**SYMPOSIUM PM01**

Architected Materials—Synthesis, Characterization, Modeling and Optimal Design  
November 26 - November 29, 2018

**Symposium Organizers**  
Katia Bertoldi, Harvard University  
Tobias Schaedler, HRL Laboratories, LLC  
Lorenzo Valdevit, University of California, Irvine  
Martin Wegener, Karlsruhe Institute of Technology

* Invited Paper

**SESSION PM01.01: Shape Morphing and Origami/Kirigami Materials I**  
Session Chairs: Katia Bertoldi and Lorenzo Valdevit  
Monday Morning, November 26, 2018  
Hynes, Level 1, Room 102

8:00 AM *PM01.01.01*  
**Shape Morphing Metamaterials** Damiano Pasini; McGill University, Montreal, Quebec, Canada.

Thermal expansion can be challenging to control in applications that require thermal stability, yet it can be exploited to generate shape transformations that are highly predictable. In this talk, I will first present compliant building blocks that can deform under temperature, and then illustrate how their sequence can be encoded to attain macroscopic reconfigurations that are reversible. Investigated through theory and computations, the metamaterial behaviour is tested through proof-of-concept prototypes that show promise for deployable satellites, morphing components, and actuation devices.

8:30 AM **PM01.01.02**  
**Mechanical Instability Tuning in Architected Magnetoelastomers** Vincent Chen¹, ², Artemii Goshkoderia³, Matthew Robinson¹, ², Carson Willey¹, ², Stephan Radykh⁴, Ahgail Juhl⁵ and Phil Buskohl⁶; ¹Air Force Research Laboratory, Wpafb, Ohio, United States; ²UES, Inc., Dayton, Ohio, United States; ³Technion–Israel Institute of Technology, Haifa, Israel; ⁴University of Wisconsin–Madison, Madison, Wisconsin, United States.

Magnetoelastomers (MAEs) are an important class of soft, strain tolerant materials that generate a stiffness increase in response to a magnetic field. Stiffness tuning under magnetic field is advantageous due to the fast, reversible and non-contact control of the material properties, which is relevant for applications such as soft actuators, adaptive vibration dampers and acoustic filters. Architected MAE composites, such as laminates and periodic MAE inclusions in a non-active matrix, have been predicted to possess novel mechanical instabilities, due to the spatial distribution of stiffness mismatch and the ability to tune the mismatch with magnetic field. To demonstrate these concepts experimentally, we fabricated MAE composites using a commercial silicone as the non-responsive soft matrix and a silicone loaded with carbonyl iron particles for the stiff, magnetoactive regions. The silicone matrix underwent several modifications to increase the stiffness ratio between the soft encapsulating matrix and the stiff MAE regions, including tuning of the crosslinker to polymer ratio, incorporation of hydride-terminated silicone to promote a linear network, and addition of silicone oil to further reduce crosslinking. Laminates and 2D periodic MAE architectures were constructed through a series of drop casting and molding steps using 3D printed molds. A custom compression test jig was developed to systematically load the laminate specimen in the presence of a magnetic field. The study provides feedback on the sensitivity of the buckling strain to experimental specimen sizing/edge effects and provides broader insight on the practical integration of MAE instabilities into functional devices.

8:45 AM **PM01.01.03**  
**Field Responsive Architected Materials** Julie A. Mancini¹, ², Mark Messner¹, William L. Smith¹, Logan Bekker¹, Bryan Moran¹, A. M. Golobic¹, Andrew Pascall¹, Eric B. Duoss¹, Kenneth J. Loh² ³ and Christopher M. Spadaccini¹; ¹LLNL, Livermore, California, United States; ²Mechanical Engineering, University of California, Davis, Davis, California, United States; ³Argonne National Laboratory, Argonne, Illinois, United States; ⁴University of California, San Diego, San Diego, California, United States.

We present a method of creating field responsive architected materials by combining additive manufacturing with magnetorheological (MR) fluid. MR fluid, a fluid that commonly consists of oil and magnetic particles, goes through a change in its rheological properties under a magnetic field. This rheological change observed in the MR fluid is on the order of milliseconds and is highly reversible. We demonstrate these field responsive architected materials by infilling tubular cuboctahedron unit cells and lattices with MR fluid. The result is a dynamically tunable structure that can rapidly and reversibly change its effective stiffness through the simple application of a magnetic field without changing its overall form. We will also discuss a predictive model that we have created to aid in future design of these field responsive architected materials. This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. Supported by LDRD Strategic Initiative 14-SI-004. LLNL-ABS-753060.

9:00 AM **PM01.01.04**  
**3D Uniaxial Chiral Metamaterials with Ultra-long Characteristic Length Scale** Patrick Ziemke⁴ and Peter Gumbsch¹ ²; ¹Karlsruhe Institute of Technology, Karlsruhe, Germany; ²Fraunhofer Institute for Mechanics of Materials, Freiburg, Germany.
Ordinary linear elastic materials do not exhibit a chiral mechanical response. This means that they do not convert a stretch or a compression into a twisting deformation mode. Recent research into the fabrication and design of metamaterials has made this degree of freedom accessible. By stacking 3D laser printed unit cells in each spatial direction, Frenzel et al.[1] created samples of a chiral mechanical metamaterial and successfully mapped its behavior onto a micropolar continuum[2]. Micropolar continuum theory predicts that the magnitude of the twist decreases inversely proportional to the ratio of the sample size to the size of one unit cell. To maintain the global chiral response for larger samples, the scaling behavior must be manipulated to minimize the decay of the twist.

This can be achieved by either increasing the intrinsic chiral response of the material or by decreasing the coupling strength for the chiral component of the distortion. We attempt the latter and approach it with three-dimensional lattices based on a geometrically simple uniaxial chiral unit cell. Such simple structures can be modeled with reduced-order finite element formulations (cf. [3]) yielding an efficient numerical technique which allows the execution of extensive parameter studies with large three-dimensional samples.

We combine chiral unit cells with non-chiral coupling elements to form different lattices. Chiral and non-chiral elements can then be arranged just like in binary crystals forming CsCl or NaCl structures and the like. By testing different structures and tuning the properties of the different unit cells, we have designed lattice structures exhibiting an ultra-long decay-length compared to previously presented chiral mechanical metamaterials. In fact, some of our structures show an increasing chiral response at small numbers of unit cells and do not show a significant decay in the chiral response up to an edge length of 20 unit cells.


9:15 AM PM01.01.05 Deployable Multi-State Structures via Pre-Strained, Multistable Elements Jochen Mueller1, 2, Jennifer Lewis1, 2 and Katia Bertoldi1, 2; 1John A. Paulson School of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts, United States; 2Wyss Institute for Biologically Inspired Engineering, Harvard University, Cambridge, Massachusetts, United States.

Inspired by the shape memory of crumpled sheets and prior art on multistable structures, we present a new type of architectural metamaterial that can be fabricated flat and buckle out-of-plane to form complex shapes with defined load bearing capacity. The material is composed of discrete elements, of which the sides are exposed to different levels of pre-strain, locally induced during fabrication. Through experiment and simulation, we show that the elements buckle out of plane into different, stable states when exposed to small, orthogonal loads. The resulting, global shape can be pre-programmed via local pre-stress levels or changed on the fly by the activation sequence. By adding additional hierarchy levels, large displacements are achieved. All deformations are elastic and, hence, reversible. This reprogrammable and versatile material is expected to find application in a range of small and large-scale structures, such as deployable space frames and robotic manipulators.

9:30 AM BREAK

10:00 AM *PM01.01.06 Morphing Sheets into Freeform Objects, at the Micro- and Macro-Scales Chiara Daraio; California Institute of Technology, Pasadena, California, United States.

Morphing two-dimensional sheets into three-dimensional objects is a classical problem in mechanics, mathematics and art. Today, the ability to manufacture two-dimensional materials with an almost arbitrary microstructure, architecture and pre-stress distribution opens the door to new approaches for bending sheets into complex forms. In this talk, I will review recent progress in the design of micro- and macro-scale, nonuniform sheets that can bend into freeform objects. Engineering the distribution of residual stresses, stiffness gradients and/or cut patterns, we control the buckling of sheets at both local and global scales. The designed distribution of responsive materials within the sheets provides a time dependent control of the developing shapes. I will discuss examples of sheets that change shape in response to environmental stimuli or the application of point loads. Programming 2D sheets into rigid, 3D geometries expands the potential of existing manufacturing tools for efficient and versatile production of 3D objects.

10:30 AM *PM01.01.07 Atomic Origami—A Technology Platform for Nanoscale Machines, Sensors and Robots Itai Cohen; Cornell University, Ithaca, New York, United States.

What would we be able to do if we could build cell-scale machines that sense, interact, and control their micro environment?

In Richard Feynman’s classic talk "There's Plenty of Room at the Bottom" he foretold of the coming revolution in the miniaturization of electronics components. This vision is largely being achieved and pushed to its ultimate limit as Moore's Law comes to an end. In this same lecture, Feynman also points to the possibilities that would be opened by the miniaturization of machines. This vision, while far from being realized, is equally as tantalizing. For example, even achieving miniaturization to micron length scales would open the door to machines that can interface with biological organisms through biochemical interactions, as well as machines that self-organize into superstructures with mechanical, optical, and wetting properties that can be altered in real time. If these machines can be interfaced with electronics, then at the 10's of micron scale alone, semiconductor devices are small enough that we could put the computational power of the spaceship Voyager onto a machine that could be injected into the body. Such robots could have on board detectors, power sources, and processors that enable them to make decisions based on their local environment allowing them to be completely unhethered from the outside world.

In this talk I will describe the work our collaboration is doing to develop a new platform for the construction of micron sized origami machines that change shape in fractions of a second in response to environmental stimuli. The enabling technologies behind our machines are graphene-glass and graphene-platinum bimorphs. These ultra-thin bimorphs bend to micron radii of curvature in response to small strain differentials. By patterning thick rigid panels on top of bimorphs, we localize bending to the unpatterned regions to produce folds. Using panels and bimorphs, we can scale down existing origami patterns to produce a wide range of machines. These machines can sense their environments, respond, and perform useful functions on time and length scales comparable to microscale biological organisms. With the incorporation of electronic, photonic, and chemical payloads, these basic elements will become a powerful platform for robotics at the micron scale. As such, I will close by offering a few forward looking proposals to use these machines as basic
programmable elements for the assembly of multifunctional materials and surfaces with tunable mechanical, optical, hydrophilic properties.

11:00 AM PM01.01.08
Reconfigurable Rotationally Symmetric Kirigami Structures and Their Applications Erin Evks, Dilara Meli, Tristan Blanzy and Max Shlein; Materials Science and Engineering, University of Michigan, Ann Arbor, Michigan, United States.

Kirigami design principles increasingly are being used to engineer auxetic materials and structures to achieve 2D to 3D transformations and precisely tune their physical properties. Here, we design, fabricate, and characterize rotationally symmetric kirigami (RSK) structures with complex elasticity, allowing for deformations of planar sheets to approximate curved surfaces and reversibly undergo large deflections. A combination of theory, finite element modeling, and experiments are used to depict the relationship between the cut pattern and the structure’s cross-plane deformation. We classify the RSK structures based on the order of rotational symmetry, defined by the number of sides and cuts along the perimeter. While in the planar, relaxed state, RSK structures exhibit inversion symmetry; this is broken upon cross-plane deformation, a property that may be of interest in controlling wave propagation. The deformed state can be interpreted as a superposition of deformed sub-units, in which the total deflection is a summation of the individual rings’ displacements. The rings can further be partitioned into beams that correspond to the cut geometry, which strongly influences the mechanical properties. We find the effective spring constant decreases with radial cut density but increases with azimuthal cut density, resulting in a theoretical 200-fold reduction in stiffness over the noncut membrane. To demonstrate the electrical characteristics and feasibility of RSK structures for flexible electronic applications, we fabricate conductive nets that maintain electrical properties even under large strains, well beyond 100,000% of the original profile. We also show the reconfigurability of the structures using thermally activated shape memory polymers and analyze their fatigue mechanism.

11:15 AM PM01.01.09
Inflatable Origami-Inspired Structures David Melancon1, Chuck Hoberman1,2, Jason Ku3, Erik Demaine4 and Katia Bertoldi1; 1John A. Paulson School of Engineering and Applied Sciences, Harvard University, Cambridge, Massachusetts, United States; 2Graduate School of Design, Harvard University, Cambridge, Massachusetts, United States; 3Wyss Institute, Boston, Massachusetts, United States; 4Computer Science and Artificial Intelligence Laboratory, Massachusetts Institute of Technology, Cambridge, Massachusetts, United States.

Origami has long been used as a source of inspiration to design creative and esthetic constructions, from the iconic paper swan to facades of multi-story buildings. More recently, the rules of folding have been applied to fabricate architected materials with functional properties such as compactness, self-foldability, and multi-stability. These properties highlight the potential of origami to become a new design paradigm for rapidly deployable structures. Whereas multiple origami-inspired deployable surfaces have been reported in the literature, there is a lack of research on enclosed deployable geometries. In this work, we introduce a novel type of inflatable origami-inspired structure comprised of a polyhedron with triangular faces and elastic hinges. From simple geometry principles, this star-shaped structure possesses two compatible configurations – flat-folded and deployed – giving rise to a bi-stable behavior. Based on experiments of prototypes and simulations of an energy-based model, we characterize the mechanics of the deployment and explore the design space of this origami-inspired structure. The insights gained from the study of this simple geometry enable the understanding of the folding principles of a novel class of enclosed origami-inspired structures that can be deployed to different stable configurations through inflation.

11:30 AM PM01.01.10
Kirigami Inspired-Metamaterials—From Morphable Structures to Soft Robots Katia Bertoldi; Harvard University, Cambridge, Massachusetts, United States.

In recent years kirigami has become an emergent tool to design programmable and reconfigurable mechanical metamaterials. Kirigami-inspired metamaterials allow the practitioner to exploit cuts in addition to folds to achieve large deformations and create 3D objects from a flat sheet. Therefore, kirigami principles have been exploited to design highly stretchable devices and morphable structures. In this talk I will show that precreased folds are not necessary to form complex 3D patterns in kirigami, as mechanical instabilities in flat sheets with an embedded array of cuts can result in out-of-plane deformation. Furthermore, by largely stretching the buckled perforated sheets, plastic strains develop in the ligaments. This gives rise to the formation of kirigami sheets comprising periodic distribution of cuts and permanent folds. As such, the proposed buckling-induced pop-up strategy points to a simple route for manufacturing complex morphable structures out of flat perforated sheets. Finally, I will also show that kirigami principles enable the design of morphable and transformable skins that facilitate the design of soft robots capable of locomotion.

11:45 AM PM01.01.11
Kirigami Inspired Self-Folding Arif M. Abdullah1 and K. Jimmy Hsia2; 1Mechanical Science and Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois, United States; 2Mechanical Engineering, Carnegie Mellon University, Pittsburgh, Pennsylvania, United States.

Realization of complex programmable metamaterials where the structural characteristics and functionalities could be tuned beyond their original, as fabricated design remains as one of the challenging problems within the research field of architected materials. To that end, Kirigami - the art of cutting and folding flat sheets of paper, provides scalable routes to generate intricate three-dimensional shapes from thin, planar (two-dimensional) sheets of materials. Researchers have utilized mechanical force actuated Kirigami structures in functional disciplines as diverse as energy harvesting, actuation, optics, stretchable electronics, and soft robotics. Achieving shape reconfiguration of freestanding, Kirigami-cut sheets in a stimuli-responsive, autonomous manner would not only enable new functionalities but also contribute to self-assembly.

This work aims to understand the self-folding behavior of Kirigami-cut bilayers where one layer isotropically expands with respect to the other in response to an external stimulus. We investigate two distinct types of cut geometries namely squares with radial cuts and rectangles with side cuts through a combination of nonlinear finite element modeling and experiments with soft polymeric systems. The Kirigami cuts decompose the pristine squares and rectangles into interconnected beams (length >> width) and plates (length ~ width) of varying aspect ratios. Our finite element calculations reveal that it is possible to tune both the bending direction and curvature values of each individual geometric units within the Kirigami-cut structures and thus transform the bilayers into complex three-dimensional architectures with spatially varying bi-directional curvatures in an on-demand manner. To experimentally demonstrate the potential of our approach, we design planar bilayered samples with side cuts and swell them in organic solvents to generate letters from the English alphabet to make up "UIUC" (University of Illinois at Urbana-Champaign) and "MRS" (Materials Research Society). We also design bilayers with radial cuts, and as they transform shapes with varying mismatch strains (solvent concentrations), we show that it is possible to use them as freestanding tunable optical systems where the transmission and reflection windows for incident light could be controlled through the deformation behavior of individual geometric units between the cuts. We also use a combination of the cuts to realize polyhedral shapes (such as tetrahedron and cube) through the self-folding of planar bilayers. The design principles proposed in this work would be applicable to a variety of material systems across length scales and contribute toward the development of smart programmable metamaterials.
Ordered sequences of motions govern the morphological transitions of a wide variety of natural and man-made systems, while the ability to interpret time-ordered signals underlies future smart materials that can be (re)programmed and process information. Here we introduce two novel classes of mechanical metamaterials, that can (1) exhibit sequential output and (2) are sensitive to sequential input. To obtain metamaterials that translate a global uniform compression into a precise multistep pathway of reconfigurations, we combine strongly nonlinear mechanical elements with a multimodal hierarchical structure, and demonstrate multi-step reconfigurations of digitally manufactured metamaterials. To obtain metamaterials that are sensitive to a sequence of mechanical inputs, we introduce the notion of non-commuting metamaterials, and show their capability for storing and processing information. Our work establishes generic principles for infusing metamaterials with sequential input and output.

2:00 PM PM01.02.02
In Situ Tunable Stiffness Using Multistable Kirigami Metamaterials Yi Yang1, Marcelo Dias2 and Douglas Holmes3; 1Boston University, Boston, Massachusetts, United States; 2Aarhus University, Aarhus, Denmark.

Materials with in situ tunable stiffness are needed for engineering programmable materials, shape-shifting structures, artificial muscles, and soft robotic actuators. While most examples of materials with in situ tunable stiffness focus on stimulus-responsive material properties, recent investigations on reconfigurable mechanical metamaterials opened another door to attain tunable material properties by systematically programming the microstructure of a constituent material. The mechanical properties of these architected materials depend on the topology of the substructure regardless of the constituent materials used. By introducing morphological reconfigurables into the unit cell, reprogrammable and reconfigurable metamaterials can be attained. Among various types of mechanical metamaterial, kirigami and origami metamaterials attracted tremendous attention due to its robust and straightforward ability to transform 2D sheets into 3D structures. Compared with its sister, origami metamaterials which have been extensively studied, understanding the mechanical behavior of kirigami metamaterials is limited. In this study, through a combination of experiments, simulation, and theoretical analysis, we demonstrate how a multistable microstructure inspired by kirigami provides a new design approach. By changing the spacing between the adjacent slits in the conventional linear parallel cutting patterns, we obtain multistable kirigami lattice structures composed of repeating unit cells whose structure is endowed with a bistable snap-through mechanism. Each stable state of the mechanical metamaterial exhibits a corresponding stiffness. By switching unit cells between the two stable states, we can tune the stiffness of this kirigami metamaterial in situ by a factor of five. Since this multistable kirigami approach is material independent, it could be integrated with stimulus-responsive materials, 3D printing technique, or combined with origami structures to be applied in multifunctional materials, deployable space structures, soft robotics, and biomedicine.

2:15 PM PM01.02.03
Observation of Mechanical Activity in a 3D Chiral Metamaterial Tobias Frenzel1, Muamer Kadic1,2 and Martin Wegener1; 1Institute of Applied Physics, Karlsruhe Institute of Technology, Karlsruhe, Germany; 2Institute of Nanotechnology, Karlsruhe Institute of Technology, Karlsruhe, Germany; 3FEMTO-ST, Université de Bourgogne Franche-Comté, Besançon, France.

In a medium exhibiting optical activity, an incident transverse linear polarization of a light wave rotates because the eigepolarizations of the chiral medium are not linear but rather circular, with a lifted degeneracy between left- and right-handed circular modes. Therefore, a linear incident polarization must be decomposed into the two circular eigepolarizations, which propagate with different phase velocities. The resulting phase difference accumulated during propagation leads to a periodic rotation of the linear polarization axis versus the propagation coordinate. A rotation by 90 degrees corresponds to mode conversion. Regarding elasticity, a different kind of mode conversion has recently been observed in two-dimensional (2D) centrosymmetric metamaterials [1]. There, the conversion between in-plane longitudinal (compression) and in-plane transverse (shear) modes did not require chirality. Chirality is crucial in quasi 2D monolayers of non-centrosymmetric tungsten diselenide crystals, for which the presence of chiral phonons [2] was deduced via selection rules of optical transitions.

