microstructure), and loading conditions is developed to inform these models. This is where the "nano-pillar" testing is of key essence and benefit.

7. **Time and Scale Dependent Mechanical Behavior of Soft Nanomaterials** Ioannis Chasiotis

In recent years novel microfabrication methods have produced a multitude of polymeric nanostructures in the form of membranes, fibers and particles. Some of them such as electrospun nanofibers may have load bearing functionality, while others, although not intended to carry mechanics loads, still interface with their thermomechanical environment and inadvertently are subjected to mechanical deformation. For longer than a decade, the mechanics of materials community has embarked into experimental studies with nanostructures but the majority of the test structures have been comprised of ceramics or metals. Such advanced nanoscale experimental studies carried out in situ inside an SEM or a TEM have already challenged the existing boundaries in experimental mechanics imposed by specimen size. Such studies dealt with ceramics and metals which are fairly stable at small strains and many materials of interest, such as carbon nanotubes and nanofibers, are also insensitive to loading rate.

On the contrary, polymers and biological materials such as collagen are highly sensitive to strain rate and their mechanical behavior is time-dependent at small and large strains. To date, the mechanical response of soft nanostructures is an uncharted territory mainly due to the limited tools available for time-resolved studies. Under small deformations, the close connection between time and length scale effects on soft nanostructures has been shown to allow for a time-length scale superposition [95], hence providing evidence for a departure from bulk-like molecular conformations and entanglements due to the relatively large number of surface molecules in polymeric nanostructures. Such studies, although very limited, depart from the norm of classical glass transition temperature, T_g, studies [96] with ultra-thin films at length scales of the order of 50 nm or below, a dimension which for some polymers signifies the onset of molecularly confined behavior. This departure is important because the correlation between Tg and mechanical behavior is not as strong [97] and the shift in Tg of molecularly confined polymers does not provide any information about their large deformation response. Furthermore, the molecular structure of polymers is closely tied to fabrication conditions that influence the cross-link network, molecular entanglements, and degree of molecular homogeneity. For instance, small changes in the fabrication parameters of electrospun polymer nanofibers have a profound effect on their mechanical behavior [98]. In such cases, the synergy of molecular spectroscopy, mechanical experiments and modeling is required to understand the molecular structure vis-à-vis the fabrication routes so that meaningful fabrication-structure-property relationships can be derived.

The void in our knowledge of the mechanics of soft nanostructures is further accentuated by the lack of experimental evidence about the viscoplastic and rate dependent mechanical behavior

of sub-micron scale and nanoscale polymeric structures. Current experimental methods for small scale experimentation place major emphasis on in situ electron microscopy which, can provide important information about the material structure but is rarely used for quantitative strain measurements on nanostructures due to the susceptibility of the latter to the electron beam. Even more so, soft materials are susceptible to molecular degradation upon exposure to electron beam imaging. To this effect, recent advances in experimentation have allowed for time resolved mechanical deformation of soft nanomaterials [99] that unraveled unique modes of nanoscale deformation stemming from molecular heterogeneity [100]. Furthermore, the reduced molecular entanglement network in 1-D polymeric nanostructures allows for large viscoplastic deformations of otherwise macroscopically glassy polymers, which leads to concurrent material strengthening and toughening [101]. These important observations about the departure of the mechanical behavior of soft nanostructures from bulk, warrant further experimental and modeling research to understand the evolution of molecular structure under small and large stresses, and the role of the initial molecular state and defect structure that permit such major deviations from bulk behavior.

The aforementioned research challenges and insights are of high relevance to biological structures, such as protein molecules and their bundles [102], collagen fibrils [103], or individual cells [104], which are of hierarchical structure and highly heterogeneous, conditions that the experimental mechanics community has not dealt with in the past. Biological nanomaterials place strict restrictions on experimental conditions, as removal from their natural 3-D stress environment may not result in physiologically relevant results. Furthermore, the exact structure of many biological materials of interest is largely unknown, while the geometry of biological structures is far from that of ideal "specimens" for mechanical experiments. Studies have shown insensitivity of the mechanical behavior of soft nanofibers to surface roughness and geometrical irregularities and imperfections [105], which provides insight into the increased toughness and strength of natural nanomaterials, but also requires further studies at the molecular scale to establish connections between experimental observations and molecular mechanisms. Challenges stemming from the highly variable nature of the structure and chemistry of bio-nanomaterials [105] require statically significant experimental data, which, currently, are not possible due to the intricacies and challenges in conducting experiments at small scales. Detailed information about the chemical and molecular heterogeneity of bio-nanomaterials coupled with high fidelity, well-targeted, mechanical experiments would reduce the need for large sets of statistical data.

It is, thus, evident that the complexity soft nanostructures imposes major challenges but also represents a very fertile area in experimental mechanics of materials where new methods and the synergy with other disciplines will lead to key advances in soft nanomaterials that will revolutionize soft lithography, high performance nanofibers, biological scaffolds research, etc.

8. **Digital Image Correlation for Quantitative Measurements at the Nano-scale**
Michael A. Sutton

Measurements at the nano-scale have become a sort of *holy grail* in mechanics of materials. To be precise, one must provide an estimate for the spatial resolution of the measurements, which the author will attempt to do during the discussions. Given the limitations of diffraction-limited optical methods, which have spot sizes on the order of 200 nm or greater, the types of imaging modes which offer the potential for nano-scale measurements include Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (AFM). Synchrotron-based volumetric imaging methods (e.g., CT scanning using synchrotron radiation) typically have voxel limitations on the order of 100 nm, which generally would provide spatial resolution in the measurement data on the order of 1 μm^3 or larger. Though additional imaging modes will inevitably be developed that are capable of high resolution measurements, the focus here will be on AFM and SEM imaging systems which are generally available to the scientific community.