In this contribution, we report the first experimental observation of mechanical activity, the elastic counterpart of optical activity, in 3D chiral polymer microlattices [3]. We have previously investigated related 3D chiral micropolar metamaterial samples made by 3D laser nanoprinting in the static regime [4]. Our present experimental approach in the dynamic regime is based on cross correlations of microscopy images taken under phase-delayed synchronized stroboscopic illumination with a light-emitting diode and sinusoidal excitation of the sample with a piezoelectric actuator. This approach is distinct from the established mapping of out-of-plane surface excitations by interferometric laser detection and laser excitation (see, e.g., [5]). For an incident linearly polarized transverse elastic wave, we demonstrate the polarization rotation by 90 degrees at 225 kHz frequency over as few as 10 unit cells in the axial direction, with 3-by-3 unit cells in the cross section. The experimental results are in good agreement with numerical finite-element calculations for finite beams as well as with band structure calculations for fictitious infinitely extended beams. These experiments pave the road for molding the polarization and the propagation direction of elastic waves in three dimensions by micropolar mechanical metamaterials.

system of batteries, sensors, microprocessors, and actuators, nature achieve responsiveness via control logic that is embodied in the compositional (material) and structural (geometric) properties of these materials. Inspired by nature, we combine anisotropic architected materials with geometric nonlinearity to design structures that precisely and sequentially self-actuate in response to multiple stimuli. We achieve this by 3D printing structures with geometric parameters that lie near nonlinear bifurcation points associated with a transition between bistable and monostable mechanical behaviors. We fabricate these structures using direct ink writing (DIW) with microfibrous architected materials, which are highly anisotropic due to shear-induced alignment of the fibers during printing. As a result, the materials swell anisotropically when exposed to suitable stimuli (non-polar solvents or water), which alters a key geometric control parameter defining the nonlinear behavior. If the control parameter passes through specific bifurcation points, rapid, large-amplitude self-actuation events can be triggered to occur at specific times, which can be harnessed to impart autonomous functional changes to structures. We demonstrate the utility of this bioinspired autonomous control strategy with several examples of structures that respond to their environment according to their embodied logic, without electronics, external control systems, or tethering.

2:45 PM PM01.02.05
Origami and 4D Printing of Elastomer-Derived Ceramic Structures Guo Liu, Yan Zhao, Ge Wu and Jian Lu, City University of Hong Kong, Hong Kong, Hong Kong.

Four-dimensional (4D) printing involves conventional three-dimensional (3D) printing followed by a self-shaping assembly step. It enables more complex shapes to be created than is possible with conventional 3D printing, and shape-morphing capabilities can improve the adaptability of structural materials to versatile application environments. However, 3D-printed ceramic precursors are usually difficult to be deformed, hindering the development of 4D printing for ceramics. To overcome this limitation, we developed elastomers which can be printed, deformed, and then transformed into ceramics, making the growth of complex ceramic origami and 4D-printed ceramic structures possible. In addition, strength-scalability synergy is achieved in the resultant ceramic architectures. This work starts a new chapter of printing geometrically complex and mechanically robust ceramics, and this concept is cost-efficient in terms of time when a series of complex-shaped ceramics with similar geometries were required. With shape-morphing capabilities of elastomers, this work on origami and 4D printing of elastomer-derived ceramics (EDCs) could lead to structural applications of autonomous morphing structures, aerospace propulsion components, space exploration, electronic devices, and high-temperature microelectromechanical systems.

3:00 PM BREAK

3:30 PM PM01.03.01
Additive Manufacturing and Architected Materials—Design, Fabrication, Materials and Performance Christopher M. Spadaccini; Lawrence Livermore National Laboratory, Livermore, California, United States.

Material properties are governed by the chemical composition and spatial arrangement of constituent elements. Over the past decade, the field of architected materials has sought to design, fabricate, and demonstrate materials with performance that is fundamentally controlled by geometry at multiple length-scales rather than chemical composition alone. There have been many advancements ranging from the maturation of additive manufacturing technologies which can be used to realize these materials, inverse design methods such as topology optimization, and unique new material feedstocks which make up the structures. This presentation will touch on all aspects of the architected materials realization process as well as evaluate performance of some of those materials. Specifically, we have demonstrated designer properties of these architected materials in polymers, metals, ceramics and combinations thereof. In addition to novel properties such as ultra-stiff lightweight materials, negative stiffness, and negative thermal expansion, I will present multifunctional architected materials with energy storage capability and architectures that respond to external fields. Many of these architected materials derived from advanced design and optimization methods which we have been developing and were fabricated with custom additive manufacturing techniques. These include projection microstereolithography (PμSL), direct ink writing (DIW), electrophoretic deposition (EPD), volumetric additive manufacturing, computed axial lithography (CAL), and diode-based additive manufacturing (DiAM) to name a few. New materials including graphene aerogel, carbon fiber composite, and printed glass will also be touched upon.

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4:00 PM PM01.03.02
Architected Nanocomposites as Model Materials for Armor Systems Anna Guell Izard, Jens Bauer and Lorenzo Valdevit; Mechanical and Aerospace Engineering, University of California, Irvine, Irvine, California, United States.

Armor systems must be carefully designed to mitigate two threats: blast and penetration. A successful armor dissipates the kinetic energy of the blast via plastic deformation while being able to resist the penetration of a projectile. These functional requirements require a material with high toughness and hardness, two properties that are generally mutually exclusive in monolithic materials. Current armor solutions consist of multi-layer systems encompassing one or more metallic or polymeric phases for energy absorption and a ceramic phase for penetration resistance. Here we propose a novel strategy based on the optimal design of functionally graded micro/nano-architected ceramic/polymer or ceramic/metal composites. The optimal intertwining of the two phases at the nanoscale has the potential to vastly outperform current solutions, while reducing weight. In this talk, we will present the performance of ceramic/metal and ceramic/polymer architected nanocomposites based on ordered lattice topologies and spinalod architectures.

4:15 PM PM01.03.03
Mechanical Behaviour of Architected Auxetic Hybrid Lattice Structures Frédéric Albertini1, Justin Dirrenberger2, Andrey Molotnitskiy2 and Cyrille Sollogoub2; 1PMM, CNRS, Paris, France; 2Material Science and Engineering, Monash University, Clayton, Victoria, Australia.

Architected Materials are an emerging class of advanced materials that bring new possibilities in terms of functional properties, filling gaps in Ashby’s material performance maps. The term architected materials describes any heterogeneous material that exhibits improved specific properties due to a thoughtful and predetermined morphology and/or topology design. This usually induces characteristic length-scales comparable to the size of the final
component being produced, i.e. the millimetre scale in the case of lattice structures. Different strategies have been studied in the literature for mitigating the surface defects of additively manufactured metallic lattices: chemical etching, electro-erosion, mechanical polishing. A new proposition is presented in this work: polymer coating or embedding of metal struts, by analogy to the soft-hard turtle-like strategy for mitigating crack propagation. Besides processing of such architectured lattice structures, the present work brings experimental and numerical results concerning the mechanical behaviour in compression for negative Poisson’s ratio lattices, also known as auxetics. As a matter of fact, one engineering challenge is to predict the effective mechanical properties of architectured materials; computational homogenization using finite element analysis is a powerful tool to do so when considering quasi-static behaviour; difficulties arise when analysing the effective damping behaviour. A straightforward solution is to rely on full-field finite element dynamic simulation, accounting for both the intrinsic viscoelastic damping of the constitutive material, as well as the structural damping due to the geometrical definition of the lattice structure considered in the present work. Homogenised behaviour of architectured materials can thus be used in large structural computations, hence enabling the dissemination of such materials in the industry. Comparison is made between the metal and hybrid lattice structures.

4:30 PM PM01.03.04
Architected Viscoelastic Impact Attenuators for Sports Padding Applications Eric C. Clough1,2, Zak Eckel1, Alvin Escober1, Jacob Hundley1 and Tobias Schaedler1; 1HRL Laboratories, Malibu, California, United States; 2Materials, University of California Santa Barbara, Santa Barbara, California, United States.

Mitigating injury as a result of an impact between multiple players or between a player and the ground presents a significant challenge to the sports protective equipment designer. These designs must consider multiple injury criteria (e.g. concentrated loads, peak acceleration, peak torque) for multiple successive impacts, while balancing human factors such as comfort and player mobility. For padding components, maximal volumetric energy absorption is key to achieving impact attenuation performance without sacrificing player mobility. Typical padding materials include urethane and vinyl nitrile closed-cell foams which attenuate impacts through the collapse of internal pores when compressed. The stochastic internal geometry of foams limits optimal design of foam pads to essentially two design variables: composition and apparent density. Conversely, lattices provide the designer with a host of architectural degrees of freedom enabling the optimal design of lattice pads that can significantly outperform foams through tuning of buckling and post-buckling response. The open-cell architecture of lattices additionally affords improvements in airflow for comfort or enhanced energy absorption. In this talk, we will present our recent work on the design, fabrication, and optimization of lattice padding materials fabricated via HRL’s rapid and scalable self-propagating photopolymer waveguide process. We demonstrate that by optimal design of the architecture, in conjunction with tuning the viscoelastic properties of the parent solid, lattice pads with impact attenuation performance exceeding 1.3X that of state-of-the-art foam padding can be achieved.

4:45 PM PM01.03.05
Plate Mechanical Metamaterials and Their Applications Igor Bargatin; Mechanical Engineering and Applied Mechanics, University of Pennsylvania, Wynnewood, Pennsylvania, United States.

Recently, we introduced the concept of plate mechanical metamaterials—cellular plates with carefully controlled periodic geometry and unique mechanical properties—as well as its initial realization in the form of freestanding corrugated plates made out of an ultrathin film. We used atomic layer deposition (ALD) and microfabrication techniques to make robust plates out of a single continuous ALD layer with cm-scale lateral dimensions and thicknesses between 25 and 100 nm, creating the thinnest freestanding plates that can be picked up by hand. More recently, we also fabricated and characterized plate metamaterials made from multiple layers of nanoscale thickness, whose geometry and properties are reminiscent of honeycomb sandwich plates or corrugated paper cardboard. The two layers are offset from each other but at the same time are connected using vertical-wall webbing, which prevents shear of the two layers with respect to one another. As a result, these “nanocardboard” plates orders-of-magnitude higher bending stiffness than the single-layer structures we reported earlier, while still possessing extremely low weight (as low as 0.5 g/m²) and mechanical robustness. The increase in the bending stiffness is expected, and its mechanism is similar to that used in conventional honeycomb sandwich plates, which offer the best possible combination of high bending stiffness and low mass. However, in contrast to sandwich composite plates, our nanoscale two-layer mechanical metamaterials can sustain extremely large deformations without fracture, fully recovering their original shape and not displaying any signs of internal damage.

Like the nanotruss-based mechanical metamaterials reported by other groups, plate mechanical metamaterials are extremely lightweight and resilient due to their nanoscale thickness and microscale cellular structure. However, in contrast to the cube-shaped metamaterials that typically form a lattice easily penetrated by the ambient air, our plates form continuous flat plates. Ultralow weight, mechanical robustness, thermal insulation, as well as chemical and thermal stability of alumina make plate metamaterials attractive for numerous applications, including structural elements in flying microrobots and interstellar light sails, high-temperature thermal insulation in energy converters, photophoretic levitation, as well as ultrathin MEMS/NEMS sensors and resonators. I will briefly discuss our experimental progress on all these applications, including demonstration of extremely robust thermal insulators that can sustain a temperature difference of ~1000°C across a micron-scale gap, macroscopic plates that levitate when illuminated by light, and hollow AFM cantilevers that offer greatly enhanced sensitivity and data acquisition rates.

SESSION PM01.04: Micro/Nano-Architected Materials II
Session Chairs: Christopher Spadaccini and Lorenzo Valdevit
Tuesday Morning, November 27, 2018
Hynes, Level 1, Room 102

8:00 AM *PM01.04.01

Creation of extremely strong and simultaneously ultra lightweight materials can be achieved by incorporating architecture into material design. We fabricate three-dimensional (3D) nano-architectures, i.e. nanolattices, whose constituents vary in size from several nanometers to tens of microns to centimeters. These nanolattices can exhibit superior thermal, photonic, electrochemical, and mechanical properties at extremely low mass densities (lighter than aerogels), which renders them ideal for many scientific pursuits and technological applications. The dominant properties of such meta-materials, where individual constituent size at each relevant scale (atoms to nanometers to microns) is comparable to the characteristic microstructural length scale of the constituent solid, are largely unknown because of their multi-scale nature. To harness the beneficial properties of 3D nano-architected meta-materials, it
is critical to assess properties at each relevant scale while capturing the overall structural complexity. We describe the fabrication and synthesis using Additive Manufacturing (AM) techniques, as well as the mechanical, biochemical, electrochemical, and thermal properties of nanolattices made of different materials with varying microstructural detail. Attention is focused on uncovering the synergy between the internal atomic-level microstructure and the nano-sized external dimensionality, where competing material- and structure-induced size effects drive overall response and govern these properties. Specific discussion topics include the nanofabrication and characterization of (often hierarchical) three-dimensional nano-architected meta-materials and their applications in chemical and biological devices, ultra-lightweight energy storage systems, damage-tolerant fabrics, and photonic crystals.

8:30 AM PM01.04.02
Nanocrystalline Aluminum Truss Cores for Lightweight Sandwich Structures Tobias Schaedler1, Lisa Char2, Eric C. Clough3, Morgan A. Stilke1, Lawrence J. Masur4 and Jacob Hundleby5; 1HRL Laboratories, LLC, Malibu, California, United States; 2Xtallic Corporation, Marlborough, Massachusetts, United States.

Substitution of conventional honeycomb—composite sandwich structures with lighter alternatives has the potential to reduce the mass of future vehicles. Here we demonstrate nanocrystalline aluminum - manganese truss cores that achieve 2 - 4 times higher strength than aluminum alloy 5056 honeycombs of the same density. The scalable fabrication approach starts with additive manufacturing of polymer templates, followed by electrodeposition of nanocrystalline Al-Mn alloy, removal of the polymer, and facesheet integration. This facilitates curved and net-shaped sandwich structures, as well as co-curing of the facesheets, which eliminates the need for extra adhesive. The nanocrystalline Al-Mn alloy thin -film material exhibits high strength and ductility and can be converted into a three-dimensional hollow truss structure with this approach. Ultra-lightweight sandwich structures are of interest for a range of applications in aerospace, such as fairings, wings, and flaps, as well as for the automotive and sports industries.

8:45 AM PM01.04.03
Additive Manufacturing and Design of Multi-Functional 3D Architected Metamaterials Xiaoyu Zheng; Virginia Tech, Blacksburg, Virginia, United States.

Rapid progress in additive manufacturing methods has led to the creation of a new class of ultralight, stiff and strong 3D architected metamaterials comprised of a network of hierarchical, stretch-dominated, micro-scale unit cells. The performance of these micro-architected metamaterials, spanning across multiple length scales, will ultimately be limited by their tolerance to damage and defects, yet an investigation of their fracture toughness has remained elusive. Here, we provide the first experimental observations of different crack initiation modes activated by model-I loading of notched low density, hierarchical metamaterials. We find that, through hierarchical micro-architected features, the low density, stretch-dominated metamaterials can achieve simultaneously higher fracture toughness and strength: properties that are usually mutually exclusive. Numerical and scaling relationships are also reported that accurately capture the measured fracture toughness and strength values. These are then used to develop design maps for the optimal hierarchical architectures as functions of the density of the metamaterial and failure strain of the parent material.

9:00 AM PM01.04.04
Subtractive Post-Processing to Create Delicate Nano-Scale Structures Andrew Gross and Katia Bertoldi; Harvard University, Cambridge, Massachusetts, United States.

Two photon polymerization (TPP) has become a popular technique for testing new devices and designer materials at the nano and micro scales. This technique provides much of the flexibility of a desktop 3D printer with access to physics that are uniquely scale dependent. Like every additive manufacturing technique, TPP is limited to a minimum achievable size for the voxel that can be rastered to create a structure. The constraint on reducing the size of the voxel is a practical limit arising from both the spatial photon density distribution within the diffraction limited focal volume of the laser illumination and the contrast curve of the photosist that describes the degree to which it polymerizes in response to exposure at a certain dose. Here, we present an approach for achieving smaller feature sizes that is accessible to researchers without specialized equipment. This approach leverages the high resolution of a piezo stage to precisely raster the voxel into shapes that upon isotropic etching allow access to a smaller length scale. Free-standing fully three-dimensional structures with feature sizes under 100 nm are demonstrated. Additionally, this technique allows for the fabrication of structures which are otherwise too delicate to make. This is achieved both by simplyetching a structure of interest to impart more slender features, and also by including secondary support material to create complex structures that cannot be fabricated by any other method at this length scale.

9:15 AM PM01.04.05
Carbon-Based Nanolattice Materials Jens Bauer, Anna Guell, Cameron Crook and Lorenzo Valdevit; University of California, Irvine, Irvine, California, United States.

From a traditional perspective, there is little room for further expansion of the accessible material property space by classical material fabrication methods. Single one- and two-dimensional nanoscale objects, such as nanowires and thin films, are known to exhibit exceptional physical properties. Yet, their properties are intrinsically coupled to their small size and their solitary nature, and therefore can hardly be accessed in actual materials of practical volume. If nanowires and thin films are simply scaled up, many of their exceptional properties, which relate to surface to volume effects, drop dramatically; similarly, if nanoscale objects are clustered in a composite material, interfaces dominate the overall behavior, again substantially reducing performance. Nanolattice materials, regular three-dimensional networks constructed from nanowires or thin films, can potentially resolve this dilemma. Nanolattice materials can be thought of as metamaterials which can scale up beneficial size effects by topological design of their architecture. In our work, we have fabricated carbon-based nano-architected lattice materials by direct laser writing (DLW) and pyrolysis. The combination of DLW and pyrolysis facilitates complex three-dimensional carbon-based ceramic structures of uniquely high resolution, with feature dimensions below 100 nm. This enables to take advantage of pronounced size-dependent effects on material strength. We systematically investigate the effects of processing parameters, dimensional scale, topology and topology-dependent strain during pyrolysis on the mechanical and functional properties of nanolattices. Here, we offer an overview of manufacturing routes, with emphasis on the key processing parameters. The interplay of size-dependent effects with different topological and material design approaches is emphasized.

9:30 AM BREAK

10:00 AM PM01.04.06
Designing and Manufacturing Cellular Mechanical Metamaterials Carolin Köhner1, 2 and Maximilian Wormser2; 1Chair of Materials Science and Engineering for Metals, FAU Erlangen-Nuremberg, Fürth, Germany; 2Joint Institute of Advanced Materials and Processes (ZMP), FAU Erlangen-Nuremberg, Fürth, Germany.
The geometrical structure of a material can drastically change its mechanical behavior. Mechanical metamaterials are architected structures with extreme properties that surpass the capabilities of conventional bulk material. Periodic cellular structures lend themselves to be used as metamaterials because of their high variability. In the past, manufacturing methods limited the possibility to create intricate three-dimensional cellular structures. This changed with the advent of additive manufacturing technologies. Nowadays, cellular structures can be printed as polymers, ceramics or metals. Finding new shapes for cellular materials can be difficult. We developed a new method to find interesting unit cell designs by using eigenmode analysis on a basic unit cell. The eigenmode shapes of the basic cell then serve as new unit cell for further simulations. Cellular materials can have an excellent ratio of stiffness to weight, making them highly suitable for lightweight construction. By adding additional metamaterial functions to these structures, we get a so-called intelligent or smart material. An example for such a feature is auxetic behavior, i.e. a negative Poisson ratio. Therefore, if the structure is compressed, it will also compress laterally to the applied force and reduce its volume as opposed to regular materials that expand laterally. Other metamaterial effects influence the way structures interact with mechanical waves. Given the right geometry, a structure can be unable to resonate in a certain frequency band. This effect is called phononic band gap and it can be used for wave guiding and filtering. Selective Electron Beam Melting (SEBM) is a powder-based additive manufacturing technology that enables us to produce complicated structures on a millimeter scale with very few limitations. We used this technology to create auxetic structures as well as phononic band gap structures using the above mentioned eigenmode analysis method and tested their properties. A new measurement method for mechanical wave transmission in lattice materials was developed. Piezoelectric transducers were used to create and measure the incoming and outgoing signal. The experimental results were compared to numerical simulations and were found to be in agreement with each other. By further improving the design based on gradient based optimization the phononic band gap of a macro-level unit cell could be pushed into the audible range below 20 kHz.

10:30 AM PM01.04.07
Single Atom Scale Fabrication by Scanning Transmission Electron Microscopy Ondrej Dyck1, 2, Sergei V. Kalinin1, 2 and Stephen Jesse1, 2; 1Center for Nanophase Materials Science, Oak Ridge National Laboratory, Oak Ridge, Tennessee, United States; 2Institute for Functional Imaging of Materials, Oak Ridge National Laboratory, Oak Ridge, Tennessee, United States.

Fabrication of atomic scale structures remains the ultimate goal of nanotechnology. The reigning paradigms are scanning probe microscopy (SPM) and synthesis. SPM assembly dates to seminal experiments by Don Eigler, who demonstrated single atom manipulation. However, stability and throughput remain issues. The molecular machines approach harnesses the power synthetic chemistry to build individual functional blocks, yet strategies for structural assembly remain uncertain. In this presentation, I discuss research activity towards a third paradigm — the use of the atomically focused beam of a scanning transmission electron microscope (STEM) to control and direct matter on atomic scales. Traditionally, STEM’s are perceived only as imaging tools and beam induced modifications as undesirable beam damage. Our team and several groups worldwide have demonstrated that beam induced modifications can be more precise. We have demonstrated ordering of oxygen vacancies, single defect formation in 2D materials, and beam induced migration of single interstitials in diamond lattices. What is remarkable is that these effects often involve one atom or small group of atoms and can be monitored real-time with atomic resolution. This fulfills two out of three requirements for atomic fabrication. I will introduce several examples of beam-induced fabrication on the atomic level, and demonstrate how beam control, rapid image analytics, better insight through modelling, and image- and ptychography based feedback allows for controlling matter on atomic level. This research is supported by and performed at the Center for Nanophase Materials Sciences, sponsored at Oak Ridge National Laboratory by the Scientific User Facilities Division, BES DOE.

10:45 AM PM01.04.08
Facile Electrochemical Fabrication Route for Multicomponent and Multifunctional Inverse Opaline Films for Highly Sensitive VOC Detection Pei-Sung Hong1, Yu-Szu Chou1, Shih-Cheng Chou1, Guang-Ren Wang1, Chuan-Yuan Wang1, Yu-Ting Cheng1, Po-Chun Chen1, Tsung-Eong Hsieh1, Kea Tiong Tang2 and Pu Wei Wu1; 1National Chiao Tung University, Hsinchu, Taiwan; 2National Taipei University of Technology, Taipei City, Taiwan; 3National Tsing Hua University, Hsinchu City, Taiwan.

Non-invasive diagnosis for early detection of diseases have drawn considerable attention during past few decades. Earlier, several biomarkers have been identified in exhaled breath specimens using GC-MS technique. However, the GC-MS analysis is a tedious process requiring high-end instruments, spacious facility, and well-trained personnel. With rapid development of artificial intelligent e-nose system, there is a promising potential serving as a portable alternative to the aforementioned analytical technique. With ultra-high specific surface area and activities, numerous nanomaterials have been explored for sensing applications. However, a dilemma often exists as researchers trying to increase the amount of materials used for enhanced sensitivity and meanwhile maintaining accessible surface area. Unlike 2D thin film sensors, 3D nanostructured sensing materials provide a larger surface-to-volume ratio with enhanced mass transfer due to their open cell configuration. Several architectures have been proposed and designed for these purposes. Among them, inverse opal (IO) is considered as a promising candidate as it possesses ordered and monodispersed porous scaffolds with numerous interconnected channels for superior permeability. Herein, we report a facile fabrication route through electrochemical methods to construct inverse opaline sensing materials. Their pore sizes can be tailored from meso to macro scale, providing a large specific surface area and rendering a lower tortuosity pathway for gas stream. In addition to the structural design, we further introduce heterojunctions into the sensing units by using composite metal oxides as the molding materials for enhanced sensitivity. Lastly, a top-bottom electrode configuration is adopted with a metal IO upper layer as a gas permeable electrode. Structural, morphological, and compositional characterizations are conducted by SEM, EDX, XRD, and XPS techniques. The responses of different composite IO films toward VOC vapors are then recorded and analyzed.

11:00 AM PM01.04.09
Mechanics and Fabrication of Architected Graphene-Metal Lattice Materials Richard L. Li, Shruti Rastogi and Jeffrey W. Kysar; Mechanical Engineering, Columbia University, New York, New York, United States.

Much of graphene research to date has focused on graphene in a 2D, planar geometry, but the idea of extending a 2D material to span three dimensions optically and mechanically is an exciting possibility. A variety of techniques have been widely explored for assembling graphene into 3D configurations such as foams, aerogels, and lattice networks. However, the arrangement of individual graphene sheets is stochastic in most cases, and none have produced periodic graphene materials with precisely controlled and tunable 3D features at small scales. The creation of such a material would enable the systematic study of graphene in 3D space and could lead to many new insights.

We have taken a step towards this goal by developing a method for depositing graphene on micrometer-scale 3D periodic lattice structures. Polymer lattice templates are fabricated by two-photon lithography and then metallized by electroless plating of either copper or nickel-boron. After the polymer is
removed, the hollow metallic lattice is used as a substrate for graphene growth by chemical vapor deposition (CVD). The result is a 3D periodic lattice with graphene-metal composite struts. We can then test the mechanical behavior of these composite lattices under uniaxial compression and observe the reinforcing effect of the graphene layers.

Raman spectroscopy and scanning electron microscopy reveal conformal sheaths of single-layer to few-layer graphene enveloping the metallic lattices. Compression tests indicate that the graphene coatings significantly enhance the mechanical properties of the lattice. These preliminary results demonstrate a potential pathway for the research and application of 2D materials in 3D space.

11:15 AM PM01.04.10 Fabrication of Ideally Ordered Anodic Porous TiO2 and Its Application to Photonic Crystal Toshiki Kondo, Shota Hirano, Sanami Nagao, Toku Tamura, Takashi Yanagishita and Hideki Masuda; Tokyo Metropolitan University, Hachioji, Japan.

Ideally ordered nanohole array of TiO2 has attracted attention due to its wide applicability based on the periodicity of the nanoholes, high refractive index and semiconductor properties of TiO2. Various types of optical devices based on an ideally ordered nanohole array of TiO2 have been proposed, such as photonic crystal, photocatalyst, solar cells and so on. Until now, several kinds of fabrication process of nanohole array structure of TiO2 have been proposed. By applying semiconductor microfabrication process using electron beam lithography and focused ion beam etching apparatuses, an ideally ordered nanohole array could be obtained. However, this process is time consuming. By anodization process, porous TiO2 could be obtained in large sample area. However, it is difficult to obtain ideally ordered nanohole array structure because the nanoholes in anodic porous TiO2 usually form multi domain structure. In this presentation, fabrication process of anodic porous TiO2 having ideally ordered nanohole array and its application to photonic crystal will be presented [1~4]. Prior to the anodization of Ti, concaves were fabricated onto the surface of Ti through a texturing process. In subsequent anodization, each concave acts as a starting point of the formation of nanoholes, resulting in the formation of an anodic porous TiO2 having ideally ordered nanohole array. Photonic crystal properties of the anodic porous TiO2 were evaluated by measuring reflection spectra. Then, formation of photonic band gap at visible wavelength range was confirmed. The present process is expected to be applied to fabricate various types of functional optical devices requiring an ideally ordered nanohole array structures, such as photonic crystals.


11:30 AM PM01.04.11 Strength and Fracture Toughness of Hierarchical Nanomaterials with Porous TiO2 by Ion-Beam Milling and Chemical Etching Xinchen Ni1, Reed Kopp1, Nathan Fritz2, Estelle Kafion-Cohen1, Carolina Patutado1, Alberto Arteiro2, Gregor Borstnar3, Mark Mavrogordato3, Lukas Helfen4, Ian Sinclair1, Mark Spearing1, Pedro P. Camanho2 and Brian L. Wardle1; 1Massachusetts Institute of Technology, Cambridge, Massachusetts, United States; 2University of Southampton, Southampton, United Kingdom; 3European Synchrotron Radiation Facility, Grenoble, France; 4Karlsruhe Institute of Technology, Karlsruhe, Germany.

Aerospace-grade unidirectional carbon fiber reinforced plastic (CFRP) composite laminates were reinforced in the resin-rich weak interfaces using high densities of aligned nanoscale fibers, in particular, aligned carbon nanotubes (A-CNTs), in a hybrid architecture termed “nanostitching.” Here, we investigate the effect of A-CNT interlaminar reinforcement on laminate strength and toughness via ex situ and in situ mechanical testing, leveraging both synchrotron radiation computed tomography (SRCRT) and lab-based high resolution micro-computed tomography (µCT). Mode I and II interlaminar fracture toughness were assessed by conducting ex situ double cantilever beam (DCB) and end-notched flexure (ENF) tests, respectively. We find a ~6% lower steady-state mode I fracture toughness and no improvement of precracked mode II fracture toughness over baseline laminates. However, unique crack propagating behaviors in A-CNT reinforced laminates were revealed by scanning electron microscopy and µCT of fractured specimens. A-CNTs were found to force the crack into the intralaminar region in both DCB and ENF loadings, suggesting a tougher interlaminar region with the addition of A-CNTs. The measured toughness values of the A-CNT reinforced laminates were found to be associated with the toughness inside the ply. In situ SRCRT tensile testing of double edge-notched tension (DENT) specimens shows a ~9% increase in ultimate tensile strength (UTS) over the baseline specimens. No significant differences in progressive damage features (up to 90% of UTS) near the notch were observed regardless of the presence of A-CNTs. 3D visualization and damage segmentation software identified matrix cracking and fiber/matrix interfacial debonding as dominant damage mechanisms for loads up to 90%. However, large interlaminar delaminations, which are known to be a primary damage mechanism suppressed by A-CNT interlaminar reinforcement, were revealed by post-mortem CT of DENT specimens. These findings reveal for the first time the multiscale strengthening and toughening mechanisms induced by A-CNTs, which influence the macroscopic behavior of composite laminates. Future work will focus on acquiring 3D data beyond 90% UTS of DENT configurations and performing in-situ mode I testing to non-destructively elucidate the crack propagation behavior in real-time.

11:45 AM PM01.04.12 Self-Assembly of Polyhedral Metal-Organo Framework Nanoparticles Civan Avci1, Inhar Izaz2, Amna Carné2, Jose Aangel Pariente1, Javier Perez Carvajal1, Maria Isabel Alonso1, Álvaro Blanco1, Marjolein Dijkstra3, Daniel Maspoch2 and Cefe Lopez1; 1Consejo Superior de Investigaciones Científicas, Madrid, Spain; 2Catalan Institute of Nanoscience and Nanotechnology, Barcelona, Spain; 3University of Utrecht, Utrecht, Netherlands.

Self-assembly of nanoparticles into long-range, three-dimensional, ordered superstructures is crucial for the design of a variety of materials, including plasmonic sensing materials, energy or gas storage systems, catalysts and photonic crystals. This kind of structures has been demonstrated mostly with spheres that, owing to their entropy-favoured fcc packing, form the densest structures possible.[1] Polyhedra have been tested but so far only with metals and oxides.[2]

Here, we have combined experimental and simulation data to show that truncated rhombic dodecahedral particles of the metalorganic framework (MOF) ZIF-8 can self-assemble into millimetre-sized superstructures with an underlying three dimensional rhombohedral lattice with photonic crystal properties.[3] These superstructures feature a photonic bandgap that can be tuned by controlling the size of the ZIF-8 particles and is also responsive to the adsorption of guest substances in the micropores of the ZIF-8 particles. Superstructures with different lattices can also be assembled by tuning the truncation of ZIF-8 particles, or by using octahedral UiO-66 MOF particles instead. These well-ordered, submicrometre-sized superstructures might ultimately facilitate the design of three-dimensional photonic materials for applications in sensing.

References

Cellular materials with designed nanoarchitectures have redefined the limits of the accessible material-property space throughout different disciplines. Having characteristic features in the micro to nanometer regime, such architected materials exhibit exceptional mechanical properties at low density, including ultra high strength, damage tolerance, and stiffness. However, the design of lightweight structures that are both strong and damage tolerant is still a challenge and requires a better understanding of cyclic deformation and failure of the strength-optimized structures. Not only the absorbed energy during one loading cycle is important, but also the ability of the lattice to recover, maintain the strength, and be able to absorb energy during additional cycles. The highest resolution in manufacturing is currently achieved by 3D direct laser writing, which enables the fabrication of highly customized polymeric microlattices. We then apply additional processing steps, such as additional heat treatments and ceramic or metal coatings, to further improve or tailor the effective mechanical properties. While a ceramic coating in general results in enhanced strength, the failure mechanisms depend on the coating thickness. Brittle fracture of the struts rather than buckling is observed, when comparing the effect of 10 nm and 100 nm thick alumina coatings. However, in addition to high strength our solid-beam microlattices exhibit significant energy dissipation during cyclic loading experiments. Overall, the energy dissipation is a function of progressively failing ligaments, while upon multiple loading cycles to the same strain stable cyclic behavior is approached. In situ experiments showed that post-yield softening was induced by plastic buckling and crushing of individual ligaments. The contributions of the different deformation and failure mechanisms to the specific dissipated energy were quantified through progressive cyclic loading of the microlattices.

Additive manufacturing has opened new opportunities for engineering the mechanical properties of materials via geometry. While there have been significant efforts in the architected materials field to exploit geometry to realize materials with unique properties such as high specific stiffness and the ability to recover from large deformations, the engineering of fracture properties and crack growth resistance via geometry is less studied. Here, we use a higher flexural and inelastic buckling strength [1] and higher compressive strength due to greater resistance to plastic buckling when compared to the biomedical fields. Among periodic lattice structures hollow lattice structures are gaining popularity due to their improved mechanical properties, such as mechanical properties of the constituent materials. Analytical and finite element fracture mechanics models are used to establish quantitative relationships between toughness and geometry and to demonstrate how compliance introduced by structuring can reduce the strain energy release rate at crack tip, resulting in higher effective toughness. Toughness of architected interfaces made via laser patterning as well as additive manufacturing were measured using double cantilever beam experiments. Measurements on a relatively brittle acrylic (PMMA) and higher toughness ABS structured specimens were performed. The experimental results are in agreement with model predictions. A key and significant finding of this work is that less material can lead to higher toughness when the geometry of the interface is designed based on an understanding of the mechanics. The experiments, mechanics modeling, and an overall design strategy for using geometry to enhance interface toughness will be presented.

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Control of Interfacial Crack Behavior via Architected Cellular Materials in 3D Printed Structures Chengyang Mo and Jordan R. Raney; Mechanical Engineering and Applied Mechanics, University of Pennsylvania, Philadelphia, Pennsylvania, United States.

Natural materials, with their remarkable fracture toughness and low density, have long been a source of inspiration to engineers. In recent years, researchers have made great strides in cataloging the specific mechanisms by which these properties are attained. These mechanisms arise from the very complex microstructures that are observed in natural materials, including mechanisms that arise due to internal geometric parameters (e.g., structural hierarchy, interface density, interface angle, etc.) and due to material parameters (e.g., interfacial adhesion between two distinct material types). It is often difficult to isolate the respective contributions, particularly in an experimental context, where it is difficult to precisely and uniformly control the interfacial properties between two materials along an internal interface. As one example, the Conch Shell has three different levels of hierarchy to prevent shell fracturing under puncture loading allowing dissipation of a large amount of energy by crack diversion, with characteristic hard calcium carbonate sheets separated by soft protein layers where initial cracks form during loading. The goal of this research is to separate the effects of geometry from that of the material interface, and to thereby determine how toughness can be maximized purely through a structure’s internal geometry. To do this, we fabricate structures (from a single material) that contain internal lattices, which act as weak interfaces due to their decreased relative density. Rather than relying on a material interface, internal cellular structures act as crack arresting regions with variable effectiveness based on such properties as relative density and lattice shape. We experimentally characterize fracture behavior, measuring properties such as fracture toughness, energy dissipated, and crack path in 3D printed single edge notched bend samples (SENB). We study geometric parameters including defect region orientation, lattice density, and lattice shape for crack diversion to occur and maximize energy dissipation. The effect of lattice parameters to critical defect region orientation is studied analytically and compared with experiments. Numerical simulation is also implemented to better predict the critical angle taking account of differences in fracture toughness of cellular materials under different modes.

Selective Laser Melting of Ti6Al4V Lattice Structures with Hollow Struts—Processability, Mechanical Behaviour and In Vitro Response Ezgi Ezgi Osmay, Wencheng Liu, Bernard Chen, Jessica E. Frith, Xinhua Wu and Andrey Molutnikov; 1Department of Materials Science and Engineering, Monash University, Melbourne, Victoria, Australia; 2Department of Mechanical and Aerospace Engineering, Monash University, Melbourne, Victoria, Australia; 3Monash Centre for Additive Manufacturing, Monash University, Melbourne, Victoria, Australia.

Advances in additive manufacturing are enabling fabrication of multitude of complex cellular structures for numerous applications in aerospace and biomedical fields. Among periodic lattice structures hollow lattice structures are gaining popularity due to their improved mechanical properties, such as higher flexural and inelastic buckling strength [1] and higher compressive strength due to greater resistance to plastic buckling when compared to the
equivalent solid lattice structures [2]. Furthermore, additional channels provided by hollow struts provide ample space for tissue in-growth and cell colonization. The latter aspect is of a particular interest since our recent study demonstrated [3], that pore size and distribution significantly affect the cell migration in the lattice scaffolds.

In this work, we explore the potential use of hollow lattices for bone implant scaffolds. We have designed and fabricated Ti6Al4V hollow body centered cubic (BCC) lattice structures with different hollow thicknesses and unit cell pore sizes using selective laser melting process. We also included additional hollow x-struts at the nodes and centres of each BCC unit cell to study the effect of vertical channels. The mechanical properties and failure behaviour of the obtained scaffolds are investigated using compression testing. Furthermore, in-vitro studies with pre-osteoblast cells are conducted to study cell migration and colonization behaviour in channels and hollow struts. This study explores the use of novel lattice structure designs for orthopaedic implants and contributes to understanding of mechanical and biological response of hollow architectured materials.


2:45 PM PM01.05.05
Collective Magnetic Behavior and Domain Formation in Magnetite Nanoparticle Assemblies Nils Neugebauer1,2, Alexander Fabian1,2, Matthias T. Elm1,2, Christian Heiliger1,2 and Peter J. Klar1,2; 1Institute of Experimental Physics I, Justus-Liebig-Universität Gießen, Gießen, Germany; 2Center for Materials Research, Justus-Liebig-Universität Gießen, Gießen, Germany; 3Institute of Theoretical Physics, Justus-Liebig-Universität Gießen, Gießen, Germany; 4Institute of Physical Chemistry, Justus-Liebig-Universität Gießen, Gießen, Germany.

Ferromagnetic nanostructures, such as arrays of magnetic nanodots, are of high interest for applications on the field of high density storage media, non-volatile logic in spintronic devices, as the magnetic properties of the arrays can be tuned by their shape and their arrangement. An alternative approach to patterning magnetic thin films is the fabrication of arrangements of ferromagnetic nanoparticles. Such low dimensional assemblies show collective magnetic behavior due to dipolar coupling between the nanoparticles, which offers an additional degree of freedom in manipulating the magnetic interactions inside the magnetic elements. However, in order to tune the magnetic interactions of the assemblies a detailed understanding of the magnetic coupling phenomena is necessary.

Here we present the investigation of the magnetic properties of magnetite nanoparticle assemblies using angle-dependent ferromagnetic resonance measurements [1]. Nanoparticle assemblies of different shape were prepared using the meniscus force deposition method. For this, electron beam lithography was used to prepare PMMA openings. In a horizontal dip-coating process the nanoparticles with an average diameter of 20 nm are arranged in these openings by self-assembly. To investigate the influence of the shape on the magnetic interactions between the particles, rectangular assemblies with different aspect ratio varying from 1:1 to 1:1000 were prepared. The angle-dependence of the resonance was described using the Smit-Suhl formalism. For assemblies with aspect ratios below 1:10 the demagnetization factors obtained are in good agreement with the values expected from the aspect ratio revealing a single magnetic domain behavior due to dipolar coupling. For larger aspect ratios the ratio of the demagnetization factors is much smaller than the expectation indicating the formation of a magnetic domain structure inside the assemblies. This assumption is further supported by simulations of the magnetic structure and probably arises due to inhomogeneities in the filling of the openings. Furthermore, magnetic coupling between the arrangements was investigated by varying the distance between circular shaped arrangements. Due to their shape, isolated arrangements reveal an isotropic behavior of the angle-dependence of the resonance field in in-plane geometry. However, below a critical distance coupling between the arrangements occurs which is manifested by an angular dependence of the resonance field.


3:00 PM BREAK

3:30 PM *PM01.05.06
Nano to Macro Architectured Materials and Structures—Processing Challenges and Opportunities Haydn N. Wadley, University of Virginia, Charlottesville, Virginia, United States.

The development of light metals and ceramics with internal topologies that offer novel combinations of properties has been greatly accelerated by the recent development of additive methods for their fabrication. These enable the fabrication of complex shaped structures containing internal features with length scales that range from a few tens of nanometers to the centimeter scale. However, each of the processing methods suffer from various deficiencies such as a limited materials palate, difficult to control internal surface roughness, the presence of residual stresses and distortions of structure, and properties of the solid material that are sometimes inferior to those of conventionally processed counterparts. As a result, innovative applications of traditional fabrication/process methods can still compete with additive approaches in many areas. Here three approaches for making microarchitected materials are described and the challenges confronting their further development discussed. At the centimeter scale, we describe the use of electron beam melting approaches for making Ti-6Al-4V octet lattice structures and show how surface roughness of the trusses impact mechanical performance, especially as the strut diameter decreases towards 1 mm. This approach is then compared with a perforated sheet folding/braze bonding approach for making stainless steel octet lattices with sub-millimeter diameter trusses. In this case, surface roughness is not the challenge, instead the precision of the perforation and sheet folding processes together with the knock down in braze region properties limit the smallest cell size that can be achieved. To reach cell sizes in the micron to tens of nanometer range, we have begun to investigate the use of template electroplating and related micro- to particle space holding based methods. While this approach holds great promise for making nano-architected materials, the challenges of scaling the approach to meter scale structures still remains.

4:00 PM PM01.05.07
Additive Manufacturing of 3D Nano-Architected Metals Andrew Vyatskiikh1, Stephanie Delalande2, Akira Kado1, Xuan Zhang2, Carlos M. Portela1 and Julia R. Greer1; 1California Institute of Technology, Pasadena, California, United States; 2Scientific Department, PSA Group, Velizy-Villacoublay, France; 3Department of Engineering Mechanics, Tsinghua University, Beijing, China.