Modern SEM systems have reported electron beam spot sizes down to 1 nm or slightly smaller, so that ideally they can provide images with spatial resolution on the order of a nanometer. Even so, the interaction volume that gives rise to a specific recorded digital "intensity" value is generally much larger than the spot size, indicating that the image data provides a form of integrated response to the electron beam. It is noted that the interaction volume size can be adjusted by judicious choice of imaging parameters, such as voltage and current. Since SEM systems can magnify images well over 100,000X, imaging of a 1 μm^2 region on a specimen with spatial resolution of 1 nm/image pixel is readily achievable. In such cases, the primary factor affecting the accuracy of the SEM image-based methods are image distortions. Fortunately, good methods have been developed and shown to be effective in removing most of the effects of e-beam distortions [106-108]; recently, SEM-based measurements have been reported in the literature with accuracy of +/-0.01 pixels for displacement and 0.0002 for strain in some cases [109]. The primary factor affecting spatial resolution of the data is the ability to incorporate extremely small features (contrast) into the specimen region of interest. Recently, we have successfully applied a lovely 10 nm random pattern on a silicon specimen using recently developed approaches [110], and we are now attempting to apply random 2nm patterns. If a 2nm pattern can be applied, then the spatial resolution of the individual image-based measurements would be on the order of 20nm (about 100 atoms) or slightly smaller using subset-based matching methods. Issues that are as yet unknown include (a) stability of the 2nm pattern when subjected to e-beam radiation, (b) effectiveness of distortion correction methods at extremely high magnification and (c) optimal SEM imaging parameters for accurate measurements at these magnification levels.

AFM systems offer the potential for even higher magnification imaging and lower spatial resolution in the measurements, providing the ability to visualize the nano-structure of a material through detection of the interactions between the probe and a nearby surface. Unfortunately, the scanning process used in typical AFM systems introduces substantial image distortions that affect the accuracy of the image-based measurements. For example, recent AFM imaging by the

author indicated that the strain variability over each 300 nm^2 area was ~1500 με, even after removing distortions using the same approach as was employed with SEM images [111, 112]. Such variability would be acceptable when large strains are expected, but would fall short when seeking measurements of small elastic deformation. Clearly, additional modifications/improvements will be needed to increase AFM image stability and reduce distortions.

In summary, existing imaging modes offer the potential for displacement and deformation measurements with "nano-scale" spatial resolution. As the need for measurements is pushed to even smaller scales, it will be important to determine how changes in magnification affect the quality of the images obtained and hence the accuracy of the image-based measurements that is achievable.

9. **The State of the Art in Small Force Metrology** Gordon A. Shaw

Small forces are ubiquitous in nature; the forces between protons and electrons literally dictate the properties of matter. Measurement of these forces has implications for both fundamental and applied science. This summary is intended to provide a brief overview of several emerging areas that could have substantial impact on commerce in the US that are related to the metrology of force from piconewtons to micronewtons. It will focus on methodology that is traceable to the International System of Units (SI), as the industrial standards used to ensure equitability in the production and distribution of goods typically require measurements be made SI traceable. This is an internationally competitive area, and is being actively pursued by both the US and the EU [113].

The first area to consider is the impending redefinition of the SI [114]. Starting as early as 2017, the values of the fundamental constants (Planck's constant, the speed of light, etc.) will be fixed and the SI units (the Kilogram, the Ampere, etc.) will be derived from those fundamental constants. One of the implications of this is that the calibration of force within the SI no longer needs to rely directly on the use of calibrated mass sets. This is particularly advantageous for the measurement of small forces. Calibration and manipulation of the dust particle-sized masses necessary to accurately calibrate the nanonewton-scale forces that are used, for example, in materials characterization in atomic force microscopy (AFM) is prohibitively difficult. As part of the SI redefinition, force can be derived from approaches other than a mass in a gravitational field. One particularly promising approach involves the use of electrostatic force to generate a traceable calibration from electrical measurements [115]. These electrical measurements scale very well into the small force regime, and can even be miniaturized to provide an SI traceable measurement in-situ [116].

Another area where the redefinition has potential to impact the measurement of small force is in the measurement of laser power. There has long been an understanding that light exerts a force on objects it reflects from or is absorbed by. Recently, it has been shown that the relationship between laser power and mechanical force can be used for calibration. For very small forces, the power of a milliwatt-scale laser reflecting off a force sensor can be measured to provide a more

accurate calibration of force at the piconewton level [117, 118]. Conversely, at very high laser power, the measured photon pressure force could provide a better calibration of laser power used in laser machining or defensive weapons systems.

Once these new measurements are in place, another possibility is to use them to calibrate so-called intrinsic forces. These are forces that are linked to natural phenomena, and are often application specific. For example, recent work is shown that it is possible to calibrate a single molecule of DNA as a force sensor [119, 120]. Since DNA molecules can be replicated exactly using modern polymerase chain reaction (PCR) methods, this provides a cheap and easy way to make a calibration reference for biomolecular force measurements.

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