Most existing methods for additive manufacturing (AM) of metals are inherently limited to feature sizes of 20-50 μm, which renders them inappropriate for generating complex 3D metallic structures with smaller dimensions. We demonstrate a lithography-based process to create complex 3D nano-architected metals with ~100 nm resolution. We synthesize metal precursor solutions that contain Ni clusters and use them to produce a metal-rich photoresist. We use two-photon lithography to sculpt the computer-designed architectures out of the resist and pyrolyze them to volatilize the organic constituents, which results in a > 90wt% metal architecture. Using this approach, we demonstrate the fabrication of Ni octet nanolattices with a unit cell size of 2 μm, and beam diameters of ~300 nm. TEM analysis reveals that the microstructure of Ni beams in the lattice is nanocrystalline and nanoporous, with a 20 nm mean grain size and 10-30% porosity within each beam. In-situ nanocompression experiments show the specific strength of as-fabricated octet nanolattices to be 2.1-2.7 MPa g⁻¹ cm², which is comparable to that of metal lattices with 0.1-1.0 mm beam diameters fabricated using alternative metal AM technologies. These findings suggest an efficient pathway to create complex three-dimensional metallic structures with nano-scale resolution.
4:15 PM PM101.05.08
Alignment of Barium Titanate Platelets for Textured 3D Piezoelectric Architectures Using Direct Writing
Rebecca Walton, Richard Meyer and Gary L. Messing; The Pennsylvania State University, State College, Pennsylvania, United States.

Direct writing textured piezoelectric ceramics can allow for increased flexibility of 3D designs for a variety of applications through greater geometric possibilities, as well as novel orientations of crystallographic alignment. Generally, direct writing of ceramics and ceramic composites involves the extrusion and deposition of a ceramic containing paste which is then gelled to maintain its shape. Some studies on the alignment of platelets using a circular nozzle have explored the densification of textured ceramic as a function of position in the filament cross-section and length of the deposition nozzle. This work will highlight the fabrication of textured Pb(In0.53Nb0.47)O3-Pb(Mg1/3Nb2/3)O3-PbTiO3 ceramic designs with crystallographic orientations that are difficult to fabricate with traditional forming techniques. The effect of shear field shape and duration and ceramic paste rheology on the alignment of barium titanate templates and the fidelity of the printed geometries will be explored. Increasing the magnitude of the shear field in the nozzle during direct writing and controlling the rheology of the ceramic paste via pH control of the aqueous phase allows for the fabrication of dense, textured Pb(In0.53Nb0.47)O3-Pb(Mg1/3Nb2/3)O3-PbTiO3 ceramics with varied geometries and alignment directions.

4:30 PM PM101.05.09
Small Scale Materials with Tunable Impact Resistance Based on Electrospun Fibers via Integrated Additive Manufacturing Methodologies
Arief S. Budiman and Avinash Baji; Singapore University of Technology & Design, Singapore, Singapore; 1La Trobe University, La Trobe, New South Wales, Australia.

We used additive manufacturing methodologies to fabricate impact resistant composites. Similar to natural materials, such as mantis shrimp dactyl club, these lightweight synthetic analogues display superior structural properties and functionalities – such as fracture toughness and impact resistance. The present study is based on the hypothesis that synthetic impact resistant materials with extraordinary fracture toughness can be obtained and tuned by the 3D hierarchical micro-architectured molecular structures – often enabled with nanoscale sub-structures – and that such 3D structures and mechanical properties could be enabled by additive manufacturing approach, especially with an integrated electrospinning technique that combine multi-length scale fabrication capabilities. Further, more specifically, this study investigated the fracture toughness and impact resistance properties of the 3D helicoidal architecture inspired by the mantis shrimp dactyl club and elucidate the associated deformation mechanisms. Having layers of multiscale fibers with rotated alignments in each layer, the 3D helicoidal architecture could potentially lead to very efficient (high rate) as well as highly tunable damage dissipation mechanism. We further explore the effect of the 3D helicoidal micro-architectured structures and the possible size effects of the substructures on the fracture toughness and impact resistance properties of the resulting composite materials as well as the associated damage mechanisms. In this study, we fabricated structures by controlling the orientation of the polymer fibers within the composites. This is enabled by recent advances in additive manufacturing technologies. We plan to use electrospinning further as well as a novel integrated approach involving melt near-field electrospinning (NFES) technique and another deposition method called the fused deposition modeling (FDM). The 3D architecture would enable the composites to absorb mechanical energy before they fracture. Ultimately this high performance, impact-resistant composite technology will have other widespread technologically important applications, such as in aerospace, space, military/defense to automotive and biomedical/prosthetics. We also aim to enable design of such advanced composite materials through creation of new manufacturing technologies for micrometer and nanoscale materials and for their assembly and integration at larger scale.

4:45 PM PM101.05.10
Complete Space Filling Cellular Network of Graphene Bubbles for Ultralight, Strong and Superelastic Materials
Seon Ju Yeol1, Min Jun Oh2, Hyun Min Jun3, Minhwon Lee4, Jung Gun Bae1, Yeseul Kim2, Kyung Jin Park2, Seungwoo Lee5, Daeyeon Lee1, Byung Mook Weon7, Won Bo Lee3, S. Joon Kwon1 and Pil J. Yoo2; 1Korea Institute of Science and Technology, Seoul, Korea (the Republic of); 2Sungkyunkwan University, Suwon, Korea (the Republic of); 3Seoul National University, Seoul, Korea (the Republic of); 4University of Pennsylvania, Philadelphia, Pennsylvania, United States.

Advanced materials with low density and high strength will have transformative impacts in construction, aerospace and automobile industries. These materials can be realized with assembling well-designed modular building units into interconnected structures. This study uses a hierarchical design strategy to demonstrate a new class of carbon-based, ultralight, strong, and superelastic closed-cellular network structures. In contrast to conventional top-down approaches such as three-dimensional (3D) printing, the building units are prepared by a multi-scale design approach starting from the controlled synthesis of functionalized graphene oxide nanosheets at the molecular- and nano-scale, leading to the microfluidic fabrication of solid-shelled bubbles with shape diversity at the macro-scale. Then, bubbles are strategically assembled into meso-scale 3D structures. Subsequently, these structures are transformed into self-interconnected and structurally-reinforced closed-cellular network structures through post-treatment, leading to the generation of a 3D graphene lattices with rhombic dodecahedral honeycomb structure at the centimeter-scale. The 3D graphene superstructure simultaneously exhibits the Young’s modulus above 300 kPa while retaining a light density of 7.7 mg/cm3 and sustaining the elasticity against up to 87% of the compressive strain benefits from efficient stress dissipation through the complete space-filling closed-cellular network. The fabricated 3D graphene closed-cellular structure opens a new pathway for designing lightweight, strong, and superelastic materials.

8:00 AM *PM101.06.01
Exploring Vast Design Spaces—Computational Optimization Methods for Additive Manufactured Lattice Structures
Kristina Shea; ETH Zurich, Zurich, Switzerland.

Recent advances in Additive Manufacturing (AM) enable the fabrication of complex 3D lattice structures at many scales and with many materials. Through computational design optimization methods for AM lattice structures, it is possible to control the distribution of materials and structure within objects with a high degree of precision. This has the potential to dramatically improve structural performance and even enable new functionalities that can not be realized with conventional manufacturing. However, this is highly challenging for designers due to the vast design space of new possible shapes, materials and structures and often unknown AM fabrication constraints.

To address these challenges, a Design for Additive Manufacturing (DFAM) framework is developed that starts with AM material and process
characterization from which quantitative models are created. These models are then used within design optimization methods for discrete lattice structures considering design variables of material, member size, shape and topology. Two methods are focused on including a Generalized Optimality Criteria (OC) method for multi-material optimization and optimization for anisotropy considering displacement, local yield stress and Euler buckling constraints. A Sequential Linear Programming (SLP) method is also discussed for the efficient optimization of member size and shape of truss lattice structures considering local yield stress and Euler buckling constraints. While the methods are process and material independent, results are shown for the characterized materials of the multi-material polyjet printing process. The results highlight the necessity of including AM fabrication constraints and tested material properties in the optimization process and the tight coupling needed among material understanding, the AM process and optimization methods to achieve optimized results.

8:30 AM PM01.06.02
Three-Dimensional Multiscale Design Using Neural Net Surrogate Models of Lattice Material Response Bill Arrighi, Jun Kudo, Dan Tortorelli, Seth Watt and Dan White; Lawrence Livermore National Laboratory, Livermore, California, United States.

Topology optimization is a methodology for assigning material or void to each point in a fixed design domain in a way that extremizes some objective function, such as the stiffness of a structure under given loads, subject to various imposed constraints, such as an upper bound on the mass of the structure. This generative design approach has been used to design both macro-structures, such as bridges, as well as lattice micro-structures, comparable to the familiar octet truss architecture. In both settings, the structural response is evaluated with the continuum finite element method, and a design sensitivity analysis provides the first-order derivatives of the objective and constraint functions to a gradient-based optimization algorithm, which iteratively updates the design to improve its performance.

Even with today’s supercomputers, it is generally not feasible to design both the macrostructure and microstructure simultaneously on the same computational mesh; the necessary spatial resolution and domain size require too great a computational cost. Thus, multiscale topology optimization methods use some form of information mapping between the scales, enabling separate, smaller analyses at each scale. Homogenization is the most common mapping in the linear elastic regime we are interested in. Except in the case of certain specialized microstructures, e.g. ranked laminates, homogenization still requires solution of the elasticity PDE for several assumed test strains for each different microstructural design, and thus the overall design problem can still be quite costly, especially in three dimensions.

Our solution to reduce the overall computational cost of the multiscale topology optimization design problem is to generate neural net surrogate models of the homogenized microstructural response. Our particular interest is in open truss micro-architectures, which can be described in a very low-dimensional manner as a union of rods with given endpoint locations and cross-sectional diameters. Fixing the endpoint locations within a unit cell reduces the dimensionality further and guarantees a priori structural continuity from one unit cell to another. The off-line expense of creating the surrogate models is amortized over the solution of many design problems.

We generate a separate model for each of the 21 independent components of the homogenized elasticity tensor, as well as the volume fraction occupied by solid material (the balance is void space). These surrogate models are very fast to evaluate, enabling efficient design of the macrostructure while retaining accurate microstructural response, and additionally enable recovery of the full microstructure when the design is complete. We have generated surrogate models for a number of open truss micro-architectures, including the octet truss, ORC truss, and isotruss. We demonstrate our multiscale design capability via minimal-compliance designs on domains with millions of macro elements.

8:45 AM PM01.06.03
Closed-Cell Architected Materials at the Hashin-Shtrikman Upper Bound Cameron Crook1, Jens Bauer2 and Lorenzo Valdevit2; 1Chemical Engineering and Materials Science, University of California, Irvine, Irvine, California, United States; 2Mechanical and Aerospace Engineering, University of California, Irvine, Irvine, California, United States.

Over the past decade, many beam-based micro- and nanolattice materials with exceptional mechanical properties have been reported. However, no beam-based lattice capable of achieving the Hashin-Shtrikman (HS) upper bounds for isotropic elastic strength or stiffness has been shown. Honeycombs, though mechanically efficient, are generally used in curved structures which make them highly anisotropic. This is undesirable in large-scale materials and engineering design. Very recently, specific close-cell, shell-based designs were numerically predicted to achieve the HS bounds for stiffness, but no experimental verification has been produced to date.

Here, we experimentally demonstrate closed-cell cube-octet micro- and nano-shell-based architected materials achieving the Hashin-Shtrikman upper bounds for both isotropic elastic stiffness and strength. Cube-octet materials with relative densities of 20-60% were fabricated by two-photon polymerization Direct Laser Writing (DLW) using a Nanoscribe Photonic Professional GT. These structures were in-situ compression tested to failure. Furthermore, a second batch of structures was pyrolyzed, converting the polymeric material to glassy carbon. Approaching the “ultimate” HS strength upper bound, which corresponds to an optimal topology combined with a constituent material achieving the theoretical material strength, these glassy carbon architected materials are shown to outperform all known beam-based micro- and nanolattice materials in specific strength.

9:00 AM PM01.06.04
The Representative Cellular Element (RCE) Method – Model, Implementation and Validation with Additive Manufacturing Dhruv Bhate; Arizona State University, Mesa, Arizona, United States.

Material modeling approaches for cellular materials broadly fall into three different categories, depending on the level of discretization at which the property of interest is modeled. In order of the scale at which material behavior is represented, these models can operate at either the level of the material point (bulk property models), the connecting member (member models) or finally, at the level of the cell (homogenization models). Bulk property models are often not representative of the microstructure and surface effects at the scale of the members that constitute cellular structures in Additive Manufacturing. Member models are challenging to characterize due to the small sizes of the structures involved and the specimen geometries do not always accurately capture behavior of the cellular material. Homogenization models, while efficient to implement computationally, are shape and size dependent and are unlikely to represent irregular and stochastic cell shapes and distributions accurately.

In this work, being presented for the first time, we propose a Representative Cellular Element (RCE) method for modeling cellular materials. Our approach follows from the concept of the Representative Volume Element (RVE) developed for heterogeneous materials. The RCE method essentially involves the identification of a structural element that represents the cellular material for the property of interest. For the model to be both valid and accurate, we show that it must represent the cellular material in three critical ways: geometry, processing history and the physics of its deformation and failure. We assess the performance of the RCE model against prismatic cellular structures (honeycombs) in a wide range of conditions: different shapes (regular, graded and stochastic), Additive Manufacturing processes and materials (ABS with Fused Deposition Modeling, Inconel 718 with Laser Powder Bed Fusion, and Ti6Al4V with Electron Beam Melting) and strain rates (ranging from 10⁶ to 10⁹ s⁻¹). The model is also evaluated against a multi-material hexagonal...
honeycomb made with a nylon composite and Kevlar continuous fiber. In each case, we examine the model's predictability of the nonlinear stress-strain response in the elastic and plastic regimes. Three different design strategies for the RCE are compared under these conditions to identify the ones that yield the best results. Challenges in implementation and limitations of the RCE approach are also discussed.

9:15 AM PM01.06.05
Multi-scale Geometric Design Principles Applied to 3D Printed Architectured Materials
Seved Mohammad Sajadi1, Peter S. Owuor1, Cristiano F. Woehler2, Variles Rodrigues2, Robert Vajtai1, Jun Lou1, Douglas S. Galvao2, Chandra S. Tiwary1 and P. M. Ajayan3; 1Materials Science and NanoEngineering, Rice University, Houston, Texas, United States; 2Applied Physics Department, State University of Campinas, Campinas, Brazil.

The emergence of 3D printing has enabled scientists to innovate complex geometrical designs in materials which were unattainable using conventional synthesis methods. The topological material design is becoming a common occurrence aided by 3D printing. Here we use inverse methods (function-to-shapes to build innovative engineered materials with tunable properties. Hold great promise as high load bearing and impact-resistant materials due to a unique layered deformation mechanism that emerges in these architectures during loading. Easily scalable techniques such as 3D printing can be used for exploring mechanical behavior of various predicted complex geometrical shapes to build innovative engineered materials with tunable properties.

9:30 AM BREAK

10:00 AM PM01.06.06
When Trusses Become Continuous—Predicting and Optimizing the Mechanics of Truss-Based Metamaterials
Dennis M. Kochmann1,2, Raphael Glaesener1, Bastian Telgen1, Claire Lestringant1 and Greg Phlipot2; 1ETH Zurich, Zurich, Switzerland; 2Graduate Aerospace Laboratories, California Institute of Technology, Pasadena, California, United States.

Truss networks have attracted significant attention as metamaterials whose mechanical, optical, thermal or acoustic properties can be controlled by the truss architecture (including the network topology, truss geometry and base materials). Enabled by advances in additive manufacturing, large arrays of periodic, hierarchical or functionally graded truss unit cells have been assembled at the micro- to nanoscales, producing new ultra-lightweight cellular solids. With enhanced experimental capabilities comes the need for new theoretical and computational tools to describe and predict the performance of truss networks containing millions and more of individual struts and junctions, where classical approaches such as direct numerical calculations incur prohibitive computational expenses. We show that such truss networks can be described efficiently by advanced constitutive models and numerical tools that replace the discrete truss network by an equivalent continuum. Our formulation captures nonlinear and inelastic material behavior as well as finite deformations. We discuss the theoretical and numerical techniques (involving nonlocal homogenization based on both translational and rotational degrees of freedom), and we present representative examples of truss lattices undergoing not only homogenous deformation but also instabilities such as buckling and shear banding. Finally, we combine the methodology with topology optimization to predict optimal cellular networks with spatially varying truss architecture. Unlike classical two-scale optimization, this approach results in compatible non-uniform truss architectures that can readily be fabricated by methods of additive manufacturing and offer opportunities for lightweight metamaterials with optimized mechanical properties.

10:30 AM OPEN DISCUSSION

10:45 AM PM01.06.08
Fracture Toughness of Truss and Shell-Based Architectured Materials
Meng-Ting Hsieh and Lorenzo Valdevit; University of California, Irvine, Irvine, California, United States.

The fracture toughness of brittle 2D lattices, such as triangular and kagome lattices, has long been investigated using elastic K-field approaches. However, much less is known about the fracture toughness of 3D cellular materials, particularly those built from ductile components. Here, we numerically investigate the linear elastic and elasto-plastic fracture toughness of octet lattices and spinodal shell-based architectured materials. We address the issues of mesh dependence where stress singularity arises by incorporating a characteristic element size. To extract R-curves for cellular materials, local damage is modeled using the Johnson-Cook criterion and fracture toughness is obtained by the J-integral compliance with SENB (single-edge notched bend) specimens. We compare the performance of truss and shell-based architectured materials in terms of scaling laws and R-curve behavior, and extract design principles. Numerical results are verified by fracture toughness experiments.

11:00 AM PM01.06.09
On the Correlation Between Topology and Elastic Properties of Imperfect Truss-Lattice Materials
Panos Pantidis1, Andrew Gross2, Katia Bertoldi1 and Simos Gerasimidis2; 1Civil Engineering Department, University of Massachusetts, Amherst, Amherst, Massachusetts, United States; 2Harvard University, Boston, Massachusetts, United States.

The pursuit of new materials with properties superior to the current state of the art, has led many investigators to examine the behavior of materials with a truss-lattice microstructure which accommodates member sizes in the range of micro- and nanometers. Aided by the immense progress of 3D additive manufacturing techniques, such as self-assembly (bottom-up techniques), material scientists have been enabled to fabricate novel materials with complex architectures which can attain unique, unprecedented and tunable properties. However, defects of various forms and concentrations are unavoidable in any fabrication process, and it is anticipated that the application of self-assembly techniques to larger three-dimensional volumes will increase the concentration of defects. Of particular interest for the self-assembly of truss-lattice materials is the influence of struts that are missing from the network, in various defect forms such as missing blocks (clusters) or randomly missing members. In this study, the dependence of the elastic properties on the concentration and distribution of missing struts is investigated for several three-dimensional lattice-truss materials of varying coordination number. This work constitutes a systematic experimental and numerical approach to examine and identify the mechanical elastic regime of defected architectured metamaterials. The experimental part of this project is conducted with a two-photon lithography approach, an advanced additive manufacturing technique capable of printing struts with sub-micron cross-sectional dimensions, while the numerical part utilizes finite element simulations accounting for the randomness of the damage spatial distribution through exhaustive Monte Carlo simulations. Focusing on a variety of elastic mechanical properties (Young’s, bulk and shear modulus), their evolution is monitored as the total defect percentage increases in magnitude, providing a comprehensive picture of the defected architectured metamaterials elastic property space. Finally, this work thoroughly explores the connection between defected truss-lattices and well-established homogenization techniques for composite mediums, elaborating on the applicability of the latter methods to accurately describe the response of defected lattice-based materials.
Spiders are abundant in most ecosystems in nature, making up more than 47,000 species. This ecological success is due to the web architectures and the exceptional mechanical properties of spider silk. Silk’s combination of strength, elasticity, toughness, and robustness originates from its hierarchical structure and has been a template for high-performance material design. In particular, spiders have optimized and adapted their web architecture to survive in their environment.

The most studied and familiar spider web is the 2D orb web which is composed of radial and spiral threads. However, 3D webs, such as sheet, funnel, or cob webs, are more common in nature. In contrast to 2D webs, where the spider is vulnerable to attacks, 3D webs surround the spider and offer a defensive advantage by warning the spider of intruders, blocking its predators and entangling prey.

Here, we investigate the architecture and mechanical properties of a Cyrtophora citricola 3D web. For the first time, we build a model of the 3D spider web generated through automatic laser scanning. The web is scanned by taking a high resolution picture of slices of the web illuminated by a sliding sheet laser. Using image processing algorithms, we construct a 3D fiber network. We study the response of a realistic web structure to mechanical loads using a coarse-grained bead-spring models based on the network model created through scanning and image processing.

Using this new method to trace the fiber network, we can study the connection between material and performance of numerous 3D spider webs. Understanding the roles of structure and material in the functionality and evolutionary fitness of spider webs could lead to innovative 3D spider web-inspired structures such as high performance light-weight long-span structures or fiber reinforced composite materials.

Harnessing Design Principles from Glass Sponges for Structurally Robust Lattices

Glass sponges are predominately deep sea sponges that live in ocean depths of 100-2000m. Beyond their fracture propagation inhibiting material composition, these sponges are perceived to exhibit large structural rigidity and strength against buckling. Since these sponges are primarily made of 'brittle silica', buckling strength may be a crucial property in making them resistant to impact and environmentally applied stresses. Structurally, they exhibit a base square-grid architecture and regular ordering of vertical and horizontal struts that form the skeletal system. Furthermore, their base structure is overlaid with double diagonal reinforcement struts, which create a checkerboard-like pattern of open-closed cell structure. This diagonal reinforcement design is conjectured to give the sponge greater buckling resistance and strength to localized damage then it would experience having a single diagonal reinforcement strut (while allocating the same amount of mass to the diagonal reinforcement.) Analogous to the sponge, many engineering structures, such as buildings and bridges, exhibit diagonal reinforcement struts as a stability mechanism. Based on this similarity, we explore the following research question: Can we generate design principles for diagonal reinforcements of square beam lattices that are optimally designed to avoid global structural buckling? Here, we present a numerical analysis of the structure deformation under various loading conditions as well as survey different arrangements within similar design space of the sponge. Furthermore, we present experimental evidence that supports our numerical analysis. Through the various design iterations we look for the critical buckling strain and the elastic load carrying capacity. Finally, we compare the results from the sponge design to what is typically used in engineering of structures such as buildings and bridges.
dimensional (2D) band gaps. 1D topological band gaps exhibiting longitudinal and bending interface modes were achieved using a beam composed of a 1D periodic arrangement of elastic unit cells [3].

In this paper, we present an application of topological mechanical band gaps that specifically takes advantage of chirality. We design a 1D chain of two different alternating 3D elastic chiral unit cells [4]. The individual unit cells have been inspired by our previous work [5]. The chain’s topological band gap, a result of the alternation of unit cells combined with their chirality, guarantees a protected edge state at one end of the beam, corresponding to a resonant localized twist mode. This mode can be excited by an axial motion at the other end of the beam, via evanescent modes in the gap. The topological robustness of the edge state allows us to add a micro-mirror, turning the arrangement into a resonant laser beam scanner with scalable operation frequency and adjustable quality factor.

Our work starts from calculations based on a simplified 1D mass-spring model, which we solve analytically. The system resembles two Su-Schrieffer-Heeger (SSH) models [6], one for the longitudinal displacement and one for the twist, which are chirally coupled via additional springs. We justify this model by the fact that the longitudinal (or pressure) and the twist mode on the one hand, and the two transverse (or shear) modes on the other hand, live in orthogonal subspaces according to micropolar continuum theory for chiral media. Within this model, the topological protection is due to a combination of time-reversal and a mirror symmetry, generalizing the classification of mechanical metamaterials [2]. The results of the model are verified by numerical finite-element calculations for the complete 3D microstructures. These microstructures should be amenable to laser nanoprinting.

Topology describes the properties of space under continuous deformation in mathematics. The concept has been used to explain band structures in condensed matter physics, resulting in the theoretical predications and experimental observation of topological insulator in electronic system, and recently also in photonic and phononic systems. Topological elastic metamaterials offer insight into classic motion law and open up opportunities in quantum and classic information processing. Theoretical modeling and numerical simulation of elastic topological states have been reported, whereas the experimental observation remains relatively unexplored. Here we present an experimental observation and numerical simulation of tunable topological states in soft elastic meta-materials. The on-demand reversible switch in topological phase has been achieved by changing filling ratio, tension, and/or compression of the elastic meta-materials. By combining two elastic meta-materials with distinct topological invariants, we further demonstrate the formation and dynamic tunability of topological interface states by mechanical deformation, and the manipulation of elastic wave propagation. Moreover, we provide a topological phase diagram of elastic metamaterials under deformation. Our approach to dynamically control interface states in soft materials paves the way to various phononic systems involving thermal management and soft robotics requiring better use of energy.

Non-reciprocal acoustic and elastic wave propagation is of significant interest due to its potential to enable direction-dependent devices that augment mechanical wave sensing and transmitting capabilities. They also open up the possibility for the construction of novel materials or structures for isolation from vibration and impacts. However, non-reciprocity can only occur under very specific circumstances, many of which are very difficult to achieve in practice. This work considers mechanical modulation as a potential means to obtain non-reciprocal elastic wave propagation in architectured materials. The specific case studied is the application of a slowly-varying, large amplitude, mechanical pump wave whose motion is orthogonal to the direction of wave propagation. This pump wave acts as a spatio-temporal modulation of the mechanical structure, resulting in time and space varying effective material properties and non-reciprocal elastic wave phenomena. The mechanical system of interest is modeled using both discrete and continuous mechanical lattices. A detailed analysis that quantifies the degree of non-reciprocity for excitation with finite bandwidth, as well as robustness to geometric variability associated with known additive manufacturing techniques, is provided and demonstrated via numerical examples.

Acoustic meta-surfaces that can manipulate and control sound waves at two-dimensional subwavelength scales open new avenues to unusual applications, such as sound barrier, super-resolution imaging, and particle manipulation. However, the long-standing goals of pushing frontier metamaterials research into real practice are still severely constrained by cumbersome configuration and rigid structure of the existing metamaterials. Here we fabricate an ultra-thin meta-surface (10-300 μm in thickness, up to ~1/650, λ the wavelength) that is capable of imparting sound wave with a non-trivial phase shift with high transmittance (>80%) in the range of 5 kHz and 30 kHz by electrospinning method. Besides, we incorporate the traditional paper-cutting art to carve the ultrasound meta-surface into hollow-out patterns, resulting in a variety of remarkable functions, including acoustic vortex, focusing, and super-resolution. Our hollow-out patterning approach innovates the traditional one-step metadevice fabrication process into two separated steps: (1) fabrication of ultrathin meta-surfaces; (2) hollow-out patterning of meta-surfaces. Our strategy opens an avenue to mass production of acoustic metadevices, shedding light on the applications of the meta-surfaces in architectural acoustics.

Moreover, benefited from the flexibility of our hollow-out patterning meta-surfaces, we propose the unprecedented non-planar acoustic meta-surface. Actually, most objects and occasions refer to non-planar surface in nature. Non-planar meta-surface is sorely in need to match these application scenarios but there has been hardly thought or design about non-planar acoustic meta-surface so far. We demonstrate a high-efficiency focusing on nearby arbitrary non-planar surface and design an “umbrella structure” to implement focusing with dynamic tunable focal length.

Elastic Properties of 3D-Printed Micro-Trusses Measured by Laser Ultrasonics

Assessing nondestructively the elastic properties of micro-scale architected materials is of key importance in several applications. Here we propose a method based on the laser generation and detection of ultrasounds to extract the vibrational mode frequencies of 3D-printed micro-truss samples. Experimental results show that the first bending and compressional modes of the truss systems can be monitored in the MHz frequency range. The corresponding frequencies are then compared to finite element simulations results, which allow to assess the effective elastic parameters, Young's modulus and shear modulus, of the micro-scale trusses. Capabilities and limitations of this laser ultrasonic characterization method will be discussed in the context of micro-scale architected materials, and some prospects in wave control problems at the micro-scale will be proposed.

Additively Manufacturable Micro-Mechanical Logic Gates

Early examples of computers were almost exclusively based on mechanical devices. Although electronic computers became dominant in the past 60 years, recent advancements in 3D micro-additive manufacturing technology provide new fabrication techniques for complex microstructures which have rekindled research interest in mechanical computations\(^1\). The authors propose a new digital mechanical computation approach based on additively-manufacturable micro-mechanical logic gates. The proposed mechanical logic gates (i.e., NOT, AND, OR, NAND, and NOR gates) utilize multi-stable micro-flexures that buckle to perform digital computations based purely on mechanical forces and displacements with no electronic components. Unprecedented properties have been achieved simultaneously including functional completeness, continuous operation, scalability, and near-zero energy consumption. A key benefit of the proposed approach is that such systems can be additively fabricated as embedded parts of microarchitected metamaterials\(^2\) that are capable of interacting mechanically with their surrounding environment while processing and storing digital data internally without requiring electric power.

Additive Manufacturing of Lightweight Mirrors

Additive manufacturing offers new routes to lightweight optics inaccessible by conventional methods by providing a broader range of reconciled functionality, form factor, and cost. Predictive lattice design combined with the ability to 3D print complex structures allows for the creation of low-density metamaterials with high global and local stiffness and tunable response to static and dynamic loading. This capacity provides a path to fabrication of lightweight optical supports with tuned geometries and mechanical properties. Our approach involves the simulation and optimization of lightweight stretch-dominated lattices for anticipated stresses due to polishing and mounting loads via adaptive mesh refinement. The designed lattices are 3D-printed using large area projection microstereolithography (LAPuSL), coated with a metallic plating to improve mechanical properties, and bonded to a thin (1.25 mm) fused silica substrate. We demonstrate that this lightweight assembly can be polished to a desired flatness using full aperture polishing under a load, and subsequently treated with a reflective coating. 

Additive Manufacturing

We present an approach to fabricating glass with a gradient refractive index profile by combining additive manufacturing and conventional ceramic processing techniques. Silica and silica-titania nanoparticles are synthesized and formulated into a slurry, which is tuned rheologically to enable extrusion through a nozzle via direct ink writing. During printing, two feedstocks of different compositions are fed at variable flow rates and mixed inline, allowing for controlled compositional profiles in the printed part. After printing, an optimized heat treatment is performed to ensure the removal of organic material and the formation of fully dense, transparent glass. We demonstrate 3D-printed gradient index lenses, and we show that our 3D-printed glasses have comparable properties to commercial glasses. This approach is promising for the development of optics with new functionality that cannot be accessed.
This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344 within the LDRD program 16-SI-003. LLNL-ABS-937900.

8:30 AM PM01.08.02
Design and Electrohydrodynamic Jet Printing of Layered Periodic Photopolymers Brian Lezzi1, Zahra Afkhami2, David Hoelzle2, Kira Barton2 and Max Stien1; 1Materials Science and Engineering, University of Michigan–Ann Arbor, Ann Arbor, Michigan, United States; 2Mechanical Engineering, University of Michigan–Ann Arbor, Ann Arbor, Michigan, United States; 3Mechanical and Aerospace Engineering, The Ohio State University, Columbus, Ohio, United States.

Periodic layered mirrors and filters are used for wavelength selection in spectroscopy and imaging. These mirrors have long relied on inorganic materials that have the refractive index contrast necessary to obtain desired electromagnetic wave interference patterns but require cumbersome and expensive manufacturing processes. Photopolymers have been formulated with refractive indices ranging from 1.3 to 1.7, and are, in principle, compatible with versatile, low-cost additive manufacturing processes. We use commercially available high (n=1.70) and low (n=1.38) refractive index photopolymers to create periodic layered mirrors and filters. We design mirrors having fewer than 15 alternating index layers with reflectance exceeding 60% in the visible range and complementary Fabry-Perot filters using a polymer resonant cavity with narrow transmission peaks of 15 nm FWHM with fewer than 30 layers. Experimental validation of these constructs is done using electrohydrodynamic jet (e-jet) printing; achieving individual layer thicknesses between 50-150 nm and well-defined filter “pixels” below 100 square micrometers. The reflectance/transmission characteristics of the printed mirror/filter “pixels” are measured using custom microspectrophotometry. Initial transmission results of five-layer samples are shown to be within 5-30% of prediction in the 500 to 700 nm wavelength range. Various printing parameters, such as utilizing continuous versus pulse printing and their effect on layer interfaces are discussed, along with how this information is used in the multi-physics design of monolithically-integrated organic imaging devices with predictable external quantum efficiency characteristics.

The authors would also like to acknowledge Aaditya Hambarde, Parag Deotare, and Steven Morris for their contributions to the work.

8:45 AM PM01.08.03
Fabricating Nanostructured Ceramics via Block-Copolymer Soft Templates Lisa M. Rueschhoff1, Luke Baldwin2, Robert Wheeler1, 2, Hilmar Koerner1, Matthew Dalton1, John D. Berrigan1, Michael Cinibulk1 and Matthew Dickerson1; 1Air Force Research Laboratory, Wright Patterson AFB, Ohio, United States; 2UES, Inc., Dayton, Ohio, United States.

Ceramics and ceramic composites architected at the nanoscale can exhibit extraordinary mechanical properties, including elastic deformation and high toughness, but are difficult to fabricate using scalable production methods. The ability to control bottom-up templates for preceramic polymers (PCPs) allows for the templating of the PCPs as a result of the self-assembly of the BCPs. Subsequent pyrolysis of the material converts the PCP into a structural ceramic material while removing the self-assembled BCP fugitive template. Using this method we have created novel silicon carbide based ceramic structures with nanoscale features and porosity. Insight into the relationship between polymer chemistry and structure in thin films of a novel and relatively inexpensive BCP/PCP system, as well as in-situ mechanical characterization of porous ceramic micropillars, will be presented.

9:00 AM PM01.08.05
Improved Strength and Functionality in Polymer Lattice Additive Manufacturing Joshua DeOtte1, 2, James Oakdale1 and Eric B. Duoss1; 1Lawrence Livermore National Lab, Livermore, California, United States; 2University of California, Davis, California, United States.

Polymer lattices produced with stereolithography additive manufacturing often have mechanical performance lower than expected from bulk properties. During fabrication, oxygen inhibits polymerization at pattern edges, which is oftentimes exploited to reduce adhesion to the print window. For small structures, this region of reduced polymerization could include entire features, dramatically reducing stiffness. Typically, a UV post-cure step is used to finish polymerization at the surface, but improvement in stiffness is minimal. Raman spectroscopy was used to measure the conversion ratio along the radius of a 500um diameter, additively manufactured cylinder, which showed that conversion ratio begins decreasing 100um from the surface. UV post-cure was insufficient to complete polymerization over the full region, so simultaneous thermal analysis was used to probe the temperature required to finish polymerization as well as the temperature for thermal degradation. After thermal post-processing, the average conversion ratio increased, improving stiffness in an additively manufactured lattice by an order of magnitude.

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LLNL-ABS-755077

9:15 AM PM01.08.06
3D Printed Ultrastrong and Ultratough Metallic Architectures Wen Chen1, Cheng Zhu2, Thomas Voisin2, S. K. McCall2, Andrew Pascall2, Joshua Kuntz2, Eric B. Duoss3 and Christopher M. Spadaccini1; 1Mechanical and Industrial Engineering, University of Massachusetts Amherst, Amherst, Massachusetts, United States; 2Lawrence Livermore National Laboratory, Livermore, California, United States; 3Mechanical and Aerospace Engineering, The Ohio State University, Columbus, Ohio, United States.

Ultrafine grain metals are a class of advanced structural metals with superior mechanical properties due to the inherent refined microstructure. Processing of ultrafine grained metals is however very challenging and often lacks versatility and scalability. Here, we introduce a novel approach that allows fabricating a variety of ultrafine grained metal 3D architectures by combining direct ink writing based additive manufacturing with nanocrystallization of a metallic glass precursor. The developed 3D ultrafine grained metal architectures demonstrate unprecedented mechanical performance such as high strength and energy absorption compared with other metallic architectures. More generally, our finding offers a new routine to achieve high performance complex metallic architectures with high precision, scalability, and versatility.

Prepared by LLNL under Contract DE-AC52-07NA27344.
**9:30 AM PM01.08.07**

Print Path Optimization and Computation of Machine Motions for Direct Ink Writing on Non-Planar Substrates [Eric B. Duoss, Adam Jaycox, Christopher M. Spadaccini, Seth Watts, Todd Weisgraber and Thomas Wilson; Lawrence Livermore National Laboratory, Livermore, California, United States.]

Recent work in direct ink writing (DIW) of elastomers on planar substrates has shown that different lattice morphologies display interesting characteristics, including tunable compressive and shear response. A natural question is whether we can achieve the same characteristics when printing on non-planar substrates. This presents two challenges: first, the curvature of such substrates induces distortion on planar designs, preventing a direct translation of these lattices; and second, determining the machine motions necessary to print lattices on non-planar substrates via DIW is non-trivial. We discuss our approach, which uses optimization techniques to address both challenges. Specifically, we pose the mapping of lattices to non-planar substrates as a minimization of areal distortion subject to constraints on angular distortion, and we determine the machine motions by solving an inverse problem using a forward kinematics model of a six-axis DIW machine to determine the translations and rotations necessary to print the lattices of interest.

**9:45 AM BREAK**

**10:15 AM PM01.08.08**

Designing Elasticomeric 3D Printed Architectures with a Mechanical Reduced Order Model [Todd Weisgraber, Ward Small, Jeremy Lenhardt, Christopher M. Spadaccini, Robert Maxwell, Eric B. Duoss and Thomas Wilson; Lawrence Livermore National Laboratory, Livermore, California, United States.]

Direct ink writing of silicone elastomers enables printing with precise control of porosity and mechanical properties of ordered cellular solids, suitable for shock absorption and stress mitigation applications. With the ability to manipulate structure and feedstock stiffness, the design space becomes challenging to explore to obtain an architecture with a desired mechanical response. Here we derive an analytical design approach for structure and feedstock. Results from finite element simulations and quasi-static mechanical tests of two different parallel strand architectures were analyzed to understand the structure-property relationships under uniaxial compression. Combining effective stiffness-density scaling with least squares optimization of the stress responses yielded general response curves parameterized by resin modulus and strand spacing. An analytical expression of these curves serves as a reduced order model, which, when optimized, provides a rapid design capability for filament-based 3D printed structures. To demonstrate the capability, we present computed optimal architecture designs that satisfy prescribed loading conditions and porosity constraints along with validating mechanical characterization data.

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**10:30 AM PM01.08.09**

Redox-Based EHD-Printing—Chemically Architectured Metals with Submicron Chemical Resolution [Alain S. Reiser, Marcus Lindén and Ralph Spolenak; ETH Zurich, Zurich, Switzerland.]

The concept of architectured materials, i.e., the design of properties through architecture, is of widespread interest, e.g., for the fabrication of optical and mechanical metamaterials, advanced microelectromechanical systems or biological scaffolds. Two-photon lithography (TPL) of polymeric materials is currently the major enabling technology for additively manufactured (AM) small-scale-architected materials, i.e., architectures with a smallest feature size <1 µm. TPLs combination of submicron feature size and high fabrication speed is unmatched by any competing AM technology, and post-print coating- or pyrolysis-techniques extend the accessible materials range towards metals and ceramics. Nonetheless, multi-material capabilities of TPL are currently very limited, especially for inorganic materials, and property design mostly relies on design through cellular geometries—an approach that invariably renders porous materials, with absolute properties that are typically inferior to dense designs.

Microscale multi-material AM has great potential to advance the synthesis of complex 3D architectural architectures with properties or functionalities that go beyond those of cellular designs. Yet, while multi-polymer approaches are relatively advanced, similar techniques for inorganic materials face serious limitations: because almost all methods are variations of ink-based, multi-nozzle strategies, the resulting as-deposited materials-quality is typically low, the chemical voxel-size, i.e., smallest feature of a fixed chemical composition, is large, and alignment can be demanding. Here, we introduce redox-based EHD-printing. This ink-free, electrochemical technique can overcome the before-mentioned limitations of multi-material microscale AM for the case of metals: it offers multi-metal printing from a single print-head with a chemical voxel-size of 250 nm, switching of the printed chemistry at a frequency of 10 Hz, and smallest geometrical feature size <100 nm. We show that the underlying electrochemical principle enables as-deposited materials quality that compares well with that of PVD-deposited metals, e.g., a flow-strength of 1 – 1.5 GPa for Cu, or a resistivity of 8 × 10⁻⁸ Cu. At the same time, redox-based EHD-printing offers a deposition speed of several µm s⁻¹, which is more than an order of magnitude faster than that of other electrochemical microscale AM methods. We demonstrate the synthesis of bi-metallic Cu-Ag 3D geometries with complex, embedded chemical architectures and discuss designs that tune local reactivity or mechanical properties through deterministic design of local chemistry.

**10:45 AM PM01.08.10**

3D Direct Laser Writing of Efficient X-Ray Lens Nano-Architectures [Kahraman Keskinbora, Umut T. Sanli, Hakan Ceylan, Iuliia Bykova, Markus Weigand, Metin Sitti and Gisela Schütz; Max Planck Institute for Intelligent Systems, Stuttgart, Germany.]

Focusing optics used in X-ray microscopy often have very demanding geometries from a fabrication point of view due to extremely small features, sub-mm scale apertures, extremely tight tolerances and high aspect ratios [1]. Such X-ray optics range from Fresnel zone plates (FZP) to three-dimensional kinoforms and several types of stacked lenses [2]. Generally, top-down subtractive methods are used for fabricating diffractive X-ray optics such as binary FZPs. Fully 3D optics, on the other hand, can be fabricated only in approximated forms by resorting to subtractive methods [2] including optical lithography, direct-write ion beam lithography or e-beam lithography followed by etching and electroplating. While these methods produce binary X-ray FZPs with the highest resolution today, they fail when high aspect ratios are required for focusing harder X-rays or optics with overhang structures such as tilted, wedged or curved lenses are desired towards high-efficiency focusing at high resolutions. Two-photon polymerization (2PP) emerged as a versatile route for manufacturing three-dimensional complex geometrical structures on the nano-scale. 2PP is an expanding field of research with a long list of technological applications in micro-robotics [3], photonics [4,5], metamaterials [6,7], and MEMS/NEMS [8] to name a few.

Here, we employed direct-write 3D femtosecond laser lithography to fabricating a highly efficient type of X-ray optic called kinoform lens, with parabolic surface profiles [9]. Then, we thoroughly characterized the focusing/imaging performances of the lenses by mounting them in a scanning transmission X-ray microscope (STXM) located in the synchrotron radiation facility BESSY II. The demonstrated an efficiency of up to 20 % efficiency, a remarkable value for soft X-ray energies. Using the fabricated lenses, we resolved 280 nm and 30 nm features via direct scanning and ptychographic imaging.
Contact: Architecture Through Active Mixing Processing

A. M. Golobic,1,2, Jason Ortega1, Jeremy Lenhardt1, Eric B. Duoss1 and Thomas Wilson1
1Lawrence Livermore National Laboratory, Livermore, California, United States; 2University of California, Davis, California, United States.

The mixing process of multimaterial systems can impart microstructure that can affect the overall material performance. This is especially true when mixing materials with vastly different densities, rheological behavior, and mechanical properties. The use of an active mixing printhead in 3D printing processes requires an understanding of how the processing dictates the microstructure, which in turn influences the material properties. The architectures formed during mixing were investigated with an active mixing printhead and siloxane inks with different rheology and mechanical behavior. The mixing was varied by controlling the rotating impeller speed and the resulting structure was studied with spectroscopic methods. The mechanical behavior was tested and correlated to microstructure. The experimental findings were compared with simulations of the mixing process and mechanical response.

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11:30 AM PM01.08.13
Additively Manufactured Metallic Cellular Materials—Energy Absorbing Characteristics
Marlini Simoes and Graham McShane; Engineering, University of Cambridge, Cambridge, United Kingdom.

Metallic cellular materials such as lattices and honeycombs are of interest for blast and impact protection, due to their ability to dissipate energy efficiently during dynamic crushing.

High-performance energy absorbing materials find applications in a wide range of industry sectors: e.g. aerospace, rail, automotive crashworthiness and structural blast protection.

Additive manufacturing (AM) processes enable new cell wall material-geometry combinations for the design of high performance cellular materials for impact energy absorption. This is of interest, for example, in the space industry, where high performance but light-weight materials that can protect structures from impact are sought.

To date, optimisation of AM cellular materials for high-strain rate loading scenarios remains relatively under-developed. Design of AM materials requires an understanding of the relationship between the AM process parameters and the properties of the resulting material across different scales: from the microstructure, to the geometric feature rendition, to the overall dynamic performance. Challenges include: (i) understanding process-property interactions for high strain rate behaviour; (ii) developing methodologies to navigate the wide design space enabled by AM processes, and hence optimise high strain rate properties; and (iii) modelling challenges spanning a range of length scales. These gaps in understanding hinder the exploitation of AM to deliver solutions for the most demanding impact protection problems.

References
First, focusing on the selective laser melting of a well-established alloy (316L), the influence of the laser heating strategy was investigated, specifically, the influence on defects, properties, microstructure and energy absorption. The laser power and the exposure time were varied, while maintaining a fixed total heat input. Also considered was the effect of five cellular material geometries: square honeycomb (SHC), lattice-wall square honeycomb, octet lattices (two variants), and origami.

It was found that a 125W laser power produces material with fewer porosity defects, which resulted in a higher yield strength and higher ductility. For the five geometries tested, the 125W power gives the cellular material with the greatest strength and energy absorbing capacity. However, the relative performance of the different geometries is insensitive to the laser power - SHC performs best in all cases. If we increase the strain rate, the same performance ranking holds between cellular geometries and laser powers. In all cases, a dynamic elevation in strength is observed.

11:45 AM PM01.08.14
Laser Power Optimization Using Feed Forward Control on the Overhang Geometry for 316L Stainless Steel  
Ava Ashby, Gabe Guss, Rishi Ganeiriwala, Neil Hodge, Wayne King, Manyalibo Matthews and Clara L. Druzgalski; Lawrence Livermore National Laboratory, Livermore, California, United States.

Arbitrarily complex architectures in metal additive manufacturing can be improved by using physics modeling and analysis to optimize the laser power throughout the scan path. Predicting the transient evolution of the thermal field to prevent overheating is critical for printing parts that meet quality criteria.

This study investigates control of the laser power during selective laser melting (SLM) of 316L stainless steel to build an overhang geometry. Diablo, a parallel finite element code that models the heat transfer is used with a control algorithm to optimize the laser power across the overhang. This study investigates strategies to use predicted physical quantities for feed forward control of the laser power. Parts printed using the feed forward control strategy are analyzed and simulation results are used towards the development of a geometry based model. Prepared by LLNL under Contract DE-AC52-07NA27344.

PM01.09.02
The Effect of Adhesion Strength Between Steel and Polymer on the Formability of Their Composites  
Sungjin Han, Jewook Yang and Woong-Ryeol Yu; Seoul National University, Seoul, Korea (the Republic of).

Due to global pressure to enhance energy efficiency, automotive industries have endeavoured to develop new lightweight structural materials. Steel/polymer/steel hybrid composites have been researched for this purpose because the weight of steel structure can be reduced maintaining its performance, e.g., the hybrid composites with a volume fraction of polymer of 30% theoretically shows that the flexural rigidity is maintained at 97%, but the weight is reduced to 75%, compared with the same volume of steel. In addition, these hybrid composites have showed improved damping and soundproof properties compared to pure steel structure. In order to apply those hybrid composites to industrial fields, their formability must be investigated in order to prevent the deterioration of mechanical properties of the hybrid composites during the forming process in various shapes. Therefore, experimental studies have been conducted to evaluate the formability of hybrid composites. However, there are few studies to predict the formability of the hybrid composites because the physical properties of core polymer and interfacial characteristics cannot be considered properly in simplified model. In this study, first formability tests were carried out for evaluating the forming limit diagram of the hybrid composite. After that, the formability test of the hybrid composites was simulated considering the viscoelastic properties of the core polymer and the interfacial properties. Finally, the effect of the interfacial adhesion on the formability of the hybrid composites was investigated, from which an optimal condition was explored for the interfacial adhesion strengths that can ensure the formability of the hybrid composites for specific applications.

PM01.09.03
Morphology and Composition of Magnesium Calcium Phosphates Prepared in a Multiple Emulsion  
Isao Kimura, Yuki Endo, Takaaki Tanaka and Yoshihara Taguchi; Niigata University, Niigata-shi, Japan.

1. Introduction
Inorganic hollow microspheres can be prepared by interfacial reaction (IR) in a W/O/W multiple emulsion. For controlling their morphology, it is worthwhile to know the transfer mechanism of the reactant ions from the outer aqueous (W_o) phase through the oil (O) phase to the inner aqueous (W_i) phase. In this study, calcium phosphate microspheres containing Mg were prepared by IR, and the effects of the solution concentration on the morphology and composition were investigated. Solution reaction (SR) was also carried out for discussing the transfer mechanism in IR.

2. Experimental
Solutions: K_2HPO_4 solution was used as W_i phase. Solutions of CaCl_2 and MgCl_2 at a Mg molar ratio X=Mg/(Ca+Mg) of 0.05–0.6 were used as W_o phase, containing Tween20. O phase was a Span80-cyclohexane solution.

<Interfacial reaction> W_i phase at C_i=0.3 mol/kg was poured into O phase and agitated to form a W/O emulsion. This was mixed with W_o phase at C_o=0.5 mol/kg to form a W/O/W multiple emulsion. The stirring was kept at 323 K for 18 h to prepare microspheres.

<Solution reaction> At 323 K, 100 cm^3 of W_o phase at C_o=0.5–0.5 mol/kg was added dropwise for 1 h into 300 cm^3 of W_i phase at C_i=0.50–0.3 mol/kg, and the stirring was kept for 18 h.

Characterization: The crystalline phase was identified by XRD. The morphology was examined by TEM. The Ca and Mg concentrations were measured by ICP-AES after dissolving in nitric acid. The relative deviation was defined as RD=(xs–xi)/xi, where xi and xs are the Mg ratios prepared by IR and SR, respectively.

3. Results and discussion
The crystalline phase of the product prepared by IR was predominantly Mg-TCP. A hollow morphology was observed, in which primary nanosheets assemble at the interface between W_i and O phases. As X was increased, primary round shaped particles were increased. x_i was as significantly low as one-tenth or less of X. On the other hand, x_i by SR was almost the same as X, whereas the predominant phase was also Mg-TCP. This result means that Mg at a given concentration can be taken in the product when reactant ions are mixed at a given concentration. Less x_i may be due to a lower concentration of the reactants at a reaction site. Therefore, it is thought that the mixing state of both the aqueous phases in IR should be inferred if the combination of C_i and C_o by which x_i becomes equal to x_s by IR could be found.
SR was carried out at a fixed \( C_\text{i} \). The resulting \( x \) was similar to \( X \). Under any condition, \( R \)D was much greater than unity. It was not consistent with the results by IR. Next, SR was carried out at a fixed \( C_\text{i} \). Lower \( C_\text{i} \) resulted in less \( x \). Regardless of \( X \), \( R \)D at \( C_\text{i}=0.03 \text{ mol/kg} \) was almost zero, which means consisting with IR. This result suggests that a low concentration of phosphate ions are mixed at a reaction site with a given concentration of Ca and Mg ions.

In the presentation, we will propose a transferring mechanism by assuming some carrier for ions.

**PM01.09.04**

**Challenges for the Additive Manufacturing of Shape Memory Ceramics**

Virag S. Raut1, Tom Glen2, Hang Yu1 and Steven T. Boles2; 1Electrical Engineering, Hong Kong Polytechnic University, Kowloon, Hong Kong; 2University of Edinburgh, Edinburgh, United Kingdom; 3Materials Science and Engineering, Virginia Tech, Blacksburg, Virginia, United States.

The multiplicity of phases (cubic, tetragonal, monoclinic, etc.) in zirconia-based systems has produced many opportunities for new applications and research directions. Interestingly, zirconia doped with ceria in certain compositions gives rise to a shape memory effect (SME) and superelasticity when exposed to applied stress and/or temperature. In this work, we have investigated SME behaviour in 12% ceria-doped zirconia (Cez0.88Zr0.12O2, CZ). CZ undergoes a martensitic transformation upon application of an applied stress which is marked by a phase change between tetragonal (T) and monoclinic (M) phases. The monoclinic content (MC) generated from the applied stress can subsequently undergo a thermally induced reverse phase transformation from M to T. The findings from our experiment suggest that the shape and morphology of CZ powder influences the MC generated during stress-induced transformation. A higher MC in pellets is produced after stress-induced transformation despite of a reduced MC i.e. fewer nucleation sites after annealing the CZ powder. The behaviour is split into two regimes: for lower temperature treatments, the morphology give rise to stress concentrations and contribute to a higher growth in MC; for higher temperature treatments, sintering and grain growth seemingly dominate the morphology and applied stress has a limited ability to increase MC. These results suggest conventional processing steps for bulk ceramics may be challenging. To avoid deleterious thermal treatments, a slurry based additive manufacturing approach is employed to produce porous ceramic structures. Infiltrating porous CZ with polymer materials using architected topologies and new design methods may lead to optimization of SME properties for meta-materials research and applications.

**PM01.09.05**

**Precise Synthesis for Designed Polymer Gel Networks from the Star Polymers and Its Properties**

Kwon Dowoo, Yuto Jochi, Yukikazu Takeoka, Takahiro Seki, Kotaro Satoh and Masami Kamigaito; Nagoya University, Nagoya, Japan.

Polymer gels have been continuously studied by researchers, because they exhibit useful functions for various applications. Also from an academic point of view, polymer networks of polymer gels have attractive properties both theoretically and experimentally. Generally, polymer networks can be easily prepared by free radical polymerization using monomers, initiators, and crosslinking agents. However, because the reaction process is extremly complicated in the reaction solution for preparing polymer gels, the resultant conventional gels have inhomogeneous and entangled polymer networks. While the structures of the polymer networks are closely related to the properties, the network structures including homogeneity, monomer sequence, and crosslinking points has been difficult to control by the methods so far. To design optimal network structures for applications, it is necessary to clarify the relationship between the structures of the polymer networks and those physical properties in an appropriate manner.

In this research, we introduced precise strategy to control the network structures and investigated thermal and mechanical properties of the network designed gels. Two types of preparative methods for synthesizing star-shaped polymers are sequentially employed. Firstly, star-shaped polymers are synthesized using ‘Core-first method’ by living radical polymerization utilizing tetra-functional initiator. The Atom transfer radical polymerization (ATRP) system of N-isopropylacrylamide(NIPA) was selected, because highly controllable polymerization system was essential to the detailed control of the networks. By the ‘Core-first method’, tetra-branched polymers with narrow polydisperity index (<1.1) were successfully synthesized. Secondly, according to ‘Linking methods’, the resultant star-shaped polymers are converted to the polymer networks by adding linking agents in one-pot. After adding the crosslinking agents when the monomer conversion reached over 95%, the polymer gels were obtained in 5 minutes. The combination of these methods facilitates to control the homogeneity and the sequence of monomers on the polymer networks.

Considering the experiment data, we have expected that the polymer gels have repetitive structure units composed of two types of sparse or dense star-shaped polymers, which are originated by the tetra-branched polymers and the crosslinking agents. Comparing with polymer networks prepared by free radical polymerization, there might be no inhomogeneity except for a few dangling chains derive from unreacted polymer chains. The physical properties of the polymer gels were investigated on the conventional gels and the network designed gels.

On the poster presentation, we would like to discuss about the detail of the synthesis data of the novel structure gels and the consideration about the structure-properties relationship.

**PM01.09.06**

**Optimizing Process Parameters in Commercial Micro–Stereolithography for Forming Polymer Microparticles in Non–Planar Microfluidic Devices**

Max Männel1, Ricardo Bernhardt1 and Julian Thiele1, 2; Leibniz-Institute of Polymer Research e.V., Dresden, Germany; 2Technische Universität Dresden, Dresden, Germany.

Droplet-based microfluidics is an established technique for fabricating microemulsions, microparticles and vesicles with tailored physicochemical properties for a broad range of applications in biology, chemistry, and material science. The state-of-the-art technique for fabricating flow cells to prepare microdroplets is a combination of photo-lithography and soft lithography based on poly(dimethylsiloxane) (PDMS), where a PDMS replica of microchannels is bonded to a glass slide, followed by post-processing steps, such as surface modification. While this multistep process yields microfluidic devices with feature sizes of a few micrometers in a parallelized fashion, each processing step is prone to experimental errors, which can make it challenging to reliably obtain high yields of functional microflow cells.

Additive manufacturing based on micro-stereolithography (µSL) is a promising alternative approach for fabricating microfluidic flow cells. This one-step three-dimensional (3D) printing technology is characterized by its high resolution and short process time. Utilizing a digital light processor, the 3D computer-aided design of a microflow cell is built up in a layer-by-layer fashion by exposing a liquid resin bath with stacks of xy-illumination patterns. Several parameters are optimized to make functional microflow cells, including the layer thickness, exposure time and energy, and the tilting up and down speed after each exposure.

To achieve resolution comparable to conventional PDMS-based fabrication methods and ultimately replace them, we have evaluated three key printing parameters in µSL: resin viscosity, alignment of the flow cell with respect to the printing direction, and the voxel compensation adjustment in the xy-plane. The same 3D-printed flow cell can be employed for forming both oil-in-water (ow/w) and water-in-oil (w/o) emulsions, with respective diameters of approximately 100 \( \mu \text{m} \) and 50 \( \mu \text{m} \). The emulsions then serve as templates for preparing hydrophobic or hydrophilic polymer microparticles with frequencies of up to 2.8 kHz in a single 3D-printed microflow cell without requiring additional steps for controlling microchannel wettability. In contrast to conventional multistep microfluidic device fabrication by photo- or soft-lithography, we report the preparation of non-planar microluidic flow cells in a single processing step that can be used for fabricating both hydrophobic and hydrophilic microparticles without requiring surface modification.

**PM01.09.07**
Bio-Inspired, Metal Based, Layer-by-Layer Structured Composites with Exceptionally High Toughness Yunya Zhang and Xiaodong Li; University of Virginia, Charlottesville, Virginia, United States.

Nature’s wisdom resides in constructing delicate architectures from limited constituents and achieving superior performances by the constructed structures. As exhibited in nacre, hard CaCO₃ platelets are cemented by soft biopolymers, forming laminated architecture. The hard CaCO₃ platelets act as main load bearers while the soft biopolymer layers dissipating energy and redirecting cracks. Such hard-soft-hard design enables a joint enhancement of strength and toughness, which are considered mutually exclusive in engineering materials. Therefore, nature inspires us that the dilemma between strength and toughness can be overcome by rational design of micro-scale architectures. In order to mimic nacre’s structures, graphene, a single layer of carbon atoms with extremely high strength, was used to composite with metals. In one study, graphene oxide sheets were incorporated with Al flakes. After freeze drying, sintering, and rolling, nacre’s three identifying features (laminated structure, nanoasperities, and mineral bridges) were effectively emulated in the composite. Graphene oxide released oxygen containing gases during high temperature annealing and the gases reacted with Al, forming AlOₓ/graphene/AlOₓ sandwiched layers at interfaces. The confluence of multiple strengthening and toughening mechanisms resulted in a joint improvement in hardness (210%), strength (223%), stiffness (78%), and toughness (30%). In another study, Ni powders were coated with graphene layers via shear mix and freeze dry. Carbon reacted with Ni at high temperature, forming rectangular NiCₓ second phase particles. The brick-and-mortar structure coupled with dissolved carbon atoms led to 97% improvement of strength with a minor reduction of ductility.

PM01.09.08
Fabrication of Electroactive ZnO Nanomesh via Vapor-Phase Materials Infiltration in Self-Assembled Pyridine-Based Diblock Copolymer Thin Films Ashwanth Subramanian², Kim Kisslinger¹, Daniel Yi³, Robert B. Grubbs³, Gregory Doerk¹ and Chang-Yong Nam¹, ²; ¹Center for Functional Nanomaterials, Brookhaven National Laboratory, Upton, New York, United States; ²Department of Materials Science and Chemical Engineering, Stony Brook University, Stony Brook, New York, United States; ³Department of Chemistry, Stony Brook University, Stony Brook, New York, United States.

Thin film nanoarchitectures can offer enhanced materials properties, such as large specific surface areas that amplify the structures’ interaction with environments, making the nanostructures useful for various sensing applications. Infiltration synthesis, a new-type of organic-inorganic material hybridization technique derived from atomic layer deposition, relies on the vapor-phase inorganic precursor infiltration into polymer matrix. When applied to self-assembled templates of diblock copolymer (BCP) thin films and combined with the subsequent removal of polymer matrix, the technique enables a real-scale large-volume inorganic material nanopatterning. Although the infiltration synthesis of alumina (Al₂O₃) nanostructures using self-assembled polystyrene-block-poly(methyl methacrylate) (PS-b-PMMA) BCP thin films has been well documented, the generation of more useful semiconductor nanostructures, such as those made of zinc oxide (ZnO), has been relatively scarce, unless the pre-infiltration (i.e., priming) of Al₂O₃ is applied, due to the weaker interaction of Zn precursor with polymers in general. Here, we demonstrate the successful infiltration synthesis of pristine ZnO nanostructures without resorting to the Al₂O₃ priming, by taking advantage of more chemically reactive self-assembled polystyrene-block-poly(2-vinylpyridine) PS-b-P2VP BCP thin films as well as the newly developed, micro-dose infiltration synthesis protocol. By exploring the self-registrering phenomenon between successively coated self-assembled BCP thin films, we generate the electroactive, stacked ZnO nanomesh thin film architecture with the controlled number of stacked layers. The electrical characterization under dark and illuminated conditions reveals an enhancing electrical conductance with increasing number of nanomesh stack layers. The results not only illustrate the first demonstration of electrical functionality based on the ZnO nanooarchitecture generated by the infiltration synthesis in self-assembled BCP thin films but also present a new, large-area scalable, metal oxide thin film nanoarchitecture fabrication method utilizing industry-compatible polymer solution coating and atomic layer deposition. The fabricated ZnO nanomesh architecture also promises potential applications as an efficient active sensing medium for chemical and optical detectors.

PM01.09.09
The Conformality of Shell Structures on GLAD Growth Rod Core Structures by Monte Carlo Simulation Mesut Yurukcu¹, Serkan Demir¹ and Fatma Merve Yurtsever; ¹University of Arkansas, Little Rock, Arkansas, United States; ²College of Computer and Information Sciences, Regis University, Denver, Colorado, United States.

Shell-core nanorod array geometry can offer enhanced durability for polymer electrolyte membrane (PEM) fuel cell electrodes, and increased surface-volume ratio for high efficiency batteries, solar cells, sensors if a structured shell is conformally coated around the nanorod support base. The quality of the shell coating around nanorod is as crucial as the quality of the nanowires in device applications. In this study, we performed an experimental investigation of different physical vapor deposition (PVD) techniques to discover the most efficient deposition technique for a conformal shell coating around nanorods. To assess the PVD conditions to produce conformal shell coatings, Monte Carlo simulation method (MC) can provide useful guidance as well as helping investigate fundamental growth dynamics during shell-core nanorod array fabrication. Monte Carlo simulations were conducted to determine a simple and scalable fabrication technique for conformal and uniform shell coatings. We conducted Monte Carlo simulation (MC) and investigated the conformality of PVD shell layers on nanorods of the same length to predict the different materials. Our results show that high pressure with normal angle deposition can generate better conformal shell coating inside all others. The results suggest that an atomic flux with an angular distribution which can be easily maintained by sputtering at high working gas pressure can form conformal and uniform coatings. Our results indicate that conventional PVD techniques, which offer low cost and large scale thin film fabrication, can be utilized for highly conformal and uniform shell coating formation in shell-core nanostructure devices. In our previous works, the pre-prepared template was used to run the shell coating simulations. In this paper, we investigated the shell conformality on situ grown GLAD rod structures by Monte Carlo simulation. This work will give the reader an understanding of how significant the geometry and the design of the rod structured arrays play a role in core-shell PVD coating device applications.

PM01.09.10
Study on Array Transmittance Improvement of Thin-Film Transistors Chengzhi Luo; Wuhan University, Wuhan, China.

Transparent electronics, such as thin film transistors, is one of the most advanced topics for a wide range of device applications. The key components are wide bandgap semiconductors, not only as passive component but also as active component, similar to what is observed in conventional semiconductors like silicon. Transparent electronics has gained special attention during the last few years and is today established as one of the most promising technologies for leading the next generation of flat panel display due to its excellent electronic performance. In order to achieve the high performance, TFTs need to possess high light efficiency. Transmittance improvement is the main method to improve light efficiency of TFT array substrate. In this paper, multilayer thin films of low temperature poly-Si array transmission area is made to study the effect of thin films and structure on transmittance. The results show that the transmittance of TFTs is greatly affected by different refractive index surface after inter layer dielectrics finished. We also find that that the effect of planarization layer thickness on transmittance is not obvious. Transmittance is decreased with the increasing of ITO layer thickness. The results show that the effect of planarization layer thickness on transmittance is not obvious. Transmittance is decreased with the increasing of ITO layer thickness. Transmittance improvement is the main method to improve light efficiency of TFT array substrate. The electrical characterization under dark and illuminated conditions reveals an enhancing electrical conductance with increasing number of nanomesh stack layers. The results not only illustrate the first demonstration of electrical functionality based on the ZnO nanooarchitecture generated by the infiltration synthesis in self-assembled BCP thin films but also present a new, large-area scalable, metal oxide thin film nanoarchitecture fabrication method utilizing industry-compatible polymer solution coating and atomic layer deposition. The fabricated ZnO nanomesh architecture also promises potential applications as an efficient active sensing medium for chemical and optical detectors.

PM01.09.11
Autonomous Printing of Functional Contact-Stylus Circuits Annika Muehlbradt and Gregory Whiting; University of Colorado Boulder, Boulder, Colorado, United States.

The results show that the transmittance of TFTs is greatly affected by different refractive index surface after inter layer dielectrics finished. We also find that that the effect of planarization layer thickness on transmittance is not obvious. Transmittance is decreased with the increasing of ITO layer thickness. Transmittance improvement is the main method to improve light efficiency of TFT array substrate. The electrical characterization under dark and illuminated conditions reveals an enhancing electrical conductance with increasing number of nanomesh stack layers. The results not only illustrate the first demonstration of electrical functionality based on the ZnO nanooarchitecture generated by the infiltration synthesis in self-assembled BCP thin films but also present a new, large-area scalable, metal oxide thin film nanoarchitecture fabrication method utilizing industry-compatible polymer solution coating and atomic layer deposition. The fabricated ZnO nanomesh architecture also promises potential applications as an efficient active sensing medium for chemical and optical detectors.
Liquid crystals (LC) is a phase of material which is intermediate to a crystalline solid and an isotropic liquid such that the molecules flow but retain a degree of long-range orientational ordering. LCs, due to their long-range orientational ordering and fluidic properties, can be used in templated synthesis of polymeric materials. In the past studies of oil-in-water emulsion based polymeric microparticle synthesis, it was showed that control over the particle shape, organization of polymer chains and incorporation of porosity can be achieved when LCs were used as the oil phase[1]. In this study, we indicated that polymeric films templated from liquid crystals provide basic design principles for the synthesis of films with predetermined pore alignment. For the synthesis of the material, we photopolymerized the mixture of reactive (4-acryloyloxypropyloxy) benzoic acid 2-methyl-1,4-phenylene ester (RM257) and nonreactive (4-cyano-4′-pentylbiphenyl (5CB)) mesogens confined in film geometries with thickness of 20-200 μm, and then extracted the unreacted mesogens with solvent to yield polymeric films of area in the order of 10 cm². The crucial point of this study was that, when we constrained the polymer
films to an area either through a mechanical or a configurational constraint, open pores were incorporated into the films. The average diameter of the pores was found to be in the range 10-40 nm, and can be tuned by varying the reactive monomer concentration. In fact, the average direction of the pores was found to be determined by the nematic director which can further be controlled by the functionality of the contacting surfaces. Having control over the sizes and directions of the pores has enabled the material to be involved in many application areas. We found that the range of the pore sizes and the alignment behavior of the pores can potentially be used for the ultratransliteration purposes as one of these applications. We also demonstrated a successful separation of protein molecules and solid nanoparticles from aqueous media using polymeric films templated from liquid crystalline media, the mass transfer performance of which was also dependent on the alignment direction of the pores with respect to the surfaces. For the synthesized polymeric materials, we used characterization methods such as thermal, optical, mechanical properties, nitrogen adsorption porosimetry and electron microscopy. Overall, the outcomes of this study provide basic tools for the synthesis of porous polymeric films with predetermined pore directions that can potentially be suitable for separation purposes, drug delivery, catalysts, etc.


**PM01.09.16**

Additive Manufacturing of Hierarchical Three-Dimensional Micro-Architected Graphene Aerogels Xiaohong Shao, Xiaoyu Zheng; Virginia Tech, Blacksburg, Virginia, United States.

Graphene foams, despite having one of the stiffest base materials ever measured, suffer incredible degradation in mechanical properties with decreasing density, so much so that polymer foams can outperform graphene foams at low-densities, despite the polymer base material being significantly less stiff than a graphene sheet. Until now there were few predictable means with which experimental approaches to alter this paradigm.

Herein we developed a light based 3D printing process to rapidly create three-dimensional graphene lattices of essentially any desired structure with graphene strut microstructure having pore sizes on the order of 60 nm. This flexible technique enables printing 3D micro-architected graphenes with complex, high-resolution form factors unparalleled by previous graphene 3D printing techniques. We utilize this ability to demonstrate improved mechanical properties when scaling to lower densities due to control of its three-dimensional hierarchical micro-architecture. These 3DGs are ultra-light, possess large surface area, and maintain good electrical conductivity, while greatly expanding the micro-architectural design space that 3DGs can realize.

**PM01.09.17**

One Pot Synthesis of a Gel with a Homogeneous Network Structure by Combining Two Methods of Synthesizing Star Polymers Yusuke Baba, Kwon Dowoo, Yuto Jochi, Yukikazu Takeoka, Takahiro Seki, Kotaro Satoh and Masami Kamigaito; Graduate School of Engineering, Nagoya University, Nagoya, Japan.

A polymer having a three-dimensional network structure and absorbing a solvent is called a gel. A gel is expected to be applied to many functional materials because of their simple adjustment method and various functionalities. Generally, when a gel is synthesized by free radical polymerization, the gel has an inhomogeneous network structure. The inhomogeneity of the network structure of the gel influences the properties of the gel. However, the relationship between the homogeneity of the network structure and the properties of the gel has not been investigated in detail. This is because there are few methods for synthesizing a gel with a homogeneous network structure. In order to investigate this relationship, our research group succeeded in synthesizing a PNIPA (poly(N-isopropylacrylamide)) gel with a homogenous network structure by combining two methods of synthesizing star polymers.

One method is a core-first method in which star polymers are synthesized by living polymerization using multifunctional initiators. Another method is a linking method in which star polymers are synthesized by adding divinyl compounds to linear living polymers and crosslinking the polymer terminations. In our study, a star polymer is synthesized from a 4-branched initiator by living radical polymerization (core first method), and then the polymer chain ends are crosslinked by adding divinyl compounds (linking method). Thereby, a gel with a homogeneous network structure can be obtained. We believe that research on how inhomogeneity affects the properties of a gel will proceed on this gel. In this study, in order to investigate the correlation between the network structure and various physical properties in a solvent-free elastomer, a monomer in which the polymer becomes liquid at room temperature was used. So we prompt to synthesize a PMEO2MA (poly(2-(2-methoxyethoxy)ethyl methacrylate)) gel which can take an elastomer state with a homogeneous network structure and measure the mechanical properties. First, we prepared 4 branched star-shaped PMEO2MA by the core first method in order to check the molecular weight and polydispersity index of the polymer. We polymerized ME02MA by ATRP (atom transfer radical polymerization) using 4 branch initiator, CuCl for catalyst, and bipy for ligand in anisole. As a result, we obtained 4 branch PMEO2MA (conversion%=84.6%, Mw=48000, Mw/Mn=1.19) in 21h.

Secondly, we synthesized star polymers by the linking method in order to confirm the progress of linking reaction. We synthesized linear polymers by ATRP and subsequently after 21h, 20 equivalents of divinyl compound, DEGDMA (Diethylene glycol dimethacrylate) to initiator was added to the reaction solution. After 48h, we confirmed a lot of linear polymer terminations crosslinked. Finally, by combining these star polymer synthesis methods, a homogeneous polymer network consisting of MEO2MA was successfully synthesized.

**PM01.09.18**

Strong Interaction Between the Long Period Stacking Ordered (LPSO) Structure and Deformation Twin in Mg-Zn-Y Alloy Xiaohong Shao, Chao He1,3, Xiuliang Ma2 and Qiang Chen1; 1Department of Mechanical Engineering, Kyushu University, Fukuoka, Japan; 2Shenyang National Safety and Disaster Mechanics, Sichuan University, Chengdu, China.

The high hardness or yield strength of an alloy is known to benefit from the presence of small-scale precipitation, whose hardening effect is extensively applied in various engineering materials. Stability of the precipitates is of critical importance in maintaining the high performance of a material under mechanical loading. The long period stacking ordered (LPSO) structures play an important role in tuning the mechanical properties of a magnesium alloy. Here, we report (10-12) deformation twinning induces decomposition of lamellar LPSO structures and their re-precipitation in an Mg37Zn17Y4 (at. %) alloy. Using atomic resolution scanning transmission electron microscopy (STEM), we directly illustrate that the misfit dislocations at the interface between the lamellar LPSO structure and the (10-12) deformation twin are corresponding to the decomposition and re-precipitation of LPSO structure, owing to dislocation effects on redistribution of Zn/Y atoms. This finding demonstrates that deformation twinning could destabilize complex precipitates. An occurrence of decomposition and re-precipitation, leading to a variant spatial distribution of the precipitates under plastic loading, may significantly affect the precursor's strengthening. Regarding the compression deformation behavior (10-13) twin stacking faults (SFs) enriched with solute atoms in the Mg27Zn14Y (at. %) alloy. The pyramidal-basal (Pyb) and basal-pyramidal (BPy) interfaces along SFs make faceted (10-13) twin boundary deviate appreciably from (10-13) twin plane. (10-13) twin associated with shear can reorient one unit of SF, but engulf two successive SFs. (10-13)(10-11) twin segment generated upon twin-SFs intersection region, and is proposed to arise from dislocation dissociation. The direct observations unravel some of the fundamental characteristics of interaction between compression twin and precipitates in magnesium alloys. In summary, we can expect strong interaction between LPSO phase and deformation twins in the Mg-Zn-Y alloy, with significant effect on the corresponding mechanical performance.
In an effort to systematically engineer the performance of multiphase materials we have developed a new electronic-ionic based coupled phase-field crystal (PFC) methodology.\textsuperscript{1,2} Our results are underpinned by a deep analysis of the free energy functional formulation which leads to the addition of an electronic density well below 1000 kg m\textsuperscript{3}, significantly enhanced stiffness (\textsim\ 5 times), and superior elastic recoverability compared to its polymer counterpart due to its composite nature. The findings imply that this highly scalable and effective route to synthesizing HEA-coated microlattices have the potential to produce novel metamaterials with desirable properties to cater specialized engineering applications.

PM01.09.19
Mechanical Enhancement of Core-Shell Microlattices through High-Entropy Alloy Coating
James U. Surjadi, Libo Gao and Yang Lu; City University of Hong Kong, Kowloon Tong, Hong Kong.

Mechanical metamaterials such as microlattices are an emerging kind of new materials that utilize the combination of structural enhancement effect by geometrical modification and the intrinsic properties of its material constituents. Prior studies have reported the mechanical properties of ceramic or metal-coated composite lattices. However, the scalable synthesis and characterization of high entropy alloy (HEA) as thin film coating for such cellular materials have not been studied previously. In this work, stereolithography (3D-printing) was combined with Radio Frequency (RF) magnetron sputtering to conformally deposit a thin layer (~ 800 nm) of CrMnFeCoNi HEA film onto a polymer template to produce HEA-coated three-dimensional (3D) core-shell microlattices for the first time. The presented polymer/HEA hybrid microlattice exhibits high specific compressive strength (~ 0.018 MPa kg\textsuperscript{-1} m\textsuperscript{3}) at a density well below 1000 kg m\textsuperscript{3}, significantly enhanced stiffness (~ 5 times), and superior elastic recoverability compared to its polymer counterpart due to its composite nature. The findings imply that this highly scalable and effective route to synthesizing HEA-coated microlattices have the potential to produce novel metamaterials with desirable properties to cater specialized engineering applications.

References:

PM01.09.20
Self-Assembly Based Fabrication of Scalable Free-Standing Nanoporous Nickel with Large Crack-Free Areas
Zhimin Jiang and James H. Pikul;

Nanoporous metals with architected nanometer-scale pores have many applications including photonic crystals, filtration membranes, biosensors, MEMS devices, and electrodes for high power batteries. Self-assembly from colloids or suspensions, followed by metal deposition into the space between self-assembled particles, is one of the most promising ways to fabricate scalable nanoporous metals with controlled nanoscale architecture, but self-assembly techniques result in a high density of cracks (~1 crack per 10 – 100 mm) due to residual strains in assembled films. In addition, the resulting nanoporous metals are typically bonded to the substrate, which prevents them from being used in applications requiring free-standing films.

Here, for the first time, we present a method for self-assembly based fabrication of scalable free-standing nanoporous nickel films with crack free areas larger than 1 mm\textsuperscript{2}. We fabricate them by first self-assembling 200 – 500 nm polystyrene (PS) particles into single-crystalline FCC packed films on indium tin oxide (ITO) coated substrates. We then electrodeposit nickel through the film voids and etch the PS particles. By controlling the PS diameter, sintering time, and adding material post-fabrication, we can precisely control the material architecture and chemical composition with 10 nm precision over cm\textsuperscript{2} areas.

To reduce crack density, we used experiments and simulations to study the crack formation and found two dominant crack types: cracks between the substrate and the PS film that caused the PS film’s delamination (d-cracks), and vertical cracks in the PS film that resulted in a network of through cracks (c-cracks). Our experiments showed that the large residual strains were due to shrinkage of polymer chains stabilized by particles’ surface nanobubbles and disappearing of electrical double layers generated by surface charges. Functionalizing the ITO substrate with sulfonate groups increased adhesion between PS nanoparticles and ITO, which prevented d-cracks. Decreasing the polymer chain thickness using a low-pressure self-assembly method reduced the residual strain and the c-crack density in PS films. Since electrical double layers disappear when PS films dry, keeping the PS films wet during self-assembly was critical for preventing the residual strain release and preventing c-cracks. Our theoretical model and experiments showed that adding ethylene glycol as a low evaporation rate additive into the nanoparticle colloid kept PS films wet and enabled PS films with large crack-free areas. Combining these results, we successfully fabricated free-standing nanoporous nickel inverse opal films with large crack-free areas. The low adhesion strength of ITO to metals enabled facile delamination of the inverse opal films to make them free-standing. Our fabrication method makes it possible to further exploit the unique properties of metals with architected nanosized pores and realize them in large area engineering applications.

PM01.09.21
Visualization and Mechanical Study of a Transparent Filled Rubber
Zach Gault, J. Zsolt Terdik, Joerg Werner, Frans Spaepen and David Weitz;
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Filled rubbers are composite materials containing two interpenetrating phases: crosslinked elastomers, and a ‘filler’ consisting of colloidal particle aggregates. Above a critical volume fraction, the colloidal aggregates form a system-spanning subnetwork that reinforces the elastomer network and introduces a new energy loss mechanism at low strains of only 1-5%. This loss mechanism, known as the Payne Effect, is one of the mechanical hallmarks of filled rubbers and is a major contributor to rolling friction in tires.

We create a model filled rubber which exhibits the rheological hallmarks of traditional filled rubbers, but can be optically imaged. Optical transparency is of filled rubbers and is a major contributor to rolling friction in tires.

Combining these results, we successfully fabricated free-standing nanoporous nickel inverse opal films with large crack-free areas. The low adhesion strength of ITO to metals enabled facile delamination of the inverse opal films to make them free-standing. Our fabrication method makes it possible to further exploit the unique properties of metals with architected nanosized pores and realize them in large area engineering applications.

PM01.09.22
Jellium Phase-Field Crystal Model—The Thermodynamic Influence of Electronic Contributions
Salvador Valtierra, Nan Wang, Nikola Ofori-Opong, Kirk B. Bevan; McGill University, Montreal, Quebec, Canada; Canadian Nuclear Laboratories, Chalk River, Ontario, Canada.

In an effort to systematically engineer the performance of multiphase materials we have developed a new electronic-ionic based coupled phase-field crystal (PFC) methodology.\textsuperscript{1,2} Our results are underpinned by a deep analysis of the free energy functional formulation which leads to the addition of an electronic free energy term in PFC.\textsuperscript{3,4} This functional is normally constructed based on an atomic number density in classical density functional theory (CDFT).\textsuperscript{3,5} Through a series of approximations from CDFT and a mean-field approach, PFC permits the modelling of multiple phases and observe microstructural features in materials. In this work, a jellium system is employed as framework to...
formulate a free energy which allows the visualization of the electron energetics that modify the stability of liquid and crystalline phases modeled in PFC. We present a profound discussion of the physical implications behind the construction of the free energy functional and architected microstructural consequences. This formulation would extend the model to study the influence of electronic features and external forces acting on the microstructure in order to tailor the multi-functional properties of materials. The aim of this study is to provide a new perspective on PFC, that will permit the inverse design of new phenomena in materials science modeling.


PM01.09.23
Optical Properties of an Exciton in a AlN/GaN/AIN Spherical Core/Shell Quantum Dot Under Pressure Mostafa Sadoqi1, Noreddine Aghoutane2, Mohamed El-Yadri3, Asmaa El Haouari3, Elmustapha Feddi3, F. Dujardin3, Chuong V. Nguyen4, Nguyen N. Hieu4, Huynh V. Phuc5 and Gen Long1; 1Saint John's University, Jamaica, New York; United States; 2Group of Optoelectronic of Semiconductors and Nanomaterials, ENSET, Mohammed V, Rabat, Morocco; 3LCP-A2MC, Institut de Chimie, Physique et Matériaux, Université de Lorraine, Metz, France; 4Institute of Research and Development, Duy Tan University, Da Nang, Viet Nam; 5Division of Theoretical Physics, Dong Thap University, Dong Thap, Viet Nam.

This work is based on our recent theoretical investigation of the effects of the hydrostatic pressure and core/shell sizes on the optical properties associated with the transition from the ground state into the first excited state (1s-1p), of the exciton in a spherical core/shell quantum dot (SCSQD). Our calculations are performed in the framework of the effective mass approximation and the energies are obtained by using a Ritz variational method. We have computed the linear, third-order nonlinear and total absorption coefficients (AC) and refractive indices (RI) as a function of photon energy for different sizes of SCSQD under the influence of the hydrostatic pressure. Our results show that the optical absorption is strongly dependent on the incident optical intensity and sensitively influenced by the confinement and pressure effects. We have found that, by increasing pressure, the AC and RI curves move toward the higher energies (blue shift). Our calculation also shows that the AC and RI are affected by the core/shell quantum dot sizes.

PM01.09.24
Direct Ink Writing of Polyimide Composites with Density/Dielectric Tunability H. Clive Liu1,2, James O. Hardin1,2 and John D. Berrigan2; 1UES Inc., Beavercreek, Ohio, United States; 2Air Force Research Laboratory (AFRL), WPAFB, Ohio, United States.

Polyimide has been an important engineering material with excellent oxidative- and thermal-stability, mechanical performance, and dielectric properties that can be employed in harsh environments. However, architectural design and exploration of novel polyimide composites are still hindered by the limited manufacturing and property-tailoring strategies. In this work, hierarchical construction of polyimide can be achieved through imidizing the additive-manufactured precursor polyamic acid or photo-curing a chemically modified polyimide. Yet these 3D process-ability are reported at the respective expense of significant shrinkage (≥ 40%) accompanied by the imidization reaction or a pronounced decrease (100 – 200 °C) in degradation temperature compared to other polyimide analogues.

In this study, we present 3D polyimide architectures through direct writing of fully-imidized polyimide ink without photo-crosslinking or post-print thermal/chemical imidization. The printed structures are programmed with intrinsic micro-porosity through a simple formulation strategy that acts to suppress density, dielectric permittivity, and dissipation. Moreover, the development of composite ink formulations allow tailoring of functionality (e.g., electrical/thermal conductivity, high-dielectric) into the structures. These micro-porous and composite polyimide materials enable the investigation of cellular and graded architectures to manipulate mechanical response, structural mechanics, and electromagnetic wave steering efficiency for future 3D printed antenna applications.

PM01.09.25
3D Direct-Nozzle Printing of Ceramic Magnetic Core for Transformers with Viscoelastic NiZn-Ferrite Paste Taekyu An and Jihoon Kim; Konju National University, Cheonan, Korea (the Republic of).

3D printing technology recently attracts a significant amount of attention due to its potential to easily fabricate complex designs of diverse products. This technical convenience leads to the step where 3D printing technology is considered an innovative and simple way of manufacturing ceramic-based electronic components. In this study, 3D direct-nozzle printing was introduced to fabricate various shape of the NiZn-ferrite soft magnetic core for transformers. The NiZn-ferrite paste for 3D direct-nozzle printing was formulated by incorporating various functional additives to impart viscoelastic property. This viscoelastic property of the paste plays a critical role in printing and maintaining the 3D shape of the transformer core. The correlation of viscoelastic property with 3D printability will be addressed in depth in this presentation. The 3D-printed magnetic cores of different designs were sintered at an elevated temperature. The volume shrinkage and magnetic functionality were investigated after sintering. Finally, we printed EFD type transformer cores and compare their performance against commercial transformers with the same type. Our successful demonstration implies the importance of viscoelasticity in 3D printing inks and the potential of 3D printing as a manufacturing process for ceramic-based electronic components.

PM01.09.26
Application of Box Benhken Experimental Design in the Fabrication of Anodized Aluminium Oxide Templates from Low Purity Substrates and Its Escalation to Extended Areas Lima M. Castro and Carlos Osto; Facultad de Ciencias Exactas y Naturales, Universidad de Antioquia, Medellin, Colombia.

In the last years, the scientific community has developed multiple variations of the aluminum anodization process, with the goal to prepare nanostructures to be adapted for new technologies. The controllable microstructural parameters, self-organized pore-arrangement, parallel nanochannels, narrow pore-size distribution, and high pore density, open a wide range of possibilities for nanomaterial synthesis and the opportunity to module the properties of the periodic nanostructures. Therefore, an optimization of the experimental conditions is mandatory to achieve the desired dimensions and best arrangements of pore features; hence providing the best performance of derived nanostructures [1].

In this work, the influence of the most important operating conditions, such as potential (30V-60V), temperature (10°C-20°C), and concentration of electrolyte (0.3M-0.6M), on the structural features of alumina was investigated by means of Box-Benken experimental design (BBD). A low purity...
aluminium foil, 99.06%), was selected to accomplish the experiments because of its low cost and high suitability when addressing real applications. The aluminium foils were first machined with an area of 1cm² and degreased using a mixture solution of acetone/ethanol; afterwards, the foils were processed using oxalic acid solutions with concentrations from 0.3M to 0.6M, temperature range from 10°C to 20°C, and potential from 30V to 60V, these parameters were used as input factors for the BBD design. The obtained models granted us the possibility to understand the individual and interaction effects of the three operational parameters on the steady state current and pore features, such as regularity ratio (Rₑₑ), pore diameter (Dₑₑ), eccentricity (Eₑₑ), aspect ratio (AR), pore density (pₑₑ), interpore distance (Dₑₑ), and porosity percentage (%P). Potential and temperature were found to significantly affect most of the assessed geometrical pore features of the Box-Behnken design. The regularity ratio was optimized with a value of 2.0134 under the next conditions: 46V-10°C-0.6M.

The optimized set of conditions were used for the scaling up of the process in 78cm² aluminium foils. We have found that anodization under these conditions can produce porous alumina membranes with defined pore diameters and thicknesses obtained by adjusting the anodizing duration and pore opening/widening time,“J. Solid State Electrochem., vol. 15, no. 11–12, pp. 2427–2436, 2011.

PM01.09.27
Monolayer-to-Thin-Film Transition in Supramolecular Assemblies on Graphene—The Role of Topological Protection
Alexander J. Marsden1,2, Zachary Laker2, Orieste De Luca1, Ada Della Pia1, Luis M. Perdigão3, Giovanni Costantini1 and Neil Wilson2; 1Materials, University of Manchester, Manchester, United Kingdom; 2Physics, University of Warwick, Coventry, United Kingdom; 3Chemistry, University of Warwick, Coventry, United Kingdom.

Supramolecular assembly is a promising route for the bottom-up synthesis of nanomaterials.1 The resulting structure is guided by the non-covalent forces between molecules, and those between a substrate and the molecules.2 The interactions with the substrate can template up through the film: successive layers of molecules are influenced by each other, and the resulting structure can be different from its expected bulk form.2 These structured nanomaterials show promise for applications in electronics and optoelectronics.4 Here we study the supramolecular assembly of benzene-1,4-dicarboxylic acid (terephthalic acid, TPA), and benzene-1,3,5-tricarboxylic acid (trimesic acid, TMA) on graphene. The thin film structure is investigated using low-dose, aberration-corrected transmission electron microscopy (TEM) as the film thickness increases, and is compared to the monolayer structure found using scanning tunneling microscopy (STM).

At the monolayer, both molecules display a structure dictated by hydrogen bonding between molecules: TMA forms a hexagonal chicken-wire structure, and TPA, a brickwork pattern. The orientations of these 2D monolayers are influenced by the graphene substrate through van der Waals epitaxy, although this is stronger in the case of TMA. Above a monolayer, TMA and TPA behave differently. The chicken-wire structure of TMA templates up through increasing film thickness, until after a certain critical thickness when the film becomes polycrystalline with random in-plane orientations. On the other hand, after a monolayer, TPA forms fibre-like islands, with the in-plane lattice parameters gradually changing with increasing thickness to those consistent with the bulk structure. These differences are thought to be anchored in the differences between the 2D and bulk structures for the two molecules. TPA can transition from its 2D structure to its bulk form through tilting and compacting of the lattice. However, TMA’s 3D structure consists of interleaving planes, which cannot be smoothly transitioned to as the film thickness increases. This idea of topological protection could be used to help design supramolecular assemblies in the future.


PM01.09.28
Bioinspired Design of Cement-Polymer Composites
Jessica Rosewitz, Habibeh A. Choshali and Nima Rahbar; Worcester Polytechnic Institute, Worcester, Massachusetts, United States.

Recent advances in the design of structural composites often mimic natural microstructures. Specifically, the structure of abalone nacre with its high stiffness, tensile strength, and toughness is a source of inspiration from the process of evolution. The inspiration from nacre can lead to design of a new class of architected structural materials with superb mechanical properties. This work presents a combined experimental and computational study on a set of bioinspired architected composites created using a cement mortar cast with brick-and-mortar and auxetic polymer phases. The impact of this unit-cell architected polymer phase on the flexural and compressive strengths, resilience, and toughness is thoroughly studied as a function of microstructural geometry. All mechanical properties of the architected composite specimens are found to be greater than those of control samples due to prevention of localized deformation and failure, resulting in higher strength. The microstructurally designed composites showed more layer shear sliding during fracture, whereas the control samples showed more diagonal shear failure. After initial cracking, the microstructurally designed composites gradually deformed plastically due to interlocking elements and achieved high stresses and strains before failure. Results also show that microstructurally designed composites with the architected polymer phase outperform control samples with equal volume fraction of a randomly oriented polymer fiber phase. Extensive computational studies of the proposed unit cells are also performed and the results suggest that the orientation of cells during loading is critical to achieve maximum performance of a cementitious composite. The implications of these results are immense for future development of high performing construction materials.

PM01.09.29
Fabrication of Photo-Activating Acryl-polyurethane Through Short Curing Time
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Thermoplastic photo-activating PU was successfully synthesized by the additional reaction of methylene diphenyl diisocyanate (MDI), poly(tetramethylene ether) glycol, and tri-acrylate derivatives as a crosslinking point. The crosslinked PU-acrylate elastomers were fabricated by the exposure to 200–400μm UV radiation. DMF was used as a solvent for the synthesis, but THF with low UV cut off was used to crosslinked the PU-acrylate in order to reduce crosslinking time. The structures and properties of the resulting acryl-PUs were evaluated by fourier transform infrared spectroscopy (FT-IR), gel permeation chromatography (GPC), ultra violet spectroscopy (UV-Vis), differential scanning calorimetry (DSC), and universal testing machine (UTM). The increase of acrylate concentration in acryl-PUs elastomers led to higher tensile strength and hardness due to the increased crosslinking density and the
enhanced interchain hydrogen bonding. The application as a 3D printing material was verified according to the shortened crosslinking time.

**PM01.09.30**

The Effects of Dynamic Transformation on the Formation of Pt-Ni Nano-Octahedra

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Colloidal synthesis has been widely used in the development of nanomaterials including metals, alloys, and semiconductors, as it can produce uniform, size-controlled and shape-controlled nanoarchitectures. It is usually desired for shape-controlled products to expose exclusive facets, correlating a collective property such as reaction activity or selectivity with the specific surface crystal planes. Compared with other conventional approaches such as the hydrothermal method, the solution-based synthesis has more variable factors that govern the formation of the nanocrystal products, due to the kinetic and thermodynamic effects in the nucleation and crystal growth processes, such as a competition between the deposition and diffusion of “free atoms” on a nanocrystal. These factors greatly impact the morphology evolution of the products with some in-depth mechanistic insights to uncover. In our synthetic system, a non-polar organic solvent is usually chosen as the reaction medium to facilitate a formation of shape-controlled nanocrystals at an elevated temperature through the hydrophobic interaction between the solvent and the hydrocarbon chains in the selected capping ligands. Thus, the mass and heat transfer that is closely associated with the progress of the nanocrystal evolution could be greatly influenced by some intrinsic properties of the solvent such as the viscosity and by some tunable conditions. In the latter, stirring rate and the ramp rate of heating are identified as two of the significant factors that could alter the balance between the deposition and diffusion of “free atoms” on a nanocrystal. Nevertheless, there are limited reports of studying these factors. Herein, we present our progress of such a study using a synthesis of Pt-Ni octahedral nanocrystals in the presence of W(CO)6 as an example. A slow stirring rate (~200 rpm) and high ramp rate for heating (8 °C/min) resulted in pod-like Pt-Ni nano-products, whereas their counterpart conditions (~400 rpm and 5 °C/min) generated uniform Pt-Ni nano- octahedra when other experimental parameters remained the same. Based on our observation, a plausible mechanism is proposed. One hand, the slow stirring rate and high ramp rate of heating can promote the atom deposition on a nanocrystal, leading to pod-like products due to an accumulation of the ad-atoms on the same planes of a seed. On the other hand, the relatively high stirring rate and slow ramp rate of heating can preserve sufficient time to allow the deposited atoms to re-distribute to those thermodynamically stable sites. In other words, the domimative diffusion, in this case, is the driving force of the nano-octahedron formation.

**PM01.09.31**

Cement Mortar Reinforced with Polypropylene Mesh

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Concrete is one of the world’s most widely used building materials for many reasons including: relatively low cost, moldability, and high compressive strength. This high compressive strength is perfect for most construction applications where the building is subjected to constant and well known static forces. However, due to concrete’s brittle nature, crack formation and ultimately failure will occur when it is exposed to dynamic or tensile loading; concrete is often subjected to such conditions in highway and military applications. Polymeric fibers, namely Polypropylene (PP), are often added to the concrete mix in order to promote toughness and impact resistance, improving the survivability of concrete under such loading conditions. In this work we consider PP mesh in lieu of fibers as impact modifier for cement based structures. In brief, we have studied the effect of the mesh addition to cement mortar on physical properties, including impact resistance. It is suggested that mesh reinforcement can offer better improvements to toughness due to its connectivity and, therefore, ability to serve as macro scale reinforcement. Samples were prepared using a cement mortar mixture of constant composition (large aggregates were excluded due to the cm-scale sample size) and reinforcement with ~ 2% by volume of varying sized PP meshes. The samples were subjected to compression, tensile splitting, and impact testing in order to quantify their mechanical properties. The effect of mesh geometry and distribution on sample properties were investigated. Additionally, the properties of mesh reinforced samples were compared to those of fiber reinforced and non-reinforced samples. In the future, hybrid geometry reinforcements will be investigated alongside with mechanical modeling of the composite systems.

**PM01.09.33**

Polymer Templating for Metallic Foam Fabrication with Wide-Ranging Compositional Control

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Metallic foam structures can be fabricated using a wide range of techniques; at the nanoscale de-alloying forms nm-sized ligament/pore foams in noble metals, while gas foaming at the meso-scale enables bulk material formation in engineering alloys. Capturing both length scales, nm-level porosity to sub-mm porosity, is challenging, and compositions accessible via these particular techniques are often mutually exclusive. This presentation addresses a new model of metal foam creating, using electrospray polymer fibers containing soluble metal acetate. Using a combination of oxidation and thermal reduction on electrospray fibers allows for multiple length scales of structural feature control, with meso-scale pores dominated by the fiber diameter and spacing, and ligament scale porosity and roughness dominated by thermal processing and diffusive processes. We demonstrate the fabrication of Cu-Ni alloys as a model solid solution structure, and create and characterize the architecture and microstructure of these materials between pure Cu and 50/50 Cu-Ni; any composition in this range is accessible using simple wet chemistry of the polymeric precursor used for electrosprining. The pyrolysis of the polymer controls the larger length scale, and the initial oxidation step creates a mixed oxide structure for the alloy systems. Subsequent reduction causes homogenization of the resulting ligaments. The alloy foams, with porous ligaments with dimensions on the order of 200 nm, and pores on the order of 50 nm, exhibit a strength four times that of the pure copper films with 400 nm ligament dimensions. The increase in strength is attributed to both solid solution strengthening and also fractionally impacted by the length scale of the ligaments.

**PM01.09.34**

Additive Manufacture of Hierarchically Porous Materials with High Resolution

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A ink system for additive manufacturing hierarchically porous materials with high resolution (ligament size < 100 μm) has been prepared by sol-gel approach. In this ink system, a resol polymer precursor was first synthesized and then mixed with other components including pluronic block-copolymer, other precursors, solvents and catalyst. The resulting clear and powder-free ink was direct-ink written into different structures and gelled at elevated temperature. The printing parts were further calcinated or carbonized to afford 3D hierarchically porous materials. The 3D products were characterized by SEM, SANS BET, TEM, Instron, FTIR/Raman spectroscopy and dielectric spectroscopy. They demonstrated potential application in energy storage and catalyst field.
PM01.09.35
**Parameterized Lattice with High Stiffness Through Engineered Functional Gradients**

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Topology optimization enables automated constraint-based design of structures. If a parametric lattice unit cell is used to construct the structure, a subsequent optimization pass can be run to redistribute mass within the generated topology and improve performance. A two-phase optimization scheme was used to generate a structure with a material gradient for supporting a cantilevered load. An isotropic unit cell with an effective modulus linearly dependent on relative density was used to populate an optimized cantilever structure. The second optimization pass then adjusted the beam diameters of each unit cell to improve stiffness. The resulting lattice structure was fabricated using a custom projection stereolithography system and mechanically tested. Four structures were used: 1) solid beam cantilever, 2) uniform lattice cantilever, 3) shape-optimized uniform lattice cantilever, and 4) shape- and material-optimized cantilever. Structures were cleaned and thermally post-processed to achieve high conversion. Mechanical testing showed that the flexural stiffness increased between the solid beam and the uniform lattice with additional gains for both shape-optimized and the shape- and material-optimized cantilevers.

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PM01.09.36
**Polypropylene and PETG 3D Printed Hybrid Structures**

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With the rise of 3-D polymer based printing, especially with the potential of the mass industrial utilization of the technology, there is a need for a better understanding for fabrication of hybrid structures utilizing the materials commonly used. To this end, using fused deposition modeling (FDM) 3D printing technique we fabricated Polypropylene (PP) and Polyethylene Terephthalate Glycol-modified (PETG) hybrid samples and analyzed their properties. The properties are directly related to the printing temperature, viscosity, and percentage within the print of the two components. Our studies confirmed that the hybrid samples have lower mechanical characteristics than the samples printed from either pure material. We associate the decrease in properties with adhesive issues at PP/PETG interface. To improve these properties, PP fiber was mixed with small amount of PETG to create diffusive bonding at the interface. Our approach is expected to be transferable to similar 3D printed polymer hybrid/composite systems that also face poor mechanical properties.

PM01.09.37
**Additive Manufacturing of New Structures for Heat Exchange**

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Recent advances in manufacturing are enabling a new range of material properties and product performance. Additive manufacturing of metals and alloys, specifically laser powder bed fusion (LPBF), allows for the production of large, robust and highly complex structures. Here we describe the development and testing of a heat exchanger for sCO2 power cycles with radically improved material efficiency and higher temperature tolerance than current technology. Triply Periodic Minimal Surfaces (TPMS) are chosen as candidate structures as previous work shows an expected order of magnitude improvement in heat transfer performance over tubes and flat plates. The project combines additive manufacturing of nickel superalloys with new, efficient hierarchical geometries, which are common in nature to achieve high interfacial area with low pressure drop, and are now achievable in synthetic with advanced manufacturing techniques. Focusing on the Schwarz-D geometry, flow characteristics and temperature fields were modeled to compare heat transfer coefficients. Thermal stress modeling was performed to couple heat transfer with solid mechanics. The process development of 5 candidate nickel super alloys is described, along with ambient and high temperature mechanical testing results. Bench scale measurements of mass transfer, pressure drop and device robustness are performed for comparison and to aid design of a viable process.

PM01.09.38
**A Hydrogel-Elastomer Hybrid Fabricated via 3D Printing**

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Mechanical integrity of hydrogel-elastomer hybrids is limited by the fracture toughness of hydrogel which disables the usage of many useful but fragile common hydrogels. This work fabricated a new hydrogel-elastomer hybrid with the help of 3D printing technique. The two materials are physical engaged into each other. Peeling test demonstrated that the new hybrid had superior mechanical integrity over the conventional hydrogel-elastomer anchored by chemicals. This method enables the mechanical integrity of hydrogel-elastomer hybrid to go beyond the fracture toughness of hydrogel.

PM01.09.39
**3D Printing of Bicontinuous Hierarchical Porous Carbons and Metal Oxides**

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Hierarchically porous materials are becoming promising candidates for biomedical, catalytic, and energy storage applications for their unique combination of low density, exceptional mechanical properties, large surface area, and excellent electrical conductivity. Recent research has focused in the topological design of cellular materials in order to satisfy multiple design objectives. Unfortunately, these design advances have not been met with similar advances in cellular material manufacturing as existing techniques constrain a designer to a predetermined part mesostructure, material type, and macrostructure. In an effort to address these limitations, we utilized 3D printing techniques to create designed architectures with bicontinuous hierarchically pores from 10nm to 1000 microns. 3D printing is one of emerging additive manufacturing techniques, which has been successfully commercialized for both prototyping and distributed manufacturing with industrial applications in architectures, automobile, aerospace, engineering, food, and biomedical areas. The key point is to develop a printable phase separable inks from precursor polymers. With this approach, we aim to demonstrate multi-scale (e.g., from the nano- to macro-length scales) assembly of arbitrarily complex, hierarchical 3D structures composed of carbons or metal oxides.
Structure and Energetic Characterization of Reactive Material Structures Formed by Kinetic Spray

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Reactive Material Structures (RMSs) are a new group of materials designed to have simultaneously the features of structural materials and energetic materials [1]. Their typical applications are considered for military uses like reactive fragments, respective bullets and reactive casings. This paper describes the manufacturing method for structurally robust RMSs, and shows the structural and energetic characteristics of the acquired RMSs. To acquire robust RMSs, we attempted kinetic spraying (i.e. cold spraying) of reactive material powders that is consisted of various compositions. As a result, we obtained tough RMSs of thickness over 1cm. The prepared RMSs were microscopy (SEM), X-ray diffraction (XRD) and universal testing machine (UTM). All RMSs show a porosity of less than 5% and a compressive strength of more than 250 MPa. To characterize energy releasing features under the environment of explosive detonation, the exothermic energy of each RMS was also analyzed using detonation calorimetric Analyzer (DCA). As a result, surprisingly, we found that RMSs can release more energy than conventional explosives.

References

Multifunctional Foam Synthesis—Paradigm Shift Towards Next Generation 3D Printable Composites

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Engineering photonic properties represents an important milestone in the synthesis of new composite materials with potential applications in photonic and electronic devices. Moreover, it is pivotal for their sustainable processing and reliability, while providing a new window to explore and further understand fundamentals.

Here, we will discuss our approach to novel materials synthesis, focusing on hierarchical-cellular-heterostructured-photonic composites, with control over their multifunctionality, for additive manufacturing. Particularly, we will present our most recent results on Ag-decorated TiO2 heterostructures' one-pot synthesis. We believe that this route is the foundation towards a series of multi-material-based 3D printable composites. Such multifunctional composites will trigger great advances in direct-write additive manufacturing, while leading to unprecedented developments in multi-functional materials and 3D processing for optoelectronics, environmental and energy related applications, and even regenerative medicine